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

National foreword

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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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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
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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 18941 was prepared by Technical Committee ISO/TC 42, *Photography*.

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[14][15][16][17][18][19][20][21][22][23][24][25][26][27]. 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 International Standard 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 needs to be gained regarding the reciprocal behaviour of the test product under the experimental accelerated ozone conditions. See Annex B 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 International Standard 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[14][15][20][21][22]. 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[22][28][29]. 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[27], dye-diffusion-transfer "instant" and other similar systems. However, since these systems have, in general, much greater stability to ozone, the validity of this accelerated test method has not yet been verified in these systems.

High levels of ozone, often found outside major conurbations 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.

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

1 Scope

This International Standard 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 International Standard 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 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 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*

ISO 18944, *Imaging materials — Reflection colour photographic prints — Test print construction and measurement*[1)]

3 Terms and definitions

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

3.1
air/gas
mixture of atmospheric air and ozone inside the test chamber

3.2
volume turnover
complete replacement of the air/gas volume within the test chamber

3.3
volumetric turnover rate
rate at which volume turnover occurs

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1) To be published.

3.4
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 Agitation can be directly related to flow rate but inversely related to volume turnover. 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.

3.5
air velocity at sample

rate of flow of air/gas across the sample plane, as opposed to the flow of air/gas within the chamber volume, or within the entering or exiting ports

NOTE Expressed in reciprocal milliseconds (ms^{-1}).

3.6
effective concentration

concentration of ozone as experienced by the test object, i.e. the concentration that results in a specific change in a specific sample after exposure for a specific time

3.7
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

3.8
open-loop system

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

3.9
ideal mixing

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

3.10
operational control point

set point for equilibrium conditions measured at one or more sensor locations in an exposure device

NOTE Adapted from ASTM G113.

3.11
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 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.

NOTE 2 Adapted from ASTM G113.

3.12
operational uniformity

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

NOTE Adapted from ASTM G113.

3.13
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 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 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 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.

NOTE 4 Adapted from ISO Guide 98-3:2008, 2.2.3.

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 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 International Standard 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 International Standard, when reported in accordance with the reporting requirements of this International Standard. 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 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 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.

6 Measurements

6.1 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 [13]).

6.2 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) % RH while waiting for measurement and while holding between test stages.

The sample holding environment shall be ozone-free [≤2 nl/l[2] average ozone concentration over any 24 h period] for ozone-sensitive samples. Ozone sensitivity is determined in accordance with this International Standard and ISO 18944. A material that is not sensitive to ozone shall have demonstrated no measurable change in minimum density, d_{min}, 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 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 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 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.

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[2] 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.

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 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. See ISO 18920 for print storage methods.

6.3 Measured attributes

6.3.1 Definition of density terms

The symbol for measured density is d.

6.3.2 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) $d\mathrm{N(R)}_t$, $d\mathrm{N(G)}_t$, $d\mathrm{N(B)}_t$

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) $d\mathrm{C(R)}_t$, $d\mathrm{M(G)}_t$, $d\mathrm{Y(B)}_t$

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) $d\mathrm{R(G)}_t$, $d\mathrm{R(B)}_t$, $d\mathrm{G(R)}_t$, $d\mathrm{G(B)}_t$, $d\mathrm{B(R)}_t$, $d\mathrm{B(G)}_t$,

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.

6.3.3 Definitions of colorimetry terms

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

6.3.4 Colorimetry values to be measured

The following colorimetry 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, which are the lightness, red-green and blue-yellow colour coordinates, respectively, 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.

7 Calculations and computations

7.1 Computation of densitometric attributes

Calculations for 7.2 to 7.8 shall be performed for selected patches with a range of initial densities.

7.2 Density change in d_{min} patches

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

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

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

7.3 Percentage density change in primary colour patches

a) Cyan patch: $\%\Delta d\text{C}(\text{R})_t = \{[d\text{C}(\text{R})_t - d\text{C}(\text{R})_0] \div d\text{C}(\text{R})_0\} \times 100$

b) Magenta patch: $\%\Delta d\text{M}(\text{G})_t = \{[d\text{M}(\text{G})_t - d\text{M}(\text{G})_0] \div d\text{M}(\text{G})_0\} \times 100$

c) Yellow patch: $\%\Delta d\text{Y}(\text{B})_t = \{[d\text{Y}(\text{B})_t - d\text{Y}(\text{B})_0] \div d\text{Y}(\text{B})_0\} \times 100$

7.4 Percentage density change in secondary (mixed) colour patches

a) Magenta in Red patch: $\%\Delta d\text{R}(\text{G})_t = \{[d\text{R}(\text{G})_t - d\text{R}(\text{G})_0] \div d\text{R}(\text{G})_0\} \times 100$

b) Yellow in Red patch: $\%\Delta d\text{R}(\text{B})_t = \{[d\text{R}(\text{B})_t - d\text{R}(\text{B})_0] \div d\text{R}(\text{B})_0\} \times 100$

c) Cyan in Green patch: $\%\Delta d\text{G}(\text{R})_t = \{[d\text{G}(\text{R})_t - d\text{G}(\text{R})_0] \div d\text{G}(\text{R})_0\} \times 100$

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

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

f) Magenta in Blue patch: $\%\Delta d\text{B}(\text{G})_t = \{[d\text{B}(\text{G})_t - d\text{B}(\text{G})_0] \div d\text{B}(\text{G})_0\} \times 100$

7.5 Percentage density change in composite neutral patch

a) Cyan in neutral patch: $\%\Delta d\text{N}(\text{R})_t = \{[d\text{N}(\text{R})_t - d\text{N}(\text{R})_0] \div d\text{N}(\text{R})_0\} \times 100$

b) Magenta in neutral patch: $\%\Delta d\text{N}(\text{G})_t = \{[d\text{N}(\text{G})_t - d\text{N}(\text{G})_0] \div d\text{N}(\text{G})_0\} \times 100$

c) Yellow in neutral patch: $\%\Delta d\text{N}(\text{B})_t = \{[d\text{N}(\text{B})_t - d\text{N}(\text{B})_0] \div d\text{N}(\text{B})_0\} \times 100$

7.6 Colour balance shift in composite neutral patch

Contrast and colour balance distortions brought about by differential fading of the three image colorants can result in significant visually degrading effects. These can be measured as shifts in colour balance from highlights to shadows and 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 colorant, or from yellow to blue or cyan to red due to fading of the yellow or cyan colorant.

