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Imaging materials — Pictorial colour reflection prints — Comparison of image degradation observed between ISO 18930 accelerated weathering test method and outdoor exposure

Matériaux pour l'image — Réflexion des impressions photographiques en couleurs — Comparaison de la dégradation de l'image observée entre la méthode d'essai de vieillissement accéléré de l'ISO 18930 et l'exposition extérieure

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Foreword

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

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

Printed digital images are used in many applications in which they are exposed to outdoor weathering. ISO 18930 provides standardized test procedures to evaluate image stability both in real-time outdoor weathering tests and in accelerated laboratory simulations of the weathering process. Accelerated laboratory weathering tests have been developed as a result of the desire to obtain test results faster than would be obtained by actual outdoor exposure. However, accelerated weathering tests only have value if they can be correlated with actual outdoor performance.

Imaging materials — Pictorial colour reflection prints — Comparison of image degradation observed between ISO 18930 accelerated weathering test method and outdoor exposure

1 Scope

This document describes the experimental framework, results, and conclusions from a round robin test that was performed in order to establish correlations between accelerated weathering according to the ISO 18930 test method and outdoor weathering at nine outdoor sites.

The types of digital printing technology that were used in this round robin test are aqueous inkjet, solvent inkjet, UV curable inkjet, digitally-exposed silver halide, and thermal mass transfer. The image print stability data and correlations of this document are to be considered illustrative of the performance of these classes of materials. Extension of these correlations to other classes of materials, such as dye sublimation, is verified by appropriate experimentation.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions 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
digital printing media
recording elements used by digital printers to receive inks or pre-formed colourants

EXAMPLE The substrate may be paper, plastic, canvas, fabric, metal, or other ink-receptive material; the substrate may, or may not, be coated with an ink-receptive layer. The category of digital printers includes inkjet, electrophotographic, and thermal transfer.

3.2
laminate
overlaminate
layer of material that goes over the top or bottom of a specimen

Note 1 to entry: Usually to provide water-resistance, physical, and/or ultraviolet (UV) light protection of the specimen during a weathering test. A layer of protective film is applied with a pressure-sensitive or heat-activated adhesive.

3.3
accelerated laboratory weathering
simulated weathering where instruments (weathering devices) are used to obtain very controlled conditions that simulate, to some degree, and generally accelerate, the outdoor weathering results

Note 1 to entry: The use of such instruments is described in ISO 4892-1[2] and ASTM G151[16].

3.4
outdoor weathering
actual placement of specimens outdoors in specific locations

Note 1 to entry: This is differentiated from simulated weathering where instruments (weathering devices) are used to obtain very controlled conditions that simulate, to some degree, and generally accelerate the outdoor weathering results. Use of such instruments is described in ISO 4892-1[2] and ASTM G151[16].

3.5
reciprocity failure
non-equivalence in weathering results between a long exposure/low-intensity experiment and its short exposure/high-intensity counterpart with an equivalent intensity-time product

3.6
daylight filter
optical filter or combination of filters that modifies the spectral power distribution of a light source to better represent some defined daylight spectrum

Note 1 to entry: These filters are not related to the blue filters used in the photographic industry for the change of correlated colour temperature of light sources.

Note 2 to entry: Adapted from ISO 18913[5].

3.7
coefficient of variation
standard deviation of a variable divided by the arithmetic mean of the variable

3.8
Pearson correlation coefficient
statistical measure of the degree of linear correlation between two variables, with value between −1,0 and +1,0 inclusive, where a value of +1,0 represents perfect positive correlation, a value of 0,0 signifies no correlation, and a value of −1,0 represents perfect negative correlation

3.9
acceleration factor
ratio of the time required to reach an endpoint in an outdoor weathering test to the time required to reach the same endpoint in a laboratory accelerated weathering test

3.10
colour fade acceleration factor
acceleration factor for which the bases of comparison are the ratios of reflected optical density during the test to the initial reflected optical density prior to the start of the test

4 General considerations for accelerated weathering tests

The ability to accurately predict the long-term outdoor performance of materials and printed images is essential to many industries. Since many of the relevant products are designed to last years or decades, accelerated weathering test methods have been developed to more rapidly assess outdoor performance and to investigate failure mechanisms associated with outdoor exposure. Unfortunately, this is an extremely complex task.

