
**Imaging materials — Processed colour
photographs — Methods for measuring
thermal stability**

*Matériaux pour l'image — Photographies couleurs après traitement —
Méthodes de mesure de la stabilité thermique*

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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 18936 was prepared by Technical Committee ISO/TC 42, *Photography*.

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Introduction

This International Standard covers the methods and procedures for measuring the long-term, dark storage stability (thermal stability) of colour photographs.

Today, the majority of photographs are made with colour dyes and pigments. The length of time that such photographs are to be kept can vary from a few days to many hundreds of years, and the importance of image stability can be correspondingly small or great. Often the ultimate use of a particular photograph may not be known at the outset. Knowledge of the useful life of colour photographs is important to many users, especially since stability requirements often vary depending upon the application. For museums, archives and others responsible for the care of colour photographic materials, an understanding of the behaviour of these materials under various storage and display conditions is essential if they are to be preserved in good condition for long periods of time.

Any change in density, contrast or stain, whether due to colourant fading, changes in colourant morphology or discolouration of residual substances will change the appearance of the photograph.

The most damaging change tends to be contrast balance distortions brought about by differential fading of the three image colourants. These manifest themselves as shifts in colour balance from highlights to shadows that are especially noticeable in a scale of neutrals, for example a shift from magenta to green due to fading of the photograph's magenta image colourant, or from yellow to blue or cyan to red due to fading of the yellow or cyan colourant.

The second most consequential change is that caused by an increase in stain. The result may simply be a discolouration of the D_{\min} areas or a change in the D_{\min} colour balance.

Cyan, magenta, yellow and sometimes black, red, green and blue colourants that are dispersed in transparent binder layers, or absorbed onto special receiver layers coated onto transparent or white opaque supports, form the images of most modern colour photographs. Colour photographic images typically fade during storage and display; they will usually also change in colour balance because the image colourants seldom fade at the same rate. In addition, a yellowish (or occasionally other colour) stain may form and physical degradation may occur, such as embrittlement and cracking of the support and image layers. The rate of fading and staining is governed principally by the intrinsic stability of the colour photographic material and by the conditions under which the photograph is stored and displayed. The quality of chemical processing or post-processing is another important factor. Post-processing treatments and, in the case of digitally generated photographs, post-production treatments, such as application of lacquers, plastic laminates and retouching colours, also may affect the stability of colour materials.

The three main factors that influence storage behaviour, or dark stability, are the temperature and relative humidity of the air that has access to the photograph, as well as atmospheric pollutants to which the photograph is exposed. High temperature, particularly in combination with high relative humidity, will accelerate the chemical reactions that can lead to degradation of one or more of the image colourants. Low-temperature, low-humidity storage, on the other hand, can greatly prolong the life of photographic colour images for typical materials. Other potential causes of image degradation are microorganisms and insects.

Most modern photographs degrade too slowly under normal room conditions to permit evaluation of their dark storage stability within reasonable periods. However, it is possible to assess the probable, long-term changes of some photographs under low and moderate keeping conditions with accelerated, high-temperature tests, because recognizable losses in image quality under high temperatures are apt to be generated also under milder temperatures, if at a slower pace. The effects of relative humidity on thermal degradation can also be evaluated with Arrhenius tests conducted at two or more humidity levels.

Long-term changes in image density, colour balance and stain level can be reasonably estimated only when good correlation has been confirmed between accelerated tests and actual conditions of use.

Density changes induced by the test conditions and measured during and after incubations include those in the support and in the various auxiliary layers that may be in a particular product. With most materials, however, the major changes occur in the image-bearing layer. An exception to this is found in some inkjet papers where the inks are thermally stable and substrate yellowing is the failure mode (see Reference [9]).

The tests for predicting the stability of colour photographic images in dark storage are based on an adaptation of the Arrhenius method described by Bard et al. (see References [2] and [3]) and earlier references by Arrhenius, Steiger and others (see References [4], [5] and [6]). Although this method is derived from well understood and proven theoretical precepts of chemistry, the validity of its application to predicting changes of photographic images rests on empirical confirmation. Although many chromogenic-type colour products yield image fading and staining data in both accelerated and non-accelerated dark ageing tests that are in agreement with the Arrhenius relationship, some other types of products do not. For example, integral-type instant colour print materials often exhibit atypical staining at elevated temperatures; treatment of some chromogenic materials at temperatures above 80 °C and 60 %RH may cause loss of incorporated high-boiling solvents and abnormal image degradation; and the dyes of silver dye-bleach images deaggregate at combinations of very high temperature and high relative humidity, causing abnormal changes in colour balance and saturation (see Reference [7] in the bibliography). In general, photographic materials tend to undergo dramatic changes at relative humidities above 60 % (especially at the high temperatures employed in accelerated tests) owing to changes in the physical properties of gelatine and other binder materials. Lower maximum relative humidities may need to be tested for some of the more humidity-sensitive inkjet materials because of phase changes such as melting point or glass transition temperature.

