ISO 18930 - A New Standard Test Method for Accelerated Weathering

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Abstract

ISO 18930 was confirmed in 2011 as a new International Standard test method for accelerated weathering. It is a four-segment test cycle that simulates day, night, rain, and condensation. The spectral power distribution of the light source was created based upon a tolerance band around the CIE 85 spectral power distribution of sunlight. A round-robin test has been conducted to validate the new method. This is the first data published to substantiate the use of ISO 18930.

Acceleration factors and Pearson correlation coefficients are presented for nine worldwide outdoor locations, along with examinations of lab-to-lab and replicate reproducibility. The results validate that ISO 18930 is a valid and versatile accelerated weathering test method that is applicable to a large range of materials. Due to the dark cycle segment with water spray followed by a light cycle segment at elevated temperature, it is believed that this cycle may perform better than ASTM G155, Cycle 1 for some materials that are brittle, porous, or hygroscopic. ISO TC42 WG-5 has also recommended that an ISO Technical Report with a detailed data analysis be written to document the results of this round robin test.

Introduction

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 [1]. This is so critical because material photodegradation mechanisms are very specific to certain wavelengths of light [2]. The UV spectrum between 295 and 400 nm is responsible for most of the damage to polymers and colorants. 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 [3] found that xenon arc weathering gave higher correlation coefficients than methods employing carbon arc or fluorescent light sources. Previous investigations by the author [4,5] also indicated high correlation coefficients for xenon arc light sources.

Water exposure is also essential because many materials exhibit hydrolytic degradation pathways. Heat, in term 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 color 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 equal 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.

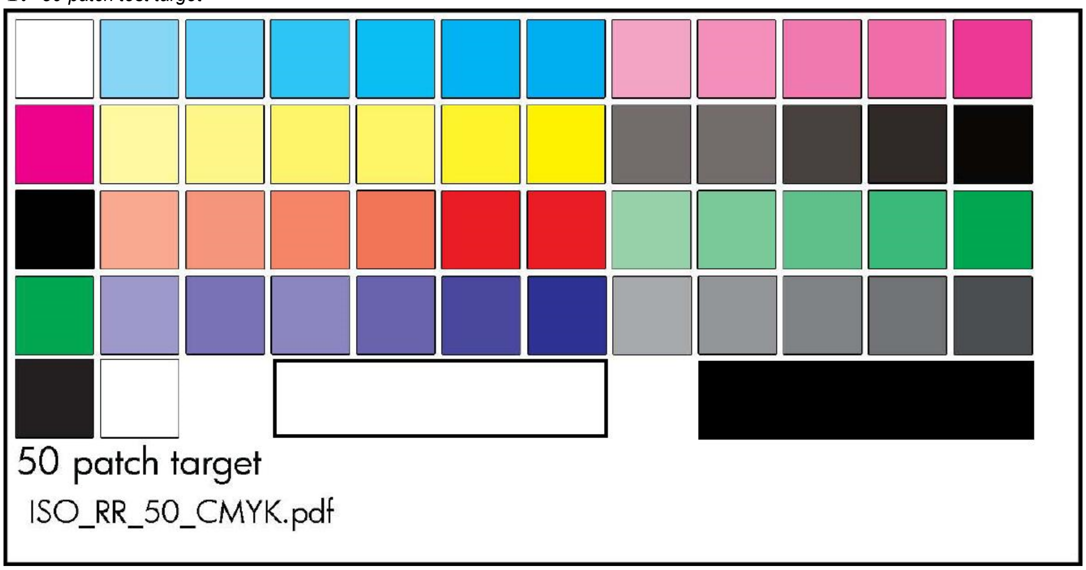
The user of any accelerated weathering method should be cautioned that the acceleration factors are specific to both the outdoor location and to the material, or combination of materials, that are tested. It should be obvious that acceleration factors depend upon the climate of the outdoor site. Average 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 color 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 [4].