The three key components of accelerated weathering tests are heat, light, and water. The primary determinant of the degree of correlation for between outdoor weathering and an accelerated test method is the degree to which the spectral power distribution (SPD) of the light source in the test chamber matches the SPD of sunlight[7]. This is so critical because material photodegradation mechanisms are very specific to certain wavelengths of light[8]. The UV spectrum between 295 nm and 400 nm is responsible for most of the damage to polymers and colourants. The current state-of-the-art light source is filtered xenon arc lamps. In a comprehensive study of the accelerated weathering of polyester gel coats, Crump[9] found that xenon arc weathering gave higher correlation coefficients than methods employing carbon arc or fluorescent light sources. Previous investigations by Klemann[10][11] also indicated high correlation coefficients for xenon arc light sources.

Water exposure is also essential because many materials exhibit hydrolytic degradation pathways. Heat, in terms of elevated chamber temperatures, is used to accelerate all of the reactions that contribute to material and image degradation. Other factors such as ozone, pollutants, freeze-thaw cycles, and abrasion due to airborne particles may also affect material longevity, but are not included in most accelerated test cycles.

Two metrics are used to gauge the efficacy of accelerated weathering test methods: the acceleration factor and the Pearson correlation coefficient. An acceleration factor is a scale factor that relates the rate of degradation in an accelerated test to the rate of degradation in real-time outdoor exposure. For example, if a colour patch fades by 40 % over one year on an outdoor rack in South Florida and also fades 40 % after 1 month of an accelerated weathering test, then the acceleration factor would be 12, as one month of accelerated testing is equivalent to 12 months of outdoor testing. The correlation coefficient is the degree to which, and the consistency of, the agreement between accelerated and outdoor testing.

The user of any accelerated weathering method should be cautioned that the acceleration factors are specific to both the outdoor location and to the material, or combination of materials, that are tested. It should be obvious that acceleration factors depend upon the climate of the outdoor site. Average radiant exposure, rainfall, relative humidity, and temperatures of an outdoor location all affect the acceleration factor. Indeed, even year to year climatic variations will change the acceleration factor to some degree. What may be less obvious is that there are also some differences in acceleration factor for different materials. This is due to the different photodegradation mechanisms and their wavelength specificity, to the rates of water absorption and the saturation moisture levels, and to any changes in degradation mechanisms as a function of temperature (for example, outdoor conditions are below a polymer glass transition temperature and the temperature of an accelerated weathering test is above it). An investigation of fade of colour patches on signs and labels showed that that the average acceleration factor for a set location may vary as much as ±50 % by material construction[10].

NOTE If use of an acceleration factor is desired in spite of the warnings given in this document, such acceleration factors for a particular material are only valid if they are based on data from a sufficient number of separate exterior or indoor environmental tests and accelerated laboratory exposures so that results used to relate times to failure in each exposure can be analysed using statistical methods, see ISO 4892-1.

No standard accelerated weathering test method results in a perfect correlation with outdoor performance. ASTM G155[17], Cycle 1, and its predecessor ASTM G26, uses one or more xenon lamps with borosilicate type S inner and outer filters, which gives an excellent approximation for the SPD of sunlight, and has a periodic water spray, but is an isothermal test. SAE J2527[12] test cycle and its predecessor SAE J1960 both include segments with high temperatures and a segment with lower temperature, water spray, and no light, to simulate night. For some materials that are sensitive to expansion and contraction, or to the stresses of drying while heating, this type of day-night cycle may give more realistic results. However, the quartz inner/borosilicate outer filter combination of these SAE tests exposes samples to light in the 280 nm to 295 nm range that would be screened out by the earth's ozone layer outdoors.

To improve upon previous accelerated weathering standards, the ISO 18930 test method was developed. It was confirmed in 2011. The light source SPD (see Annex A) is specified in terms of spectral output by 10 nm or 20 nm bands of wavelengths so as to provide a best match to the SPD of sunlight in CIE 85:1989[18], Table 4. Four cycle segments are incorporated: three at high black panel and chamber temperatures with light exposure, and one at lower temperature in the dark. Water spray is included for one of the high-temperature segments and for the cool, dark cycle segment (Table 1). The International Standard requires that testing be conducted at a 45° angle of inclination, although other angles of inclination may be added, as this maximizes the solar irradiance received by the samples[13].

