Haltbarkeitstests basierend auf den aktuellen Untersuchungen zu umweltbedingter Bildalterung

Diplomarbeit am
Institut für Medien- und Phototechnik
der Fachhochschule Köln

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Permanence testing based on current developments of image deterioration due to environmental impacts

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Zusammenfassung

Titel: Haltbarkeitstests basierend auf den aktuellen Untersuchungen zu umweltbedingter Bildalterung

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Zusammenfassung: In den letzten Jahren wurden immer mehr Bilder digital produziert. Verschiedene Ausgabegeräte und eine Vielzahl qualitativ unterschiedlicher Druckmaterialien ersetzten immer mehr die traditionelle Silberhalogenid-Fotografie. Daher werden Vergleiche bezüglich der Haltbarkeit immer wichtiger, um Vorzüge und Schwächen aufzuzeigen. Diese Arbeit handelt von den wichtigsten Einflussgrößen, die die Bildalterung hervorrufen: Licht, Ozon, Temperatur und Feuchte. Bestehende Testmethoden konnten verbessert werden, um die durchschnittlichen Innenraumbedingungen besser zu simulieren. Die Ergebnisse bestätigen die Lebenserwartung einer Auswahl an Materialien.

Stichwörter: Stabilität, Innenraum Bedingungen, Licht, Ozon, Ink Jet

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Abstract

Title: Permanence testing based on current developments of image deterioration due to environmental impacts

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Abstract: The past decade shows a significant increase of digitally produced images. The number of output devices increased and the variety of print materials at different quality levels and with different response to environmental factors more and more substitute traditional silver-halide photography. Comparisons of the technologies become important and show possibilities as well as problems of the new methods. This study deals with the main environmental influences causing image deterioration: light, ozone, temperature and humidity. Existing test methods have been improved to better simulate average home conditions. The results met the assumed lifetime predictions for a variety of different specimen.

Keywords: stability, indoor conditions, light, ozone, ink jet

Remark of closure: The thesis is not closed.

Date: 23.10.2007

Contents

1 INTRODUCTION

Since finishing technology changes from silver halide images to different digitally produced prints go on rapidly during the past years, a great variety of paper, inks and print devices are available for consumers and professionals.

The different technologies show significant differences in behavior to environmental impacts. Photographic prints are hardly effected by the influence of light, due to the fact that the colorants are embedded in the gelatin and therefore less susceptible. Otherwise, the nowadays often used microporous ink jet paper may suffer from a rapid image deterioration due to the open structure of the receiving layer. Ink sets of more than the basic CMYK are in use today. This is a new challenge on the one hand to improve image quality and color gamut and on the other hand cope with image stability issues with multiple inks that may support colorant interactions.

Print results are available which can not be distinguished from traditional silver halide images, even exceed the color gamut. A great variety of print substrates like papers with different surfaces, canvas and films are available.

Although these multiple (6-12) ink-sets have strong improvements on color gamut and increase image appearance, the additional inks may support secondary reactions that lead to image degradation.

The current standard dealing with the issue of image permanence (ISO 18909: 'Photography-Processed photographic color films and paper prints- Methods for measuring image stability') is in revision at the moment. The need for adequate test methods for the changing properties of materials is the reason why ISO technical committee 42 is currently working on several standards which address this issue.

Working drafts at present:

- ISO 18936: Imaging materials- Thermal stability of processed consumer color photographs- Method for testing imaging materials-Thermal

- ISO 18937: Imaging materials- Photographic images- Methods for measuring indoor light stability
- ISO 18940: Imaging materials- Reflection color prints- Specification for consumer-indoor stability
- ISO 18941: Imaging materials- Gas fading stability of reflection color prints- Method for testing imaging materials-Ozone

The main motivation and aim of this study was to provide a reliable test, continue previous tests [14] and building up a whole new test department in providing independent tests for manufacturers and labs. Concerning light fading, the main concentration was to find a possible solution to create a test room to occupy to main requirements:
- active (effective) test area should be sufficient to test many samples,
- alternative, less cost intensive light source than xenon arc which is also easier to handle and leading to more indoor-realistic results
This study was intended as a part of complete printer device test including image quality (resolution, color appearance) and durability aspects (water resistance etc.) see [16].