Neutral colour balance shift is calculated as the difference in percentage change between any two primary colours of a neutral patch. The percentage change of individual primary colours in a neutral patch is defined in 7.5.

a) Cyan-magenta shift: $\%\Delta d\text{N}(\text{R-G})_t = |\%\Delta d\text{N}(\text{R})_t - \%\Delta d\text{N}(\text{G})_t|$

b) Magenta-yellow shift: $\%\Delta d\text{N}(\text{G-B})_t = |\%\Delta d\text{N}(\text{G})_t - \%\Delta d\text{N}(\text{B})_t|$

c) Yellow-cyan shift: $\%\Delta d\text{N}(\text{B-R})_t = |\%\Delta d\text{N}(\text{B})_t - \%\Delta d\text{N}(\text{R})_t|$

7.7 Colour balance shift in secondary (mixed) colour patches

Secondary colour balance shift is calculated as the difference in percentage change between the two primary colours of each secondary colour patch. The percentage change of the individual primary colours in each secondary colour patch is defined in 7.4.

a) Cyan-magenta shift in Blue patch: $\%\Delta d\mathrm{B(R\text{-}G)}_t = |\%\Delta d\mathrm{B(R)}_t - \%\Delta d\mathrm{B(G)}_t|$

b) Magenta-yellow shift in Red patch: $\%\Delta d\mathrm{R(G\text{-}B)}_t = |\%\Delta d\mathrm{R(G)}_t - \%\Delta d\mathrm{R(B)}_t|$

c) Yellow-cyan shift in Green patch: $\%\Delta d\mathrm{G(B\text{-}R)}_t = |\%\Delta d\mathrm{G(B)}_t - \%\Delta d\mathrm{G(R)}_t|$

7.8 Colour balance in d_{min} patches by colorimetry

Colour balance in the d_{min} patches is calculated using the following equation:

$$\Delta E^*_{\mathrm{ab}} = \sqrt{(L^*_t - L^*_0)^2 + (a^*_t - a^*_0)^2 + (b^*_t - b^*_0)^2}\ .$$

where L^*, a^*, and b^* are the colour coordinates of the d_{min} patch at the initial time 0 and at time t, as defined by ISO 11664-4.

7.9 Effects of colorant fading and stain formation on colour photographs

Any change in density, contrast or stain, whether due to colorant fading, changes in colorant 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 colorants.

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.

8 Test methods — Gas fading (ozone)

8.1 General

For the purpose of predicting fade rates, it is assumed that increasing ozone concentration should proportionally increase the rate of fading. This has been generally shown to be the case, but exceptions are known[14][16][17][18][24] (see also Annex B).

8.2 Apparatus

WARNING — Attention is drawn to the highly toxic nature of ozone. Efforts should be made to minimize the exposure of workers at all times. In the absence of more stringent or contrary national safety regulations in member body countries, it is recommended that 0,1 microlitres of ozone per litre of air of the surrounding atmosphere by volume be regarded as an absolute maximum concentration to which a worker shall be exposed, while the maximum average concentration should be appreciably lower.

NOTE An exhaust vent to remove ozone-laden air is advised.

8.2.1 Ozone test device

8.2.1.1 General

Two general types of ozone test device can be used, each having unique systems to deliver ozone to the test samples. Both designs may be combined with either open- or closed-loop circuits for feeding with ozonized air.

Additionally, there are inherent differences in the ozone control strategies for each (see 8.2.5). It is critical that any chamber design result in turbulent, not laminar, flow to maintain consistent ozone concentrations at the sample surface (see 8.2.5 for additional information on turbulent flow).

8.2.1.2 Design 1 (chamber circulation design)

This design consists of an enclosed chamber at atmospheric pressure into which multiple test samples can be simultaneously placed and ozonized air (i.e. air whose oxygen content has been partially converted to a specific ozone concentration) can be delivered at a given concentration, temperature, relative humidity (RH), volume turnover and agitation. Preliminary tests should be run to ensure that this regime is not in the concentration-sensitive region of the materials being tested (see 8.2.5). It is recommended that the laboratory run reference samples periodically to maintain this condition.

NOTE Agitation can be directly related to flow rate but inversely related to volume turnover. 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. Therefore, it is important to meet the requirements of 8.2.5 with respect to effective mixing within the chamber and equilibration into the media.

The chamber shall be lined with, or constructed from, a material (such as stainless steel, anodized aluminium, or ozone-resistant polycarbonate) that does not readily decompose ozone. Dimensions shall be such that the requirements of Clause 9 can be met.

The chamber may be provided with a window through which the test specimen can be observed. A light to examine the test specimen may be installed. Light entering the test chamber shall be limited so as not to confound results. If an ultraviolet (UV) source is used for ozone generation, the equipment design shall prevent any UV radiation from entering the test chamber.

Figures 1 and 2 provide illustrative examples of loop-system chamber designs, in which the loop is either closed or open. In the case of a closed-loop system (Figure 1), ozonized air for feeding the chamber is only partially replenished, whereas in an open-loop system (Figure 2), ozonized air is continuously prepared fresh from purified laboratory air. In any case, replacement of air is necessary to remove reaction products. For Figure 1, make-up air would be added just ahead of the ozonizer.