Table 1 — ISO 18930 xenon arc exposure test cycle

Cycle Segment	Time (min)	Irradiance – Narrowband (340 nm) W/m²	Irradiance – Broadband (300 to 400 nm) W/m²	Black Panel Temperature °C	Chamber Temperature °C	Relative Humidity %	Water Spray
1	40	0,55 ± 0,02	60 ± 2	63 ± 2	40 ± 2	50 ± 6	None
2	20	0,55 ± 0,02	60 ± 2	—	40 ± 2	—	Front
3	60	0,55 ± 0,02	60 ± 2	63 ± 2	40 ± 2	50 ± 6	None
4	60	0,00	0	—	38 ± 2	—	Front

This paper describes the details and results of a round-robin study with nine outdoor global locations and six laboratories running ISO 18930 in order to validate the new test method.

5 Materials

This investigation encompassed 32 material/ink combinations and digital printing technologies. Technologies represented included aqueous inkjet, solvent inkjet, UV inkjet, digital silver halide, thermal transfer, and for comparison, flexography. Some were overlaminated, others remained directly exposed to the elements. For all materials, two replicates of the target below were printed. The target has six patches each varying in lightness for cyan, magenta, yellow, true black, red, green, blue, and process black (CMY) see Figure 1. Two small white patches were included for measurements of material yellowing, and large black and white patches were added below for gloss measurements.

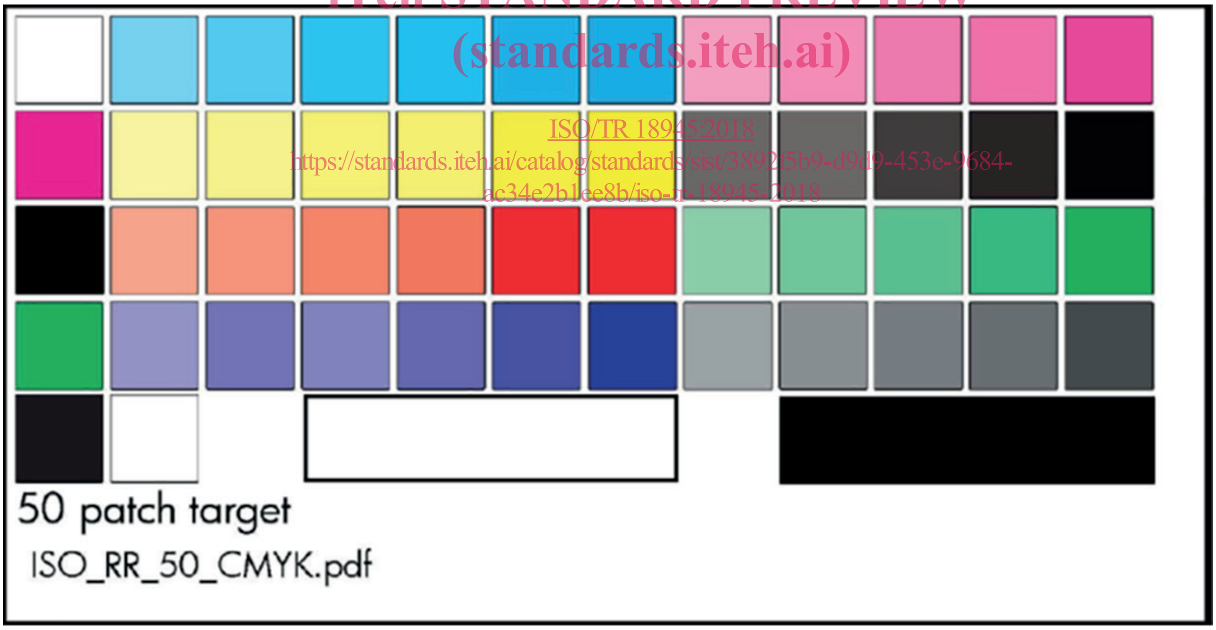

Figure 1 — ISO 18930 Round Robin Test Target

Two replicates each of the test targets were printed for nine outdoor sites and six accelerated test instruments running ISO 18930 (see Table 1). After printing, the samples were maintained at 23 °C and 50 % relative humidity until the start of the tests.