No standard accelerated weathering test method results in a perfect correlation with outdoor performance. ASTM G155, Cycle 1 [6] (and its predecessor ASTM G26) uses a 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 [7] 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 the 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 – 295 nm range that would be screened out by the earth's ozone layer outdoors.

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

Figure 1: *50-patch test target*



To improve upon previous accelerated weathering standards, the ISO 18930 International Standard test method was developed [8]. It was confirmed in 2012. The light source SPD 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 Publication 85, Table 4 [9]. 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 [10].

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.

Materials and Methods

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). Two small white patches were included for measurements of material yellowing, and large black and white patches were added below for gloss measurements.

Two replicates each of the test targets were printed for nine outdoor sites and six accelerated test instruments running ISO 18930 (see Table 1). Both accredited and non-accredited outdoor sites were included (Table 2). After printing the samples were maintained at 23°C and 50% relative humidity until the start of the 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. The outdoor panels were placed on racks at a 45° angle of inclination, south facing.

Measurements of the color patches were taken at 0, 1, and 2 years for the outdoor sites. For all accelerated testing chambers color measurements were taken at 0, 24, 200, 400, 800, 1200, 1600, and 2000 hr. For some of them, the testing time was increased to as long as 5200 hr of exposure. For all color 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.

The procedure for data analysis employed optical density ratios – the ratio of optical density to initial optical density. For primary colors only a single density ratio was tracked. For secondary colors 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 color shifts. For the two white patches, ΔE_{76} 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 color data. The acceleration factor is then calculated as 8766 hr (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.

Table 2: Outdoor Test Site Climate Data

Site	Latitude	Radiant Exposure (MJ/m^2)	Precip. (mm)	Avg Temp. (°C)
South Florida, USA	25.87° N	6588	1655	23
San Diego, CA USA	33.03° N	6602	262	18
DSET, Arizona, USA	33.90° N	8004	255	22
Tokyo, Japan	35.71° N	4959	1682	14
Chicago, IL USA	41.78° N	5100	856	10
Sanary, France	43.13° N	5500	700	13
Milwaukee, WI USA	43.14° N	5103	884	9
Marly, Switzerland	46.78° N	4590	1075	9
Mortsel, Belgium	51.17° N	3708	825	10

Results and Discussion

Color Fade Acceleration Factors

Acceleration factors were calculated for color fade, color shifts, and for background yellowing. For reasons that will be specified later, color 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 2000 hr. 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: 1) The outdoor test patch has a density ratio above 0.95 or below 0.30 and is excluded from analysis; 2) 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 – South Florida Test Site

Hours of Accelerated Testing ISO 18930	Percentage of Maximum Data Points Available	Apparent Acceleration Factor - 1 Yr Outdoors	Hours of Accelerated Testing ISO 18930
2000	9	7.77	2000
4200	51	4.34	4200
5200	56	4.13	5200

After accelerated testing was extended to 5200 hr 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 – 55°N [5,11,12].

Table 4: Color Fade Acceleration Factors (AF) by Site for 1 Year Outdoor Exposure

	Arizona	Chicago	Sanary, FRA	South Florida	Milwaukee	San Diego	Tokyo, JP	Mortsel, BEL	Marly, CH
Color Fade AF – 1 yr	4.84	7.38	6.13	4.13	7.47	5.61	8.28	8.22	8.49
Material AF Stdev	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

Replicability of Data

Consistency of the data was evaluated on lab-lab (Table 5) and replicate (Table 6) bases. In both cases it was normalized to the coefficient of variation (standard deviation / average) for comparison. Note that only Labs 3, 4, and 5 were included in

Table 5 because the other labs had different accelerated test durations. 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 Color 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 Color Fade Failure Hours

Outdoor Site	Lab 1	Lab 2	Lab 3	Lab 4	Lab 5	Lab 6	Avg.
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, FR	0.114	0.106	0.073	0.102	0.109	0.152	0.114
S. Florida	0.103	0.164	0.061	0.104	0.102	0.152	0.127
Milwaukee	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, JP	0.125	0.127	0.085	0.102	0.135	0.168	0.128
Mortsel, BE	0.154	0.122	0.076	0.116	0.086	0.156	0.132
Marly, CH	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