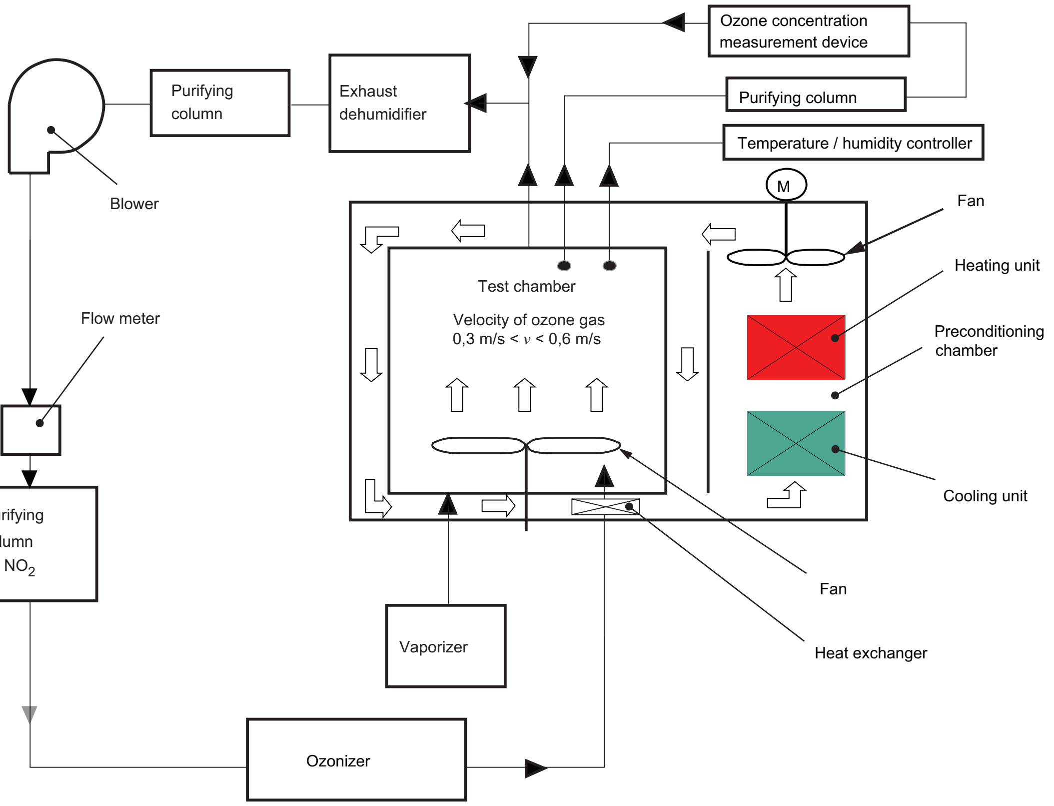
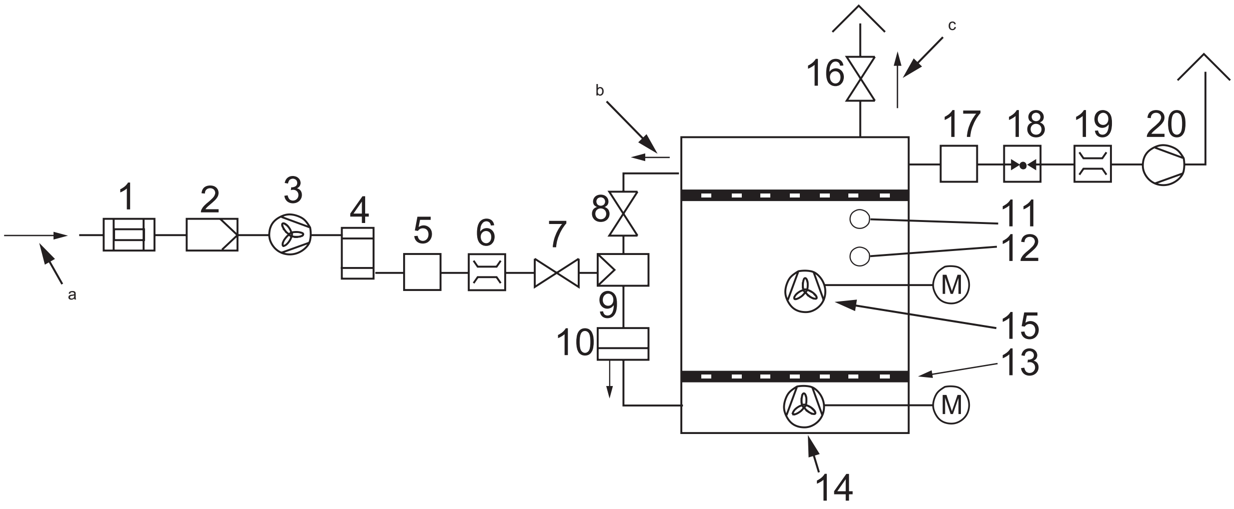


Figure 1 — Example of a closed-loop system



Key

1	activated carbon filter	11	temperature sensor
2	dust filter	12	humidity sensor
3	blower	13	plate with holes
4	air dryer	14	circulation blower
5	ozone generator	15	extra blower
6	flow meter	16	valve and exhaust hood
7	valve	17	ozone analyser
8	valve	18	pressure meter
9	temperature control	19	flow meter
10	humidity control	20	pump

[a] Fresh air.

[b] Circulating air.

[c] Exhaust.

Figure 2 — Example of an open-loop system

8.2.1.3 Design 2 (local injection or direct-impingement design)

This design consists of a delivery system that supplies aim-concentration ozonized air directly, and uniformly, to the entire surface of each individual test sample. Conceptually, each sample is contained within its own test chamber and isolated from the effects of ozone quenching by other samples in the test device.

The chamber shall be lined with, or constructed from, a material (such as stainless steel, anodized aluminium, or ozone-resistant polycarbonate) that does not readily decompose ozone. Dimensions shall be such that the requirements of Clause 9 can be met.

