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Imaging materials — Colour reflection prints — Test method for ozone gas fading stability

Matériaux pour l'image — Tirages par réflexion en couleurs — Méthode d'essai de la stabilité de la décoloration à l'ozone

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee 42, *Photography*.

This third edition cancels and replaces the second edition (ISO 18941:2017), which has been technically revised.

The main changes compared to the previous edition are as follows:

- the test methods for environmental stress factors have been changed to align with ISO 18944:2018;
- the calculations and computations section has been removed as they are now contained in ISO 18944:2018;
- [Annex A](#) has been removed as the method for interpolation is now contained in ISO 18944:2018, Annex B;
- the usage and reporting requirements have been updated to ensure consistency within the ISO 189## family.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

In image permanence testing, there are four environmental variables known to affect the stability of a photographic image: heat, light, moisture and air pollution, such as ozone^{[13][14][15][16][17][18][19][20][21][22][23][24][25][26]}. Although natural ageing under “real-world” environmental levels of these variables is considered the only certain test for image permanence, the high stability of most modern photographic products makes testing under ambient conditions too lengthy a process to be of practical use. Thus, a widely used alternative to natural ageing is accelerated ageing, whereby a sample specimen is exposed to each environmental variable individually and at levels considerably greater than ambient, forcing degradation of the image by that single factor in a far shorter length of time.

This document covers the equipment, methods and procedures for generating a known ozone exposure and the subsequent measurement and quantification of the amount of change produced within a photographic image due to that exposure. It is important to note that if predictions of absolute product longevity are of concern to the experimenter, then further knowledge shall be gained regarding the reciprocal behaviour of the test product under the experimental accelerated ozone conditions. See [Annex A](#) for more information on reciprocity.

Additionally, there are other known variables in an ozone test setup that can affect the rate at which an image will degrade in the presence of ozone. These include air flow over the sample, the nature of the chemical reaction that is occurring, the relative quantities of the reactants (ozone and colorant molecules) and the humidity content and the pH of the image recording layer. Each of these variables can affect the reciprocal response and needs to be understood for a clear analysis of the accelerated data.

In some products, such as most dyes on swellable inkjet media and in silver halide products in gelatine, the ozone reaction can be considered to be “diffusion-controlled,” whereby ozone first needs to permeate a protective surrounding matrix before coming in contact with a colorant molecule and reacting. Further, the reacted components then need to be desorbed and removed from the surface before fresh, unreacted molecules can again diffuse, adsorb and react. In this type of process, a simple increase in ozone concentration might or might not yield a proportional increase in reaction rate as diffusion, adsorption and, in some cases, desorption may be the dominant factor controlling the rate of reaction.

The relative quantities of the reactants (ozone and colorant) will also affect the rate of reaction and reciprocal behaviour. Under the assumed ambient conditions, a photographic image would undoubtedly contain a vast excess of colorant molecules relative to the local concentration of ozone molecules in the air. Here, ozone would likely be the limiting factor controlling the rate of reaction and, in the absence of other controlling factors, an increase in ozone concentration will produce a proportional increase in the rate of reaction. At some precise ozone concentration, the quantity of reactants would be equal and the reaction would proceed at a maximum rate. At this point, however, a further increase in ozone concentration would not accelerate the reaction rate, causing a failure in the reciprocal relationship that is required for converting accelerated data into predictions of ambient performance. For this reason, if product longevity predictions are to be made, this ozone concentration needs to be determined and never exceeded during testing.

This document has been primarily developed via testing with inkjet images on porous “instant-dry” photographic media, which have been shown to be susceptible to fading by oxidative gases present in polluted ambient air^{[13][14][19][20][21]}. While many chemical species may be present in polluted air, it has been shown that most of the fade observed for current inkjet systems can be explained by oxidation by ozone^{[21][27][28]}. Additionally, this method may reasonably be used for colour photographic images made with other digital and traditional “continuous-tone” photographic materials such as chromogenic silver halide, silver dye-bleach, dye transfer^[26], dye-diffusion-transfer “instant” and other similar systems. However, since these systems have, in general, been shown to be much less sensitive to oxidative degradation by ozone, relatively small levels of image degradation with this accelerated test method may not be realized within the typical duration of such a test for these imaging systems.

High levels of ozone, often found outside major metropolitan areas in summer months, together with high levels of humidity, will greatly accelerate the fade. Since ozone is a highly reactive gas, storage of photographs in any kind of gas-impermeable enclosure, such as framed behind glass or in an album,

will greatly reduce image degradation due to ozone. This method therefore relates primarily to the display of unprotected photographs.

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Imaging materials — Colour reflection prints — Test method for ozone gas fading stability

1 Scope

This document describes the equipment, methods and procedures for generating a known ozone exposure and the subsequent measurement and quantification of the amount of change produced within both digitally printed hardcopy images and traditional analogue photographic colour print images due to that exposure.