2 BASICS

2.1 Light and color perception

Light appears in the form of rays, which are electromagnetic waves and therefore two main properties are relevant to characterize them. First the wavelength, which means the distance between two peaks of a wave, and second the amount of energy that is carried by the wave during a time period (radiant flux). If these energy intensities are plotted versus all wavelengths, they show a characteristic curve for the measured light source, the so-called spectral distribution (figure 1). Many physical properties depend on the wavelength and the spectrum, for example the color or the refraction properties of the light.

Optical radiation is divided in different wavelength intervals:
- ultraviolet light (UV): 200 - 380nm, further divided to UV-A (200 - 280nm), UV-B (280 - 315nm), UV-C (315 - 380nm)
- visible light (VIS) between 380 and 780 nm and
- infrared light (IR) above 780nm.

The amount of energy transmitted by the wave is mainly dependent of the wavelength; high frequency rays (short wavelength) transport high energy (roentgen or gamma radiation), long wavelength indicate low frequency and lower energy content (microwave, radar, radio radiation).

To detect a certain color it is necessary to cover three parts:
light source, object that either transmits, absorbs or reflects certain parts of the spectrum and a detector to evaluate this information (human visual system for example). It is a great effort to define color precisely. Densitometry is the technique to measure how a reflective (paper) or transmitting (film) substrate absorbs light. The geometric and spectral conditions are listed in several standards (ISO 5:1-4). A densitometer mainly consists of a light source and a proper filtering which is matched to the spectral sensitivity of photographic paper and film. Status A filtering as used for the measurements uses filter properties shown in figure 2.

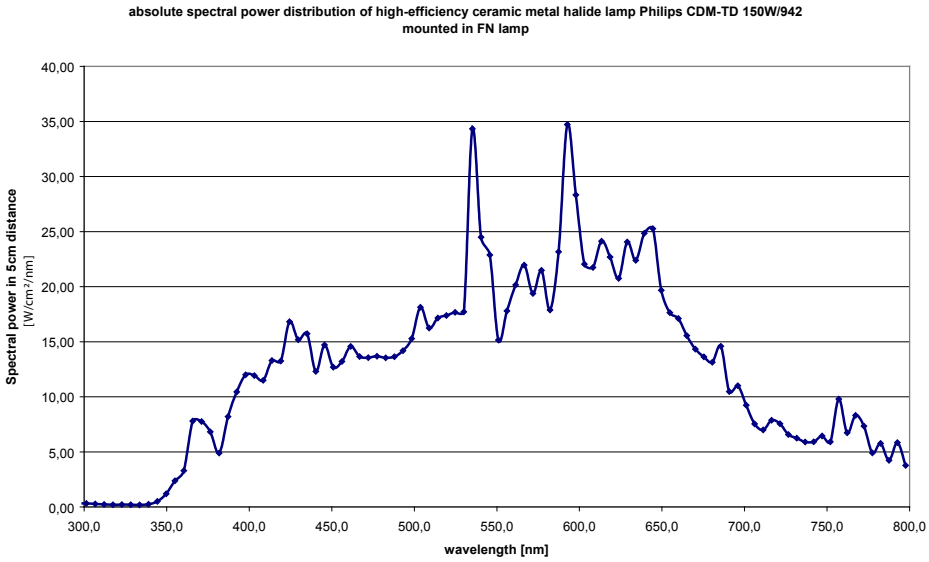

Figure 1: absolute spectral distribution for the light source used for the light fading tests

Optical density (OD) is defined as:

$$D_T(\lambda) = lg\frac{1}{\tau(\lambda)} \tag{1}$$

$$D_R(\lambda) = lg\frac{1}{\rho(\lambda)} \tag{2}$$

with $\tau(\lambda)$ = spectral transmission and $\rho(\lambda)$ = spectral reflection [18].

To receive the individual sensitivity of different dyes, a spectral photometer is needed to measure the characteristic spectral behavior over all wavelengths. Basic principle is shown in figure 3, light is scattered by a grating or prism and the intensity is detected in small nm steps depending on the optical capabilities (lenses, slits) of the device.