Figure 3 describes a typical direct-impingement design.

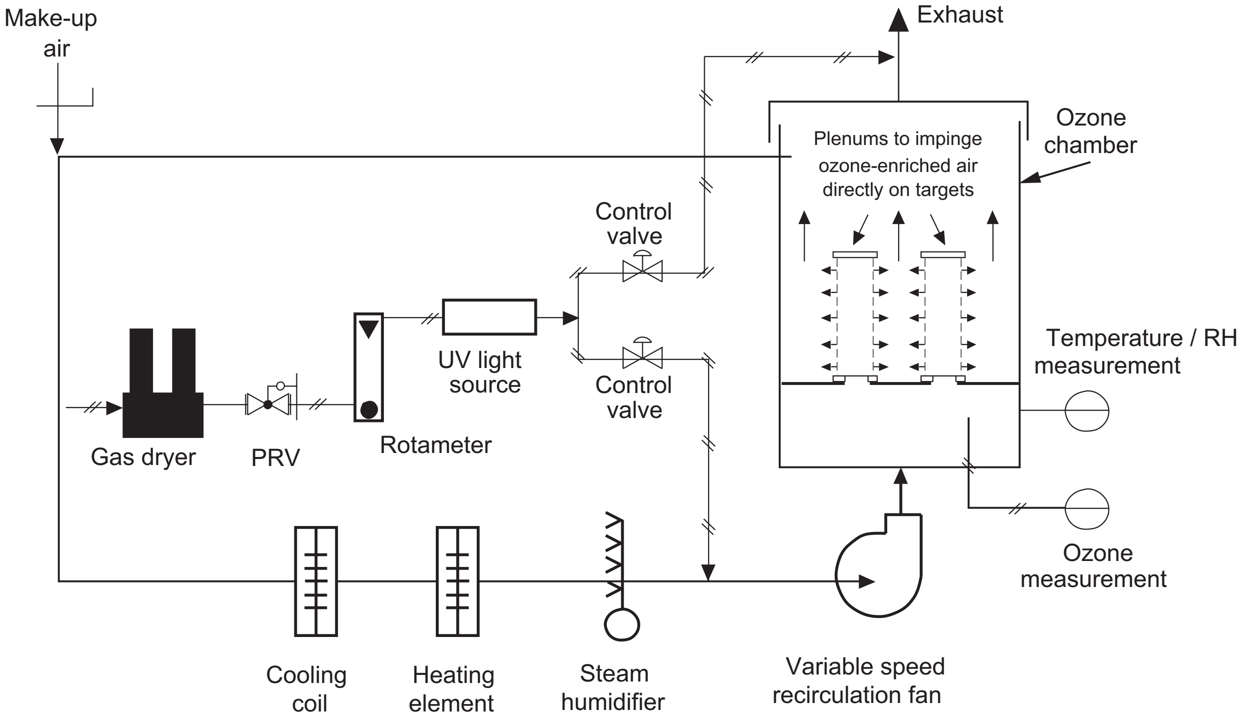


Figure 3 — Example of a direct-impingement system

8.2.2 Source of ozonized air

Any methodology that can generate ozone with sufficient quantity, purity and flow rate may be used as an ozone source. Examples of such apparatus include:

a) an ultraviolet lamp;

b) a corona discharge unit.

It is known that corona discharge units can generate other pollutants (such as oxides of nitrogen) when air, rather than purified oxygen, is used as the ozone source, and that this feature can change as the unit ages. In addition, if the air is not dried before ozonation, significant quantities of acids from oxides of nitrogen can be produced. In any case, total oxides of nitrogen in the chamber shall not exceed 2 nl/l when testing at 1 μl/l [3] or 0,2 % of ozone concentration when testing at ozone concentrations greater than 1 μl/l.

For corona discharge systems that are designed to eliminate or exclude oxides of nitrogen or their precursors (for example ones that start with pure oxygen instead of air), the measuring and reporting of oxides of nitrogen is not necessary. It should be understood that, for some systems, very small levels of oxides of nitrogen can cause rapid image degradation and would confound the measurement of change attributable to ozone. In any case, the reporting shall reference the manufacturer’s specification for ozone purity.

Air used for generation of ozone shall first be purified, for example by passing it over activated charcoal. It shall be free from any contaminants likely to affect the ozone concentration, sample fading, or estimation of ozone concentration.

The ozonized air shall be fed from the generator into the air delivery system. A heat exchanger shall be provided to adjust the gas temperature to that required by the test and shall be brought to the specified relative humidity level by the introduction of moisture into the gas flow.

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[3] 1 μl/l = 1 ppm (1×10^{-6}). Although the notation “ppm” (parts per million) 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.

8.2.3 Means for adjusting, controlling and maintaining ozone concentration

8.2.3.1 General

Adjustment of the concentration of ozone may be, but does not have to be, automatic. The voltage/drive signal to the lamp or corona discharge unit can be varied to control the production of ozone. Alternatively, a shield can be used to mask part of the lamp. The adjustment shall be such that the concentration and tolerances given in 9.4 can be maintained.

Each test device design described in 8.2.1 has unique control considerations, which are outlined in 8.2.3.2 and 8.2.3.3.

8.2.3.2 Design 1

In a multi-sample chamber, it is common for a group of samples to begin quenching significant quantities of ozone as they are placed into the chamber. As each individual sample reacts with the surrounding ozone, the effective concentration to which all other samples are exposed would consequently be lower than the entering ozone concentration. If the entering concentration is on aim, then the effective chamber concentration would be lower than aim. This is not an acceptable testing condition and would yield erroneous results.