Applicability to Multiple 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: Color Fade Acceleration Factors (AF) and Confidence Intervals (CI) by Printing Technology

Print Technology	Avg AF	Stdev AF	Lower CI	Upper CI	Materials
Aqueous Inkjet	5.32	2.22	0.88	9.76	11
Solvent Inkjet	5.83	2.24	1.35	10.31	6
UV Inkjet	4.43	1.46	1.51	7.35	6
Digital AgX	8.80	1.94	4.92	12.68	3
Thermal Mass Transfer	5.21	0.99	3.23	7.19	2
Flexography	5.96	-----	-----	-----	1

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.

Analysis of Color Shifts

Attempts to generate correlation coefficients for color shifts were less successful than the efforts for color fade. A minimum shift in color 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 color shifts, so several of the ink sets tested were so well balanced that none of the patches on their targets showed a 5% color 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 color shift during a weathering test. The color balance shift is zero both initially and when the colorants have all faded to white after severe degradation. At some point in between, the color 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 color 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 monitor each patch on the outdoor samples and to determine when the maximum color shift occurs. It also explains the large standard deviations for the color shift acceleration factors.

Correlation Coefficients and Predictive Correlations

The Pearson correlation coefficients for color fade acceleration factors are shown in Table 8. The overall average of 0.677 compares well with xenon arc weathering results from Crump [3], Klemann [4], and Bauer [11,12]. It is also observed that the correlation coefficients are highest for the sites at the lowest latitudes. Again, these sites also showed lower coefficients of variation for their acceleration factors.

Table 8: R-Squared Pearson Correlation Coefficients

Outdoor Site	R^2 Correlation Coefficient
DSET Arizona	0.736
Chicago	0.636
Sanary, France	0.708
South Florida	0.772
Milwaukee, Wis.	0.637
San Diego	0.723
Tokyo, Japan	0.606
Mortsel, Belgium	0.631
Marly, Switzerland	0.635
Overall Avg.	0.677

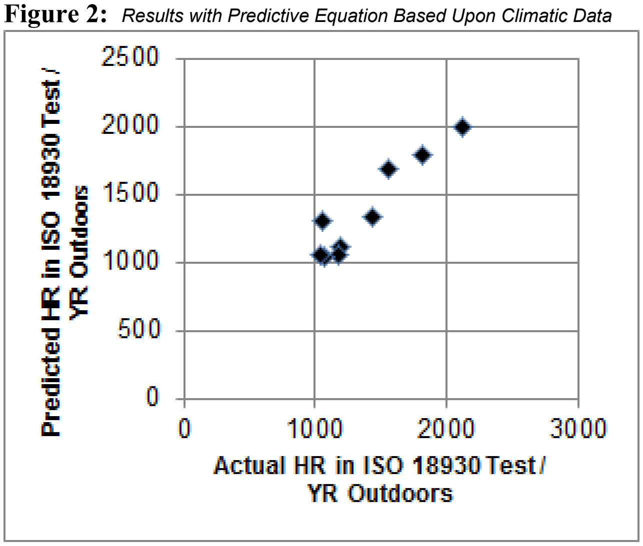
In order to predict acceleration factors for local climates, the relationships between annual climatic parameters and accelerations factors was explored. Ideally, one would like to select annual climatic parameters that represent the effects of the three key factors that drive degradation: light, water, and heat. If these parameters correlate strongly enough with the 18930 accelerated test, it would then be possible simply to look at the relevant climatic data for a site to predict an acceleration factor before running any tests on printed materials. In order to select the appropriate parameters, a series of variables for the nine outdoor sites were plotted against both acceleration factor and its reciprocal, the number of hours in ISO 18930 per year outdoors (Table 9). Average annual temperature has the highest correlation coefficient, so it is selected as representative of thermal effects. Annual solar radiant exposure also correlates well with acceleration factor, so it may be selected as the proxy for light exposure. The surprise here is how poorly the variables representative of moisture correlate; annual precipitation has almost no relationship at all with the acceleration factor. The best climatic parameter related to moisture is average annual dew point, which still has R^2 below 0.5.