6 Test methods

6.1 Outdoor exposure tests

Both accredited and non-accredited outdoor sites were included in this investigation (see Table 2). Printed test targets were mounted on aluminum panels. These outdoor panels were placed on racks at a 45° angle of inclination, south facing. For a comparison of exposures at angles of inclination of 45° and 90°, see Annex E. Measurements of the colour patches were taken at 0 year, 1 years and 2 years for the outdoor sites.

Table 2 — Outdoor test site climate data

Site	Latitude	Radiant Exposure MJ/m2/y	Precipitation mm	Average Temperature °C	Accredited Lab
South Florida, USA	25,87° N	6 588	1 655	23	YES
San Diego, CA USA	33,03° N	6 602	262	18	NO
DSET, Arizona, USA	33,90° N	8 004	255	22	YES
Tokyo, Japan	35,71° N	4 959	1 682	14	YES
Chicago, IL USA	41,78° N	5 100	856	10	YES
Sanary, France	43,13° N	5 500	700	13	YES
Milwaukee, WI USA	43,14° N	5 103	884	9	NO
Marly, Switzerland	46,78° N	4 590	1 075	9	NO
Mortsel, Belgium	51,17° N	3 708	825	10	NO

6.2 Laboratory accelerated weathering tests

All accelerated weathering instruments were set for Borosilicate Type S inner/Borosilicate Type S outer, Daylight Q, Daylight B/B, Quartz/#295, or other combinations appropriate to match the SPD requirements associated with ISO 18930. Colour measurements were taken after 0 h, 24 h, 200 h, 400 h, 800 h, 1 200 h, 1 600 h and 2 000 h of exposure to the ISO 18930 test cycle (see Table 1) for all accelerated testing chambers. For some of the chambers, the testing time was increased to as long as 5 200 h of exposure. For all colour measurements 45°/0° geometry, a 10° observer, and D65 illuminant were specified. Spectrophotometer data was converted to reflected optical densities according to the ANSI Status A Standard for densitometer filters.

6.3 Data analysis and work-up

The procedure for data analysis employed optical density ratios – the ratio of optical density to initial optical density. For primary colours only a single density ratio was tracked. For secondary colours two density ratios were tracked, and for the process black patches all four densities (C, M, Y, and K or D_{VIS}) were tracked. For the secondary and process patches, the difference in density ratios for the relevant densities were also tracked as a measure of colour shifts. For the two white patches, ΔE76 was measured to evaluate substrate yellowing.

For each outdoor site after one year of exposure the test targets were measured with the spectrophotometer. Density ratios were calculated and used on a patch by patch basis. To find the acceleration factor for a single patch in an accelerated weathering chamber, the number of hours needed to obtain the same density as that of the outdoor site were determined via linear interpolation of the accelerated colour data. The acceleration factor is then calculated as 8 766 h (one year) divided by the number of hours in ISO 18930 that gives the same density ratio. The 48 patch acceleration factors on a target could then be used for statistical comparisons by material, accelerated testing laboratory, outdoor site, print technology, etc. Only density ratios between 0,95 and 0,30 were used for analysis, as it was thought that less than a 5 % density loss did not give a large enough signal to noise ratio, and that degradation would slow down or even reach an asymptote at density ratios less than 0,30.

7 Results and discussion

7.1 Colour Fade Acceleration Factors

Acceleration factors were calculated for colour fade, colour shifts, and for background yellowing. For reasons that will be specified later, colour fade acceleration factors were found to be the most useful output of the study.

Initially, the accelerated ISO 18930 tests were scheduled to run only 2 000 h. However, it was soon determined that this test duration was insufficient, especially when correlating to the more aggressive climates of South Florida, Arizona, and San Diego. This was found to be critical in the determination of correct acceleration factors. Not all 48 patches on a test target yield useful data points, and these data points are first available when the patch on the accelerated test target reaches the same density ratio as the outdoor test patch. There are two possibilities for missing data points:

a) The outdoor test patch has a density ratio above 0,95 or below 0,30 and is excluded from analysis;

b) The outdoor test patch is in the correct density ratio range, but the accelerated test has not been run long enough to reach that density ratios.

In Case 1, the data points will never be available. For Case 2, however, more data points come in as the length of the accelerated test is extended. This causes the apparent acceleration factor to decrease over time until all of the Case 2 points come in and the apparent acceleration factor converges to the true acceleration factor. An example of this is shown in Table 3 for South Florida, one of the most aggressive climates.