In this chamber design, the effective test ozone concentration is best represented by the exiting chamber ozone concentration. It is important to note that if the exiting ozone concentration is at the desired aim and if significant quenching is occurring, then the entering ozone concentration would be greater than aim. In this situation, testing accuracy is highly dependent upon ideal (or near-ideal) mixing, to ensure that a concentration gradient does not exist across the chamber. Any concentration gradient would result in some samples receiving a greater exposure than others, depending on their proximity to the incoming air supply. Additionally, the greater the difference between entering and exiting ozone concentrations, the greater the potential of a gradient existing, and the greater the amount of mixing that would be required to diminish the effect. A test piece carrier, like that described in 8.2.6, can greatly aid in equalizing the effects of a concentration gradient present within a chamber. Concentration gradients can also be minimized by layout and choice of sample positions such that no shielding of agitated air flow occurs.

Another solution would be to have a sufficient bulk quantity of ozone in the chamber so that ozone quenching by the sample specimens is virtually undetectable relative to the total bulk ozone quantity. This can be achieved by removing samples from the chamber until quenching cannot be detected from measurement noise, or by increasing the total bulk volume of aim-concentration air flow into the chamber. It should be noted that, with fixed piping dimensions in the air/ozone delivery system, an increase in the total bulk air volume will increase the velocity of flow across the samples. If the test chamber is not already operating at a level that is insensitive to changes in flow, then this control method will yield inconsistent results.

Therefore, in a multi-sample chamber design, the exiting chamber ozone concentration shall be equal to the desired aim ozone concentration, with an operational fluctuation within ±4 % of aim, for the duration of testing, and mixing shall be sufficient to demonstrate that an ozone concentration gradient does not exist across the chamber. If rapidly fading reference samples (see 8.3) placed at different points within the chamber fade at the same rate, then a concentration gradient is unlikely to exist across the chamber and sufficient mixing has been demonstrated.

With the assumption of ideal or near-ideal mixing, the exiting chamber ozone concentration represents the concentration within the chamber. Therefore, the exit concentration shall be monitored if only one concentration is measured for the duration of the test.

In addition, after each occasion that the test chamber is opened for insertion or inspection of the test specimen, the ozone concentration shall return to the test concentration within 10 % of the total test time.

Provision to purge the chamber of ozone before allowing access should be incorporated and a suitable interface fitted to prevent ozone generation while the chamber door is open.

8.2.3.3 Design 2

In this design, the supply of ozonized air to each individual sample shall be equal to the desired aim ozone concentration, with an operational fluctuation within ±4 % of aim for the duration of testing.

In addition, after each occasion that the test device is opened for insertion or inspection of the test specimen, the ozone concentration shall return to the test concentration within 10 % of the total test time.

Provision to purge the device of ozone before allowing access should be incorporated and a suitable interface fitted to prevent ozone generation while the chamber door is open.

8.2.4 Means of determining the ozone concentration

A means of sampling the ozonized air from the desired location in the air circulation and delivery system and a means of estimating its ozone content shall be provided.

Two suitable apparatuses are:

a) electrochemical;

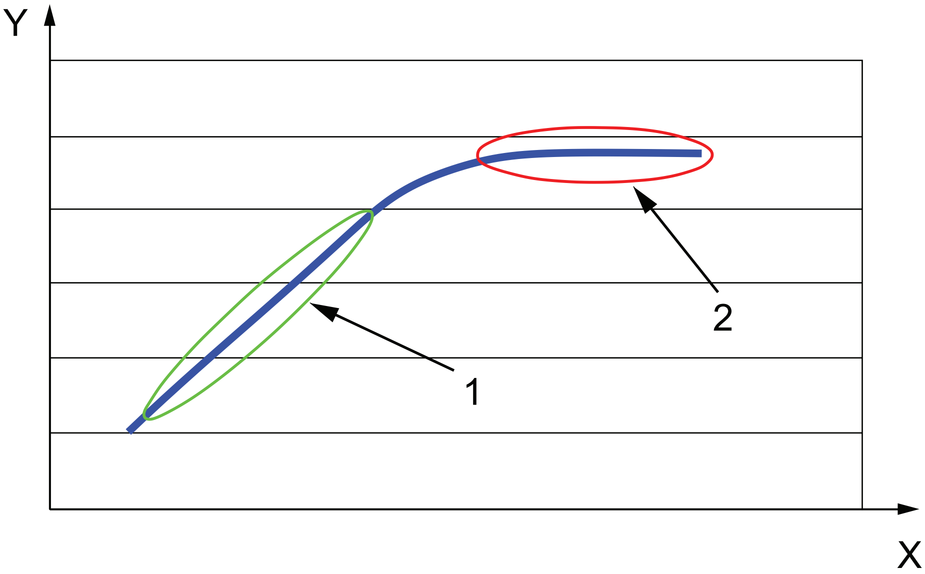
b) UV absorption.

The method used for calibrating each apparatus shall be as specified in ISO 1431-3.

8.2.5 Means of controlling gas flow

For inkjet prints, in particular those on porous media, it is known that the flow rate over the sample can have a significant impact on the results, especially for fast fading systems[17]. It is important that the velocity of gas in the chamber be high enough to provide fast and effective mixing in the chamber and equilibration into the media, as well as to prevent ozone starvation of the samples. Additionally, a major goal of this International Standard is to maximize interlaboratory reproducibility of ozone testing.

Therefore, the velocity of gas inside the chamber in the vicinity of the samples shall be such that the rate of fade or d_{min} yellowing is independent of the rate of flow, for the photographic products under test (see Figure 4).



Key

X	ozone concentration, flow rate or agitation, in arbitrary units (AU)
Y	rate of change (AU)
1	dependent portion
2	independent portion

Figure 4 — Conceptual relationship between ozone concentration, flow or agitation and rate of fade or yellowing

It is recommended that the velocity of gas inside the chamber be 0,3 ms^{-1} to 0,6 ms^{-1}. To achieve proper ozone replenishment at the sample surface, it is helpful to have an air flow that impinges on the samples at 90 °, or to provide at least a turbulent component in the agitating chamber air flow. In case of slow laminar flow over the specimen, local depletion of ozone might occur.