When the selected parameters are plotted versus the acceleration factor to establish appropriate exponents, and then combined together into an equation, the results are excellent. The predictive equation for hours in ISO 18930 testing per year outdoors shows an R^2 value of 0.89. This exercise also uncovers a reason why the correlation coefficients are larger for the low-latitude, aggressive climates. The aggressive climates are characterized primarily by radiant exposure and temperature. They can be very humid, like South Florida, or very dry, like Arizona. ISO 18930, and most other accelerated weathering test cycles, increases the dosage of light and heat more than that of water. So, low-latitude test sites are more similar to ISO 18930 test conditions and correlate more strongly with it than high-latitude sites.

Table 9: Variable Selection for Predictive Correlations

Dependent Variable	Independent Variable or Equation	R^2 Correlation Coefficient
HR in ISO 18930 / YR Outdoors	Annual solar radiant exposure (GHI)	0.70
Acceleration Factor	Annual solar radiant exposure (GHI)	0.76
HR in ISO 18930 / YR Outdoors	Average annual temperature	0.85
Acceleration Factor	Average annual temperature	0.80
HR in ISO 18930 / YR Outdoors	Annual precipitation	0.01
Acceleration Factor	Annual precipitation	0.05
HR in ISO 18930 / YR Outdoors	Average relative humidity (RH)	0.17
Acceleration Factor	Average relative humidity (RH)	0.25
HR in ISO 18930 / YR Outdoors	Average annual dew point (TDP)	0.40
Acceleration Factor	Average annual dew point (TDP)	0.32
HR in ISO 18930 / YR Outdoors	Latitude	0.67
Acceleration Factor	Latitude	0.62
HR in ISO 18930 / YR Outdoors	$(GHI)^{0.973}(AT)^{0.618}(TDP)^{0.210}$	0.89

Figure 2: *Results with Predictive Equation Based Upon Climatic Data*



Conclusions and Recommendations

It is concluded that ISO 18930 is a valid and versatile accelerated weathering test method that is applicable to a large range of materials and digital printing technologies. Due to the dark cycle segment with water spray followed by a light cycle segment at elevated temperature, it is believed that this cycle will perform better than ASTM G155, Cycle 1 for materials that are brittle, porous, or hygroscopic. The similarity of the variance in acceleration factors for both replicates in the same lab and lab-to-lab comparisons indicates that any laboratories that run the appropriate test conditions should obtain similar results. The correlation coefficients for color fade acceleration factors compare well with other, previously published, studies of xenon-arc-based accelerated weathering tests.

It is recommended that the user of the ISO 18930 test method use the one year color fade acceleration factors published here as starting points for the prediction of printed image longevity in the nine locations that were part of this round robin test. The color shift acceleration factors from this round robin test are not considered to be reliable, but the authors believe that this type of acceleration factor could easily be derived from a similar test in which more frequent outdoor sample color measurements were taken so as to accurately determine the exposure at which the maximum color shift occurred.

For future work, it is recommended that more printing technologies, and more representative materials and inks for each technology be tested. In addition to color fade and color shifts, an extensive investigation of the changes in gloss is suggested, especially for products without pressure-sensitive overlaminates; this would be particularly beneficial for comparisons with published investigations of weathering in coatings, paints, and plastics that have employed gloss changes as the metric. A longer-term correlation study, on order of 5 – 10 years, is advised to determine whether or not any reciprocity failures occur with this test, and to estimate their magnitude.

The ISO TC42 WG-5 / TG-3 image permanence committee has recommended that, in addition to this publication in IS&T NIP, a more complete analysis of this round robin test be published as an ISO Technical Report.

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