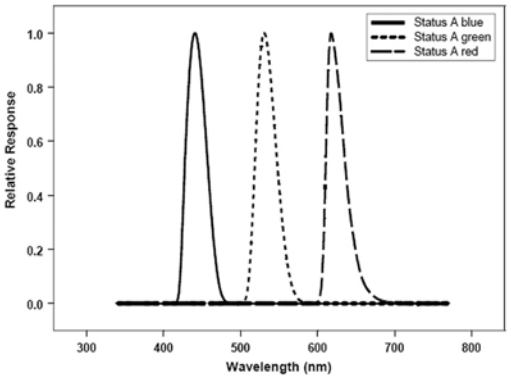

Figure 2: Spectral products of Status A filtering according to ISO 5-3

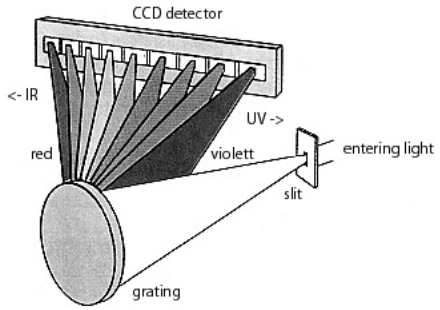

Figure 3: basic principle of a spectral photometer

2.2 Print technologies

The process to duplicate information stored in images or text was in an analogue process repeatable by duplicating a negative or mask. The traditional print processes like offset or flexography belong to the category of impact printing. The ink jet technology does not make use of physical impact to bring the ink to the substrate in contrast to impact printing. [19]

More and more substituted by direct technologies, non-impact printing has found lots of wide spread applications like large format printing for advertising, photo-quality prints as hard copy product for photographers, canvas printing etc.

The most important digital print technologies for home-printing are ink jet and dye sublimation. In case of dye sub technology heat transfers the colorants from a donor ribbon to the paper while with ink jet liquid inks are delivered to the paper drop by drop. Ink jet technologies can be further divided to continuous and drop on demand processes. [13]

2.3 Paper types

The ink characteristics can vary in combination with different paper types depending on paper coating and layer composition. Essential is the position in or on the layer where the colorant is located. Photographic papers contain the colorants embedded in the gelatin, as well as papers for dye sublimation prints, where the colorant is located in a polymer layer, which makes this type resistant to fading caused by pollution. Two main paper types can be distinguished for ink jet printing, swellable and microporous paper. The inks are absorbed mainly on the surface of the receiving layers of swellable papers, while they soak into the open structures of the receiving layer of microporous paper. Pigmented inks may have problems in penetrating into the receiving layer of a microporous paper which leads to smudging effects and less color gloss. Special receiver layers enhance interaction with the inks to avoid color to color bleed and lead to better chroma for photo-quality prints. Surface coating and polymer layers try to protect colorants against complex degradation caused by light or water. Microporous papers more and more substitute swellable type media by amount of sold m^2. [20]

2.4 Ink types

Two main groups of ink types can be distinguished, dye-based and pigment-based inks. Dye molecules are smaller (solid molecule <20nm) than pigment particles (>1000 nm to <100 nm, [21]) and soluble and therefore able to diffuse into the paper layer. This makes the print more resistant to scuff and smearing.

The advantage of pigment based inks is the superior stability to light due to the aggregation of lots of particles which form the pigment. This compound can better survive against the destroying light rays. They are dispersed as fine particles, to be connected to the media they need a dispersant, due to the fact that they are insoluble [1]. Dye-based inks produce more vivid and brilliant colors but are less resistant to environmental impacts.

2.5 Image stability

Light: The most negative effect to colorants have the lower parts of the visible light spectrum and the UV content. These are most destructive since the shorter wavelengths contain higher energy.

IR parts mainly do not lead to photo degradation, but may support thermal induced degradation as it causes heat buildup in the material. [6]

Since the light induced fading occurs as secondary reactions of the dye molecules in the paper substrate, in an indirect way by reactions with oxygen from the ambient air for example [21], it is a difficult task to reproduce these reactions and make veridical predictions concerning image degradation. The light rays animate the colorants to an excited status, from where the separation of the bounds to solid molecules can occur more easily.