Table 3 — Change in apparent acceleration factor as more accelerated test data is collected

Colour Patch Fade Data for South Florida Test Site			
Hours of Accelerated Testing ISO 18930	**Percentage of Maximum Data Points Available**	**Apparent Acceleration Factor for 1 Year Outdoors**	**Hours of Accelerated Testing ISO 18930**
2 000	9	7,77	2 000
4 200	51	4,34	4 200
5 200	56	4,13	5 200

After accelerated testing was extended to 5 200 h to ensure that all of the obtainable data points were collected, true acceleration factors could be determined for all nine outdoor sites. The average acceleration factors for the 32 materials are shown in Table 4. As would be expected, the most aggressive climates show smaller acceleration factors than the sites farther north; the trends intuitively seem to make sense. The differences between the highest and lowest acceleration factors also scale with results of previous studies that indicated approximately a factor of two ratio between South Florida and sites with latitude of 42 N to 55°N[10][14].

Table 4 — Colour fade acceleration factors (AF) by site for 1 y outdoor exposure

	Arizona	**Chicago**	**Sanary, FRA**	**South Florida**	**Milwaukee**	**San Diego**	**Tokyo, JP**	**Mortsel, BEL**	**Marly, CH**
AF Colour Fade – 1 year	**4,84**	**7,38**	**6,13**	**4,13**	**7,47**	**5,61**	**8,28**	**8,22**	**8,49**
Material Stdev – 1 year	1,91	3,17	2,19	2,53	2,96	2,00	3,38	4,06	3,85
% Data Points Available	50	54	62	56	59	57	49	63	58

7.2 Replicability of data

Consistency of the data is evaluated on a lab-lab basis in Table 5, for which the standard deviations compare the acceleration factors for data points at a given lab to the average acceleration factor for that material and outdoor location for labs 3 to 5. Note that only Labs 3, 4, and 5 were included, because the other labs had different accelerated test durations. Replicability is evaluated in Table 6, for which the standard deviations for the two replicates per lab are compared. In both Tables the standard deviations were normalized to the coefficient of variation (standard deviation/average) for comparison. The average coefficients of variation were 13,5 % and 14,6 % for lab-lab and replicate comparisons, respectively. Since the lab-lab variation is barely higher that the variation of replicates in the same instrument, in may be inferred that the standard test method is barely affected by changes of the test instrument, as long as it is capable of meeting the specifications stipulated in the test method standard. It is also seen that the variations are a bit lower for the more aggressive climates than for the higher latitudes. This will be discussed later with the correlation coefficients.

Table 5 — Lab-lab coefficient of variation for colour fade failure hours

Outdoor Site	**Lab 3**	**Lab 4**	**Lab 5**	**Average**
DSET Arizona	0,084	0,108	0,112	0,106
Chicago	0,131	0,125	0,172	0,146
Sanary, France	0,119	0,121	0,142	0,130
South Florida		0,064	0,087	0,063
Milwaukee, Wis.	0,171	0,219	0,170	0,191
San Diego	0,124	0,133	0,175	0,149
Tokyo, Japan	0,175	0,144	0,194	0,170
Mortsel, Belgium	0,175	0,156	0,215	0,184
Marly, Switzerland	0,114	0,157	0,165	0,153
Overall Average	**0,137**	**0,136**	**0,159**	**0,146**

Table 6 — Replicate coefficient of variation for colour fade failure hours

Outdoor Site	Lab 1	Lab 2	Lab 3	Lab 4	Lab 5	Lab 6	All Labs
DSET Arizona	0,117	0,174	0,061	0,123	0,105	0,136	0,139
Chicago	0,130	0,141	0,076	0,114	0,128	0,180	0,137
Sanary, France	0,114	0,106	0,073	0,102	0,109	0,152	0,114
South Florida	0,103	0,164	0,061	0,104	0,102	0,152	0,127
Milwaukee, Wis.	0,139	0,137	0,071	0,292	0,141	0,179	0,163
San Diego	0,156	0,142	0,066	0,093	0,135	0,169	0,136
Tokyo, Japan	0,125	0,127	0,085	0,102	0,135	0,168	0,128
Mortsel, Belgium	0,154	0,122	0,076	0,116	0,086	0,156	0,132
Marly, Switzerland	0,164	0,160	0,078	0,148	0,141	0,189	0,156
Overall Avg.	**0,130**	**0,140**	**0,074**	**0,126**	**0,125**	**0,164**	**0,135**