NOTE When using a rotating test piece carrier, such as that described in 8.2.6, air flow will not be impinging upon the sample at an angle of 90 ° at all times, but this is an acceptable test configuration.

If gas velocity values or geometries outside this range are to be used, the user shall conduct such experiments as are necessary, at several different gas flow rates, to ensure that the rate of change in the samples is insensitive to air flow velocity and impingement angle. This is necessary to confirm that the rate of change in image density due to ozone is independent of the rate of flow for the photographic products under test (see Figure 4).

8.2.6 Test piece carrier

Clamps or grips are often helpful for holding the test specimens. If used, the clamps or grips shall be made of a material that does not readily decompose in ozone (for example aluminium or stainless steel). The sample mounting should ensure that the back face of the sample is held against aluminium or stainless steel, or other such material, so that ozone attack from the reverse of the print is minimized.

The use of a mechanically rotating carrier mounted in the test chamber, and upon which the clamps or grips are mounted, can be useful to equalize the effect of different ozone concentrations and gas velocities within the chamber. In one example of a suitable carrier, the test specimens move at a speed of between 20 mm/s and 25 mm/s in a plane normal to the gas flow. Each follows consecutively the same path such that the same position within the chamber is visited by the same piece every 8 min to 12 min, and the area swept by the test piece (shade shown in Figure 5) is at least 40 % of the available cross-sectional area of the chamber.

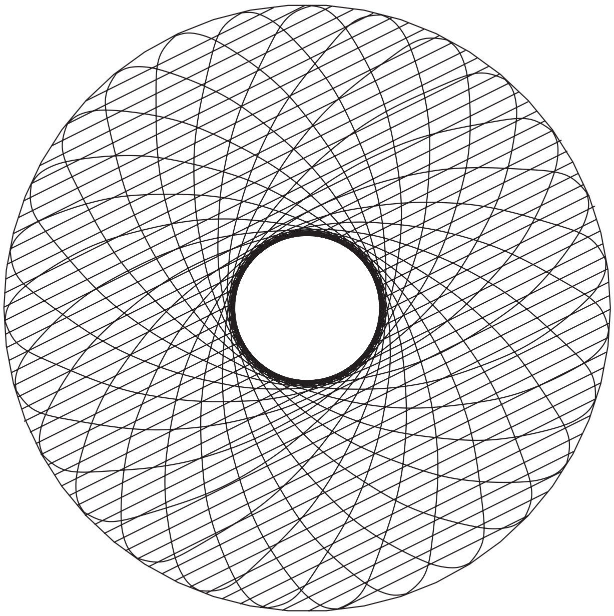


Figure 5 — Path of test piece and swept area (shaded)

Whichever geometry or velocity is used, the user shall ensure that the results are independent of the sample location in the chamber.

8.3 Test procedure

Adjust the rate of flow, temperature and relative humidity levels to that required and place the suitably prepared and conditioned samples into the chamber. Set the ozone concentration to that required. Maintain the test parameters throughout the test period. When the test period is completed, the ozone generation should be stopped and a purge cycle engaged. Although samples are preferably removed for examination as soon as the purge period is completed, they may be left in the test chamber if the ozone concentrations are known to be less than 2 nl/l.

The nature of this test is such that measurement of sample change is only possible periodically. Depending on the application, samples may be faded for a specific time period or to a designated density change, or endpoint. If test samples are being faded to a specific endpoint, it may be necessary to run the test for a number of cycles and to measure the change after each cycle of exposure, repeating until it has reached or passed this chosen endpoint. An applicable specification standard can be used to determine proper endpoint criteria, if necessary. The number of cycles required will depend on the material, but the cycle duration should be set such that the sample reaches its endpoint in no less than three cycles.

The duration of each cycle of exposure should be set such that the test is within 10 % of its set point for 90 % of the test duration.

It is recommended that each cycle of the exposure contain a reference sample, chosen such that it loses between 20 % and 40 % density during the cycle. The reference sample should be of the same technology as the test sample, for example inkjet print on porous media.

A minimum of two duplicate test specimens shall be tested. It is recommended that they not share the same exposure cycles.

Unless constrained by the requirements of a lifetime specification standard, it is acceptable to test for a shorter duration until a change is reliably detected, i.e. the test has progressed beyond the noise of the system. Low-percentage colorant loss, change in colour balance, or change in d_{min} can be used for stable materials, so long as a signal can be distinguished from test system noise. A prediction shall only apply for the endpoint used. Extrapolations cannot be made to other endpoints.

9 Test environment conditions

9.1 Humidity control calibration

Each test chamber shall be calibrated for relative humidity control and measurement accuracy by using a chilled mirror hygrometer or other type of measurement device calibrated and traceable to a national standards bureau responsible for certifying reference weights and measures. The calibration shall include the full temperature and relative humidity ranges that are to be used in the ensuing test processes. A check of the calibration shall be performed when there is any indication of sensor failure. Ongoing use of redundant sensors is recommended so that sensor integrity can be ascertained.

9.2 Relative humidity

The preferred level of relative humidity shall be 50 % at the control temperature. Other levels may be used, such as 30 % and 70 %, where such levels reflect actual product use conditions. In all cases, the relative humidity test conditions shall be reported. The relative humidity shall be maintained and controlled throughout testing with an operational fluctuation within ±6 % of aim. The 24 h running average of the operational fluctuation of the relative humidity, sampled at least every 15 min, shall be within ±2 % of aim. The running average shall not include the test condition transition time of at most 1 h after operation at the test condition is initiated. Operational uniformity of the equipment at the test conditions shall be evaluated prior to test start, and relative humidity shall be within ±3 % of aim, at a constant temperature. Regions of the test chamber shall be selected to comply with the required operational uniformity conditions. If the running average of the operational fluctuation does not meet the requirement, it shall be documented and explained.