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Imaging materials — Processed colour photographs — Methods for measuring thermal stability

1 Scope

This International Standard describes test methods for determining the long-term dark storage stability of colour photographic images.

It is applicable to colour photographic images made with traditional photographic materials. These images are generated with chromogenic, silver dye-bleach, dye transfer, dye-diffusion-transfer “instant” systems and similar systems. The test method specified in this International Standard also covers the dark-stability of digital colour images produced with dry- and liquid-toner electrophotography, thermal dye transfer (sometimes called “dye sublimation”), and inkjet printing systems.

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, *Photography and graphic technology — Density measurements — Part 3: Spectral conditions*

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

ISO 11664-4, *Colorimetry — Part 4: CIE 1976 L*a*b* Colour space*

ISO 18911, *Imaging materials — Processed safety photographic films — Storage practices*

ISO 18913, *Imaging materials — Permanence — Vocabulary*

ISO 18920, *Imaging materials — Reflection prints — Storage practices*

ISO 18924, *Imaging materials — Test method for Arrhenius-type predictions*

ISO 13655, *Graphic technology — Spectral measurement and colorimetric computation for graphic arts images*

ISO 18941, *Imaging materials — Colour reflection prints — Test method for ozone gas fading stability*

ISO 18944, *Imaging materials — Reflection colour photographic prints — Test print construction and measurement¹⁾*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18913 and the following apply.

3.1

operational control point

set point for equilibrium conditions measured at sensor location(s) in an exposure device

[ASTM G 113]

1) To be published.

3.2 operational fluctuations
positive and negative deviations from the setting of the sensor at the operational control set point during equilibrium conditions in a laboratory-accelerated weathering device

NOTE The operational fluctuations are the result of unavoidable machine variables and do not include measurement uncertainty. The operational fluctuations apply only at the location of the control sensor and do not imply uniformity of conditions throughout the test chamber.

[ASTM G 113]

3.3 operational uniformity
range around the operational control point for measured parameters within the intended exposure area within the limits of intended operational range

[ASTM G 113]

3.4 uncertainty (of measurement)
parameter, associated with the result of a measurement, that characterizes the dispersion of the values that could be reasonably attributed to the measurand

NOTE 1 The parameter may be, for example, a standard deviation (or a given multiple of it), or the half-width of an interval having a stated confidence level.

NOTE 2 Uncertainty of measurement comprises, in general, many components. Some of these components may be evaluated from statistical distribution of the results of series of measurements and can be characterized by experimental standard deviations. The other components, which can also be characterized by standard deviations, are evaluated from assumed probability distributions based on experience or other information.

NOTE 3 It is understood that the result of the measurement is the best estimate of the value of the measurand, and that all components of uncertainty, including those arising from systematic effects, such as components associated with corrections and reference standards, contribute to the dispersion.

[ASTM G 113]

4 Requirements

This International Standard specifies a set of recommended test methods with associated requirements for permitted reporting. Data from these tests shall not be used to make life expectancy claims, such as time-based print lifetime claims, either comparative or absolute. Conversion of data obtained from these methods for the purpose of making public statements regarding product life shall be in accordance with the applicable International Standard(s) for specification of print life.

The test methods in this International Standard may be useful as stand-alone test methods for comparison of the stability of image materials with respect to one specific failure mode. Data from the test methods of this International Standard may be used in stand-alone reporting of the absolute or comparative stability of image materials with respect to the specific failure mode dealt with in this International Standard, when reported in compliance with the reporting requirements of this International Standard. Caution shall be used when comparing test results for different materials. Comparisons shall only be made when using equipment with matching specifications, under matching test conditions.

Further clarification is required for this method because it makes use of the Arrhenius methodology which may directly lead to a predicted product lifetime. Fundamentally, the Arrhenius method defines a rate of change (see ISO 18924). This International Standard makes use of the Arrhenius method only as a predictor of rate of change for chemical reactions, not as a predictor of print longevity.

5 Sample preparation

5.1 Target selection

For general testing purposes, users of this International Standard are free to choose whatever target, patches and starting densities they feel are appropriate for their testing needs. An example of such a target is included in ISO 18944 along with requirements and recommendations for sample preparation. Applicable International Standard(s) for specification of print life may require the use of specific targets. Other recommendations for sample preparation are contained in ISO 18909.