The test method described in this document uses increased levels of ozone to achieve an accelerated test. If the principal “gas fading” mechanism for a system is not ozone, this method might not be suitable and might give misleading results as to resistance of the test image to polluted air.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 1431-3, *Rubber, vulcanized or thermoplastic — Resistance to ozone cracking — Part 3: Reference and alternative methods for determining the ozone concentration in laboratory test chambers*

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

ISO 18913, *Imaging materials — Permanence — Vocabulary*

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

3.1

volume turnover

complete replacement of the air/gas volume within the test chamber

**3.2
agitation**

degree to which air/gas is circulated within the chamber resulting in a mixing of the air/gas at the surface of the test sample to overcome concentration gradients

Note 1 to entry: Agitation can be directly related to flow rate but inversely related to *volume turnover* (3.1). For a given incoming gas-flow velocity, the actual flow across the samples, and therefore the agitation, can be affected by chamber volume, with, for example, larger chamber volumes resulting in lower flow over the samples. Agitation of air/gas is important to ensure mixing so that any reaction by-products are carried away from the test samples.

**3.3
effective concentration**

concentration of ozone as experienced by the test object

Note 1 to entry: Concentration that results in a specific change in a specific sample after exposure for a specific time.

**3.4
closed-loop system**

system in which the air/gas volume is recirculated within the test chamber, with ozone added as needed to maintain the desired aim concentration

**3.5
open-loop system**

system where the air/gas volume continually enters, flows through and exits the system with no recirculation

**3.6
ideal mixing**

sufficient *agitation* (3.2) that results in uniform concentration throughout the chamber, such that no localized concentration gradients exist across the test samples

**3.7
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 1 to entry: Operational fluctuations are the result of unavoidable machine variables and do not include measurement uncertainty. Operational fluctuations apply only at the location of the control sensor and do not imply uniformity of conditions throughout the test chamber.

[SOURCE: ASTM G113]

**3.8
operational uniformity**

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

[SOURCE: ASTM G113]

**3.9
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 measurement

Note 1 to entry: The parameter might 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 to entry: Uncertainty of measurement comprises, in general, many components. Some of these components can 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 to entry: It is understood that the result of the measurement is the best estimate of the value of the measurement 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.

[SOURCE: ISO/IEC Guide 98 3:2008, 2.2.3]

4 Requirements

This document 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 documents for specification of print life.

The test methods in this document can be useful as stand-alone test methods for comparing the stability of image materials with respect to one specific failure mode. Data from the test methods of this document can be used in stand-alone reporting of the absolute or comparative stability of image materials with respect to the specific failure mode described in this document, when reported in accordance with the reporting requirements of this document. Caution shall be used when comparing test results for different materials. Comparisons shall be limited to test cases using equipment with matching specifications and matching test conditions.

5 Sample preparation

5.1 Target selection

For general testing purposes, users of this document 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 Standards for specification of print life may require the use of specific targets. Other recommendations for sample preparation are contained in ISO 18909. Image prints may also be used. When specific starting densities are desired or required, there may not be a step on a printed test target that corresponds to the exact desired density. Interpolation between two neighbouring density patches can be used to predict the values for the exact desired starting density. See ISO 18944:2018, Annex B for details on interpolation between two neighbouring density patches.

5.2 Use of replicates and reference samples

At least two replicate prints are required for each test case. 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 in test conditions (see Reference [12]).

6 Holding and 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 ± 2) °C and (50 ± 10) % of relative humidity (RH) while waiting for measurement and while holding between test stages.

The sample holding environment shall be ozone-free (<2 nl/l¹⁾ average ozone concentration over any 24 h period) for ozone-sensitive samples.

Ozone sensitivity shall be determined in accordance with this document; ISO 18944 may also be used. A material that is not sensitive to ozone shall have demonstrated no measurable change in minimum density, D_{\min} , (unexposed processed media or unprinted substrate) or printed patch colour, 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 not less than 200 lx, temperature of (23 ± 2) °C, (50 ± 10) % RH and ozone-free (<2 nl/l average ozone concentration over any 24 h period) for ozone-sensitive samples.

If either sample holding or measurement is 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 RH of 75 % and a maximum temperature of 30 °C, with ambient illuminance on the sample surface up 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 not less 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, relative humidity, ozone and light levels, fluctuations and uniformity shall be reported in the test report.

The CIE colour coordinates of the D_{\min} patch (unprinted paper) shall be measured in accordance with ISO 13655 measurement conditions for the relative spectral power distribution of the flux incident on the specimen surface. The conditions are chosen to be relevant to the characteristics being investigated. White backing is recommended in accordance with ISO 13655. Report the backing used or the material opacity according to ISO 2471, stating that the backing has no influence on the measurement. Measurement conditions shall be consistent throughout the test process. In accordance with ISO 13655, calculated tristimulus values and corresponding CIELAB values shall be computed using the illuminant and standard observer conditions applicable to the material under test.

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 in accordance with 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 applicable to the characteristics being investigated, 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 5-4. ISO 5 standard reflection density as defined in ISO 5 4 shall be used, 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, 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

1) 1 nl/l = 1 ppb (1×10^{-9}). Although the notation "ppb" (parts per billion) is widely used in the measurement and reporting of trace amounts of pollutants in the atmosphere, it is not used in International Standards because it is language-dependent.