9.3 Temperature

The preferred air temperature is 23 °C. Other temperatures, such as 15 °C or 40 °C, may be used where such temperatures reflect product use conditions and are reported. The air temperature shall be maintained and controlled throughout testing with an operational fluctuation within ±2,0 °C of aim. The 24 h running average of the operational fluctuation, sampled at least every 15 min, shall be within ±1,0 °C of aim. The running average shall not include the test condition transition time of at most 1 h after operation at the test condition is initiated. Operational uniformity of the equipment at the test conditions shall be evaluated prior to the start of the test and shall be within ±2,0 °C of aim. Regions of the test chamber shall be selected to comply with the required operational uniformity conditions. If the running average of the operational fluctuation does not meet the requirement, it shall be documented and explained.

9.4 Ozone concentration

Unless otherwise specified, the test shall be carried out at 1,0 µl/l, with an operational uniformity of ±0,1 µl/l ozone. Other optional concentrations, such as 0,5 µl/l or 5,0 µl/l, may be useful for testing. If concentrations other than 1,0 µl/l are used, the operational uniformity tolerances shall be ±10 % of aim or the best achievable with the test equipment. If greater than ±10 %, the actual tolerance shall be reported. If optional concentrations are used, the tester should evaluate the materials for ozone reciprocity behaviour before making any comparative conclusions. For testing at ozone concentrations greater than 1,0 µl/l, reciprocity testing shall be conducted at an ozone concentration of 1,0 µl/l or lower (see Annex B).

NOTE Some studies have shown that ozone fade can be different under ambient conditions, with some studies showing it to be worse than predicted based on higher concentration testing[22][28].

If the tester has evaluated reciprocity behaviour, then the conditions closest to ambient shall be understood as more valid. No product comparisons shall be made between tests conducted at different ozone concentrations. For additional information on reciprocity and reciprocity testing, see Annex B.

Monitor ozone concentration as required in 8.2.3 and 8.2.4 according to the chamber design in use. The maximum deviation shall be ±4 % (operational fluctuation) at any point during the test, when sampled continuously. Cumulative time outside the tolerance range shall be less than 10 % of the test duration. If average did not meet set point, the results shall document and explain this deviation.

Before running actual tests on materials, the user shall verify that no concentration gradients exist within the chamber. This can be verified by using worst-case, high-quench-rate materials throughout the chamber and verifying uniform response throughout.

The user shall also verify that air flow within the chamber is sufficient so that the rate of change in the samples is independent of the rate of air flow. This can be done by performing preliminary testing with the samples throughout the chamber at increasingly high air flow velocities until the rate of change in the sample versus air flow is minimized. The same test set can also be used to control the overall homogeneity of the fading inside the chamber.

10 Test report

10.1 General reporting requirements

Reporting based solely on this test method shall be restricted to reporting the specific ozone fading stability test result, and shall include reporting of the ozone concentration, the ozone generation method, the percentage relative humidity, the temperature and test duration. This test method does not contain endpoint criteria and therefore the measurement results of this test method shall not be used independently to estimate or predict any aspect of print image life.

Users are cautioned that results from this test method apply only to the specific system tested. For example, in inkjet systems, a specific ink used with a specific paper may have very different results from another. Test reporting shall include this disclaimer.

10.2 Ozone test reporting

The results of these tests are reported as cumulative exposure, i.e. the product of the ozone concentration in microlitres per litre and the exposure time in hours[4] [(μl/l)·h], to reach the observed optical densities, together with the percentage losses in optical densities, or the amount of densitometric and colorimetric change observed for a given cumulative exposure.

The report of test results shall include the following items.

a) A reference to this International Standard, i.e. ISO 18941:2011.

b) The target patch encoding values of the patches selected for monitoring in the test, and the corresponding initial densities (i.e. 1,0) of the neutral and colour patches; additionally, the number of duplicate test specimens included in the test.

c) For digital output samples, the printer model, printer driver version, printer driver settings, printer front panel settings, the name of the host application used in generating the print, and the colour controls selected in that application; the cartridge configuration/ink/donor or colorant used (manufacturer’s name and part number); the paper used (manufacturer’s name and model number); and any other necessary information, such that the print file can be reproduced by another user of this International Standard. For silver-halide-based samples, the processing conditions (i.e. chemicals, procedures) shall be reported.

d) The test conditions that were actually used (i.e. the actual temperature, relative humidity, ozone generation method, test concentration and control aim level); if actual test conditions deviate from the nominal conditions specified in this International Standard, then an explanation shall be provided.

e) The duration of the test, including the extent and level to which the test was conducted. When reporting for comparison, test reporting is valid only when the test conditions have produced a fade signal (loss or gain) that can be statistically separated from test noise. Otherwise, when the test results will not be compared to other products, reporting the results from tests conducted for a cumulative exposure or test condition that does not produce can be useful.

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[4] 1 (μl/l)·h = 1 ppm-h. Although the notation “ppm-h” (parts per million-hours) 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.

f) For tests carried out above 1,0 µl/l ozone concentrations, results of reciprocity testing at lower concentrations.

g) When corona discharge equipment is used for ozone generation, the measured level of oxides of nitrogen, or the manufacturer's specification for ozone purity.

Annex A
(informative)

Method for interpolation

Often there will not be a step on a properly designed and printed test target that is of exactly the desired density. Interpolation between two neighbouring density patches can be used to predict the values for the exact desired starting density. For example, the densities of the two steps nearest to 1,0 may be used to calculate the desired density by linear interpolation. After each fading time, the same coefficient is used for interpolating the density after fading.