Gas: Trioxygen (Ozone: O_3) is a molecule of three oxygen atoms, and a high toxic oxidant.

Most sources of ozone found indoors are polluted outdoor air (especially high amounts on hot summer days), refrigerators, office devices like laser printers etc. The concentrations vary widely during time of the year and geographical position. Ozone can be generated by different sources like electric discharge (corona discharge) and radiation of UV and is favored by nitrogen oxides (NO_X) of cars and traffic. The UV energy is absorbed by the oxygen molecule, which splits into oxygen atoms (O). To create trioxygen, one single

atom reacts with an oxygen molecule to O_3 [4]. Ozone easily dissociates and is therefore known as UV absorber. Maximum concentration indoors should not exceed 0.1 ppm (parts par million)

3 PERMANENCE TEST METHODS

General test conditions: For the observation of the stability behavior of several print substrates and inks, the environmental conditions of light, ozone, temperature and humidity are tested independently in an accelerated test series. Color shifts or deterioration can hardly be assigned to one parameter in a combined test (increased humidity and high ozone concentration, for example). The ISO technical committee (TC42 WG05) tries to improve the current standard [9] concerning image permanence of photographic prints. Therefore the new standard is divided to one specifications part and the test methods parts for each impact parameter like mentioned in the introduction. These methods try to cope with a variety of available print materials and technologies, as they show totally different fading behavior to environmental impacts. The permanence tests methods like executed in this study are based on the drafts of these standards to receive a comparative view of most commonly used materials.

The test target, shown in figure 4, consists of color patches in different intensity steps. Seven colors (black, cyan, magenta, yellow, red, green, blue) are printed in twelve increasing steps, and Dmin patches are left blank in each row. The chart was printed according to the manufacturer's recommendations and possibilities of the print driver and allowed to dry for approximately two weeks before the tests are executed.

All test samples have been measured regularly during the test period, according to the fading behavior of the sample. For each type of paper-ink combination two equal test samples have been tested and the results have been averaged. The measurements have been done by a gretagmacbeth's Eye-One Pro spectral photometer mounted on the automatic reading device I-O and evaluated as Status A densities using the Measure Tool of gretagmacbeth's ProfileMaker 5.05. Further analysis and calculation of the expected lifetime for light and gas fading have been done as described in chapter 4.

The test period depends on the stability of the colorants in paper compound.

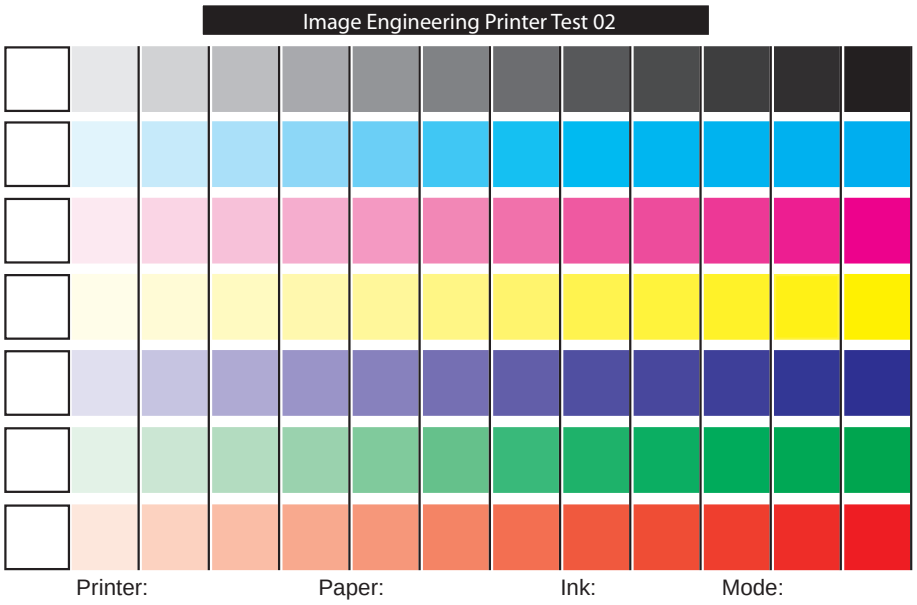

Figure 4: test chart used for the permanence tests, original size 10x15 cm

The light fading unit has a capability to simulate 0.6 years of expected lifetime, the gas fading chamber even three years, in 24 hours testing time (according to the average indoor conditions like mentioned in the following sections). One test sample is finished as soon as one of the following stop criteria is reached. These endpoints represent a noticeable change in the image appearance, not a totally destroyed image. They have been adopted from ISO:18909 with slight modifications, as they are still in discussion [10] and not finally determined for the future specifications standard.
Critical values for interruption of the test, as used for light, gas and thermal fading:

No.	Change Parameter	Allowed Change in Status A Densities at starting point densities of 0.5, 1.0 and 1.5
1	loss of cyan (red density)in neutral color patches	25 %
2	loss of magenta (green density) in neutral color patches	25 %
3	loss of yellow (blue density)in neutral color patches	30 %
4	loss of cyan (red density) in pure color patches	30 %
5	loss of magenta (green density) in pure color patches	30 %
6	loss of yellow (blue density) in pure color patches	35 %
7	cyan-magenta color imbalance in neutral patches	15 %
8	cyan-yellow color imbalance in neutral patches	15 %
9	magenta-yellow color imbalance in neutral patches	15 %
10	changes in $D_{min}(R), D_{min}(G), D_{min}(B)$	0.1
11	chances in Color balance $D_{min}(R-G), D_{min}(R-B), D_{min}(G-B)$	0.06

Table 1: Stop criteria like used for current tests; slightly modified values from ISO 18909:2006

3.1 Light induced deterioration

The most realistic test method concerning light fastness is of course a real-time test, which is an advantage, taking into account real environmental conditions and showing true lifetime of the tested sample. This is at the same time not realistic and leads to the method to simulate a long time period through accelerated fading tests, where the samples are exposed to a high intensity of either parameter.

When a print is exposed to light the energy of the absorbed light destroys the colorants. To receive meaningful results in accelerated tests the light source used for the test is the most important part. The most commonly light found indoors is window filtered sunlight [6]. For the installation of a light fading test unit the aim was to find a solution which can meet or even improve the properties of commercially available light testing devices concerning light source (spectral distribution compared to indoor light), handling and lifetime of the illuminants (efficacy), temperature and humidity conditions in the testing room and available test sample area. Available test chambers usually have problems in keeping the temperature at the sample surface below 30 °C, due

to small volumetric test chamber size and the used xenon light sources lead to significant running costs.

A main approach to defining an average indoor light distribution was made by D.E. Burger et al., (Eastman Kodak Company) [7]. In this and the following study [8] various homes around the world have been monitored for light levels and spectral distributions as well as temperature and humidity conditions in a long-term test (between 6-12 months). The key conclusion is that "ambient home display conditions are dominated by low intensity, indirect, window-filtered daylight" [7]. The spectral energy distribution found as 'average home spectrum' is shown in figure 5 in comparison to other standard distributions and the light source used for the fading tests at Image Engineering.