5.2 Use of replicates and reference samples

At least two replicate prints are required. Replicates shall be located for testing in different regions of the test chamber volume.

It is recommended that reference samples be included in every exposure test to track consistency of the test procedures as well as unintended changes of test conditions (see Reference [21]).

6 Measurement conditions

Measurements and sample holding for measurement and next test phase preparation shall be conducted in a controlled environment with no time constraint, or in a less controlled environment with a time constraint. The measurement environment and sample holding environment can influence measured densities.

NOTE 1 "Sample holding environment" refers to the environment in which samples are held between test phases, such as before and after measurement, while the samples are not in the active test environment.

The controlled sample holding environment with no time constraint shall meet the following set of conditions: samples shall be kept in the dark at $23 \text{ }^{\circ}\text{C} \pm 2 \text{ }^{\circ}\text{C}$ and at a relative humidity of $(50 \pm 10) \%$ while waiting for measurement and while holding between test stages.

The sample holding environment shall be ozone-free ($\leq 2 \text{ nl/l}$ average ozone concentration over any 24 h period) for ozone-sensitive samples. Ozone sensitivity is determined in accordance with ISO 18941 and ISO 18944. A material that is not sensitive to ozone shall have demonstrated no measurable D_{\min} or printed patch colour change at ambient ozone exposure levels and measurement condition temperature and humidity, over time periods consistent with measurement and test staging time periods.

The controlled measurement environment with no measurement process time constraint shall meet the following set of conditions: ambient illuminance on the sample surface no greater than 200 lx, temperature of $23 \text{ }^{\circ}\text{C} \pm 2 \text{ }^{\circ}\text{C}$ and a relative humidity of $(50 \pm 10) \%$, and ozone-free ($\leq 2 \text{ nl/l}$ average ozone concentration over any 24 h period) for ozone-sensitive samples.

When sample holding and/or measurement are conducted in a less controlled environment, samples shall be held or measured in the less controlled environment for a maximum of 2 h for each test stage. The less controlled environment may be unfiltered for ozone, and shall have a maximum relative humidity of 75 % and maximum temperature of $30 \text{ }^{\circ}\text{C}$, with ambient illuminance on the sample surface less than or equal to 1 000 lx.

NOTE 2 Stray light decreases the accuracy of measurements taken in less controlled lighting environments. Shielding the measurement instrument from direct lighting so that the actual measurement surface lighting is no more than 200 lx can improve measurement accuracy and repeatability.

The temperature and humidity tolerances for the sample holding and measurement environments apply specifically to the vicinities in which the samples are held and measured. Operational fluctuations, operational uniformity and uncertainty of measurement shall be contained within the stated tolerances in those vicinities.

The measurement environment and sample holding environment, with respect to temperature, percentage relative humidity, ozone and light levels, and with respect to fluctuations and uniformity shall be reported in the test report.

The CIE colour coordinates of the D_{\min} patch (unprinted paper) shall be measured using ISO 13655 measurement condition M0 for the relative spectral power distribution of the flux incident on the specimen

surface. White backing is recommended in accordance with ISO 13655. Report the backing used or report the material opacity, according to ISO 2471, such that backing has no influence on the measurement. Measurement conditions shall be consistent throughout the test process. Conforming to ISO 13655, calculated tristimulus values and corresponding CIELAB values shall be computed using CIE illuminant D50 and the CIE 1931 standard colorimetric observer (often referred to as the 2° standard observer).

NOTE 3 With completely opaque materials such as the aluminium substrate used in outdoor testing, the backing has no relevance.

Optical densities shall be measured according to ISO 5-3, with the relative spectral power distribution of the flux incident on the specimen surface conforming to CIE illuminant A, ISO 13655 measurement condition M0, and spectral products conforming to Status A or Status T density, as appropriate for the material under test. White backing is recommended in accordance with ISO 5. Use ISO 5 standard reflection density as defined in ISO 5-4, allowing either annular influx mode or annular efflux mode. Either white or black backing is allowed. Report the backing used. Measurement conditions shall be consistent throughout the test process.

NOTE 4 When testing in accordance with an image life specification standard, then either standard status A or status T density is selected according to that specification standard.

A single measurement instrument shall be used for all of the measurements taken pertaining to a particular test. For example, initial patch values of a test target print and subsequent degraded patch values of that particular test target print shall be measured using the same measurement instrument. Replicate prints may be measured on separate measurement instruments as long as each is consistently measured on the same instrument used for its initial readings. According to best practice, in the case of equipment failure the test should be invalidated. A replacement instrument with a known offset, determined for the test measurement conditions and materials such as those being measured, may be used when the original instrument is not available. In this case, all measurements shall be corrected with the known offset.