First, on the unfaded strip, the step with the density d_1 just below 1,0 and the step with the density d_2 just above 1,0 are chosen (see Figure A.1). After fading, these two steps will have reached the densities $d_1(t)$ and $d_2(t)$. The density $d(t)$ of the initial $d = 1{,}0$ is then estimated through linear interpolation:

$$d_t = d_{1(t)} + [d_{2(t)} - d_{1(t)}]\alpha$$

where

$$\alpha = \frac{1{,}0 - d_{1(t=0)}}{d_{2(t=0)} - d_{1(t=0)}}$$

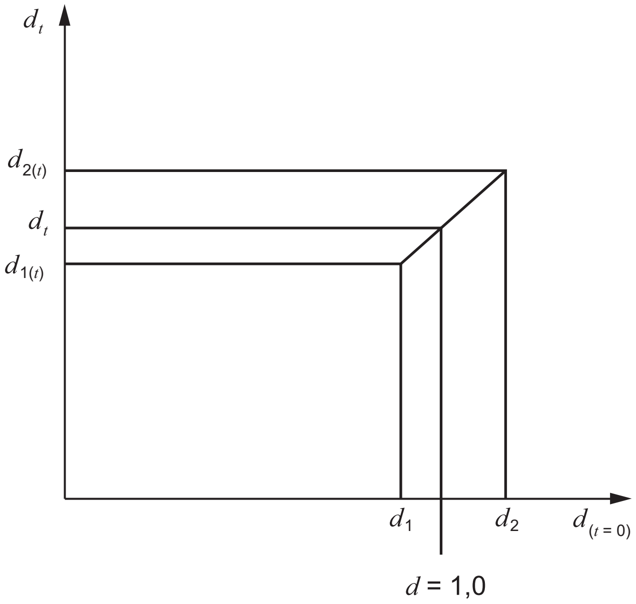


Figure A.1 — Interpolation of a test target starting density patch

Annex B
(normative)

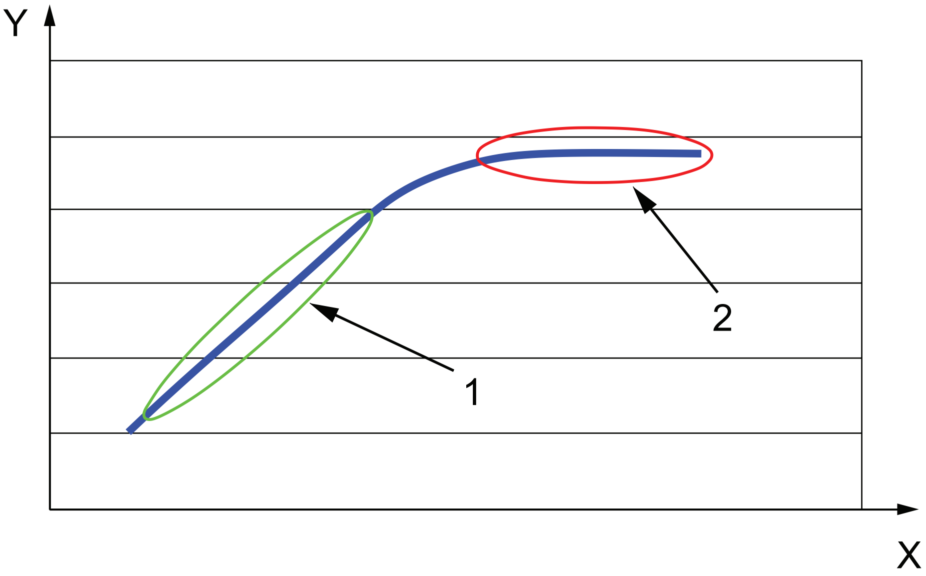
Reciprocity considerations

Reciprocal behaviour in ozone testing, or reciprocity, relies on the assumption that a given increase in the concentration of ozone will produce a proportional increase in the rate of fading, yielding equal amounts of change at equal cumulative exposures[14][17][18][22][24]. For example, if the assumed ambient environmental concentration of ozone were 10 nl/l and a product faded 10 % over a ten-year period under those conditions, then that same product would fade an equal 10 % if exposed to conditions of 100 nl/l over a one-year period.

NOTE For a discussion of the concept of reciprocity in imaging systems, see References [29] and [34].

This reciprocal response is the premise on which predictions of product performance under ambient conditions can be made from accelerated testing conditions. If a reciprocal response cannot be demonstrated or determined between two ozone exposure conditions, then product longevity predictions cannot be made with confidence or certainty[15][17][24][31].

Fade with respect to an ozone exposure at a given concentration should generally proceed as illustrated in Figure B.1.



Key

X	ozone concentration, in arbitrary units (AU)
Y	rate of fade (AU)
1	linear portion
2	insensitive portion

Figure B.1 — Conceptual relationship between ozone concentration and rate of fade

Over a practical range of ozone concentrations, an increase in concentration should yield a proportional increase in the rate of fade. This is denoted in Figure B.1 as the linear portion of the curve. However, at a finite concentration, increases in ozone levels will no longer produce an increase in the rate of fade. This is denoted in Figure B.1 as the insensitive portion of the curve. The exact nature of this curve is product-specific and potentially different for all systems[24].

Once a reaction has progressed beyond the linear portion of the curve, a reciprocal response will no longer be attainable. Since this reciprocal response is the premise on which predictions at ambient conditions can be made, if reciprocity cannot be demonstrated, any prediction would be erroneous.

A common test for reciprocity would be to select two differing concentrations, for example 0,1 µl/l and 1,0 µl/l, running each condition to a common cumulative exposure and verifying that they produce equal amounts of change. Each condition shall be run until a reliable amount of change can be measured from test system noise. It is reasonable to assume that, if a reciprocal relationship exists at these elevated conditions, the test, with respect to ozone concentration, has progressed in a reciprocal fashion through the entire range of exposure.

It is important to note here, however, that other factors within an ozone test setup may contribute to differing rates of change, potentially confounding reciprocal performance. These include, but may not be limited to, gas flow over the samples in the chamber, the nature of the photographic product and type of reaction with ozone, temperature, humidity, etc.[32].

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BSI Group Headquarters

389 Chiswick High Road London W4 4AL UK

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