7.3 Applicability to multiple digital printing technologies

The scope of ISO 18930 covers all digital printing. Since standards are generally developed to have universal applicability across large classes of materials and technologies, materials and inks from five digital printing technologies plus analog flexography were included in the round robin test. Table 7 shows a comparison of the acceleration factors for the different groups and confidence intervals of ±2σ around the averages. It is observed that the confidence intervals for the print/ink technologies are larger than the differences between the averages of the technologies. So, with this small data set, it is not possible to say that any of the print technologies show statistically significant differences. Indeed, the only one for which that looks possible is digitally-exposed silver halide.

Table 7 — Colour fade acceleration factors by printing technology

Print Technology	Average AF	Stdev AF	Lower Confidence Interval	Upper Confidence Interval	Materials
Aqueous Inkjet	5,32	2,22	0,88	9,76	11
UV Inkjet	4,91	1,46	1,99	7,83	8
Solvent Inkjet	5,83	2,24	1,35	10,31	6
Digital Silver Halide	8,80	1,94	4,92	12,68	3
Thermal Mass Transfer	5,21	0,99	3,23	7,19	2
Flexography	5,96	—	—	—	2

Table 8 — Classification of test materials by print technology

Print Technology	Number of Materials Tested	Materials without Protective Layers	Materials with Pressure-Sensitive Overlaminates	Materials with Liquid Clear Coats	Inkjet Materials	
					Piezoelectric Printheads	Thermal Printheads
Aqueous Inkjet	11	8	3	0	7	4
UV Inkjet	8	7	0	1	8	0
Solvent Inkjet	6	3	3	3	6	0
Digital Silver Halide	3	0	3	0	—	—
Thermal Mass Transfer	2	1	1	0	—	—
Flexography	2	1	1	0	—	—

7.4 Effects of colour and patch darkness

The use of six patches for each colour of ascending colour density on the test target makes it simple to break down the results by colour and patch darkness. The surprise here is that the average correlation coefficient did vary to some extent with those two factors. While the rest of the colours are within 10 % of the overall average, yellow patches faded 22 % faster than the average for all colours. There is also a slight increase in acceleration factor as the patches in a series get lighter (from 6 to 1 in the target). However, no satisfactory explanation for this behaviour has been proposed. Indeed, it is not known whether these results are due to a few materials in a small data set, or whether they signify a real phenomenon. See G.5 for an example of several materials for which the Tokyo data showed very high acceleration factors, but the South Florida data was very close to the overall average acceleration factor.

Table 9 — Average colour fade AF by colour

Colour	Average AF	AF as % of Overall Average
Cyan	5,25	80,8
Magenta	6,19	95,4
Yellow	7,97	122,7
Black (K)	5,96	91,7
Red	6,95	107,0
Green	6,67	102,8
Blue	6,11	94,2
Process Black (CMY)	6,57	101,2
Overall Average	6,49	100,0

Table 10 — Average colour fade AF by patch darkness

Patch	Average AF	AF as % of Overall Average
1	6,85	105,5
2	6,75	103,9
3	6,61	101,8
4	6,55	100,9
5	6,11	94,1
6	6,03	92,8
Overall Average	6,49	

7.5 Analysis of colour shifts

Attempts to generate correlation coefficients for colour shifts were less successful than the efforts for colour fade. A minimum shift in colour balance of 5 % was used as a threshold for inclusion in this data set. However, ink sets are often formulated with a mind to minimize any colour shifts, so several of the ink sets tested were so well balanced that none of the patches on their targets showed a 5 % colour shift. For example, for Mortsel, Belgium, the least aggressive climate in this study, only 8,1 % of the possible data points were available. A greater concern is the life cycle of a colour shift during a weathering test. The colour balance shift is zero both initially and when the colourants have all faded to white after severe degradation. At some point in between, the colour balance shift reaches a maximum. For the accelerated data, which is taken frequently, it is easy to see which side of the maximum the data is at. For one year outdoor data, on the other hand, it is very hard to determine whether a data point is before or after the maximum colour shift. If it is before the maximum, then a correct data point will be obtained. If it is after the maximum, then a false data point will be obtained with an acceleration factor that is higher than the correct data point. This effect skews the data unless one has the ability to