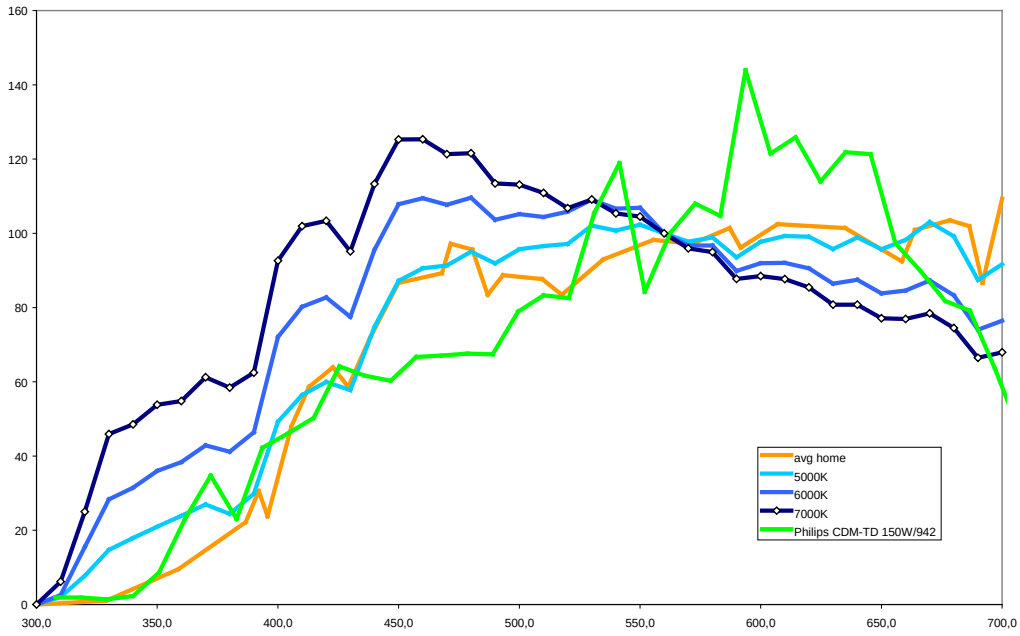

Figure 5: comparison of different spectral distributions

Further conclusions of the studies are the comparison of the average home distribution to other light sources used for fading tests by other labs and researchers. Two types are widespread: xenon arc illuminants and cool white fluorescent (CWF) lamps.

Image degradation is mainly dependent on the spectral distribution of the light source in the test and the absorption characteristics of the colorant. Comparisons between the two mainly used light sources have shown that CWF is less efficient as indoor filtered xenon arc. [11]

The search for alternative light sources lead to high-efficiency, discharge lamps produced by Philips (CDM-TD 150W/942), which are now used in the light fading test chamber at Image Engineering (see figure 6).

Figure 6: Light fading lamp unit and rotating sample area

The spectral distribution is not as close to the daylight as the Xenon lamps but it seems to be close enough for the tests. The big advantage in using the discharge lamps is the lifetime of 6000 hours and the efficiency which is with approx. 70 % close to the one of fluorescent tubes.

The light fading unit consists of 25 devices, each with 150 W output arranged in an array of 5 by 5 and surrounded by aluminum plates to achieve a uniform illuminance on the sample area. A proper air conditioning system is fitted in hight of the lamp array to achieve low temperature of average 25°C on the test sample surface.

The Kodak study claims that "neither glass-filtered xenon nor glass-filtered

fluorescent adequately match the average home spectrum" [8]. The high energy UV and blue part are most responsible for degradation of the colorants, especially for yellow colorants, that have most absorption in the blue part.

Both light sources mentioned above show significant differences to the average home spectrum in the lower wavelengths (UV and blue) which is displayed in figure 7. As a comparison the same plot is displayed in figure 8 where the average home spectrum and the metal halide lamp spectrum are shown as a histogram of nm bands. This shows a good correlation to the average home spectrum in case of the most important wavelengths of the UV.

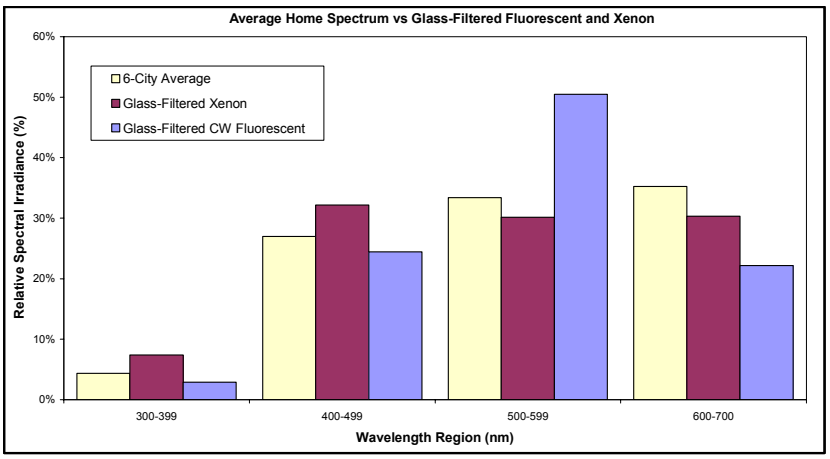

Figure 7: Histogram of spectral irradiance in nm bands [8]