NOTE 5 It is useful to retain freezer check print samples of the measurement materials so that instrument offsets can be measured if needed. Offset measurements from materials matched to those under test are preferred to measurements using BCRA tiles. Refer to ISO 18920 for print storage methods.

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7 Measured attributes

7.1 Definition of density attributes

The symbol for measured density is d .

7.2 Density attributes to be measured

The following Status A or Status T densities of the specimens shall be measured before and after the treatment interval.

a) $dN(R)_t$, $dN(G)_t$, $dN(B)_t$

the red, green and blue Status A or Status T densities of neutral patches that have been treated for time t , where t takes on values from 0 to the end of the test;

b) $dC(R)_t$, $dM(G)_t$, $dY(B)_t$

the red, green and blue Status A or Status T densities of cyan, magenta and yellow colour patches that have been treated for time t , where t takes on values from 0 to the end of the test,

c) $dR(G)_t$, $dR(B)_t$, $dG(R)_t$, $dG(B)_t$, $dB(R)_t$, $dB(G)_t$

the red, green and blue Status A or Status T densities of the composite secondary R, G, B colour patches that have been treated for time t , where t takes on values from 0 to the end of the test.

7.3 Definitions of colourimetry terms

L^* is CIELAB lightness, a^* and b^* are the CIELAB a^* and b^* coordinates respectively, as defined in ISO 11664-4.

7.4 Colourimetry values to be measured

The following colourimetry values of the specimens, prepared as described in Clause 5, shall be measured before and after the treatment interval.

L^*t , a^*t , b^*t

The lightness, red-green, and blue-yellow colour coordinates for the unprinted areas of specimens (paper white) that have been treated for time t , where t takes on values from 0 to the end of the test.

8 Calculations and computations

8.1 Computation of densitometric attributes

The calculations given in 8.2 to 8.9 shall be performed for patches with initial densities of 0,5, 1,0 and 1,5. Other starting densities may be used in addition, depending on the service needs of the specific product being tested.

8.2 Density change in d_{\min} patches

a) Red density change: $\Delta d_{\min}(R)_t = d_{\min}(R)_t - d_{\min}(R)_0$

b) Green density change: $\Delta d_{\min}(G)_t = d_{\min}(G)_t - d_{\min}(G)_0$

c) Blue density change: $\Delta d_{\min}(B)_t = d_{\min}(B)_t - d_{\min}(B)_0$

8.3 Percent density change in pure primary colour patches

a) Cyan patch: $\% \Delta dC(R)_t = [(dC(R)_t - dC(R)_0) \div dC(R)_0] \times 100$

b) Magenta patch: $\% \Delta dM(G)_t = [(dM(G)_t - dM(G)_0) \div dM(G)_0] \times 100$

c) Yellow patch: $\% \Delta dY(B)_t = [(dY(B)_t - dY(B)_0) \div dY(B)_0] \times 100$

8.4 Percent density change in secondary (mixed) colour patches

a) Magenta in Red patch: $\% \Delta dR(G)_t = [(dR(G)_t - dR(G)_0) \div dR(G)_0] \times 100$

b) Yellow in Red patch: $\% \Delta dR(B)_t = [(dR(B)_t - dR(B)_0) \div dR(B)_0] \times 100$

c) Cyan in Green patch: $\% \Delta dG(R)_t = [(dG(R)_t - dG(R)_0) \div dG(R)_0] \times 100$

d) Yellow in Green patch: $\% \Delta dG(B)_t = [(dG(B)_t - dG(B)_0) \div dG(B)_0] \times 100$

e) Cyan in Blue patch: $\% \Delta dB(R)_t = [(dB(R)_t - dB(R)_0) \div dB(R)_0] \times 100$

f) Magenta in Blue patch: $\% \Delta dB(G)_t = [(dB(G)_t - dB(G)_0) \div dB(G)_0] \times 100$

8.5 Percent density change in a composite neutral patch

a) Cyan in neutral patch: $\% \Delta dN(R)_t = [(dN(R)_t - dN(R)_0) \div dN(R)_0] \times 100$

b) Magenta in neutral patch: $\% \Delta dN(G)_t = [(dN(G)_t - dN(G)_0) \div dN(G)_0] \times 100$

c) Yellow in neutral patch: $\% \Delta dN(B)_t = [(dN(B)_t - dN(B)_0) \div dN(B)_0] \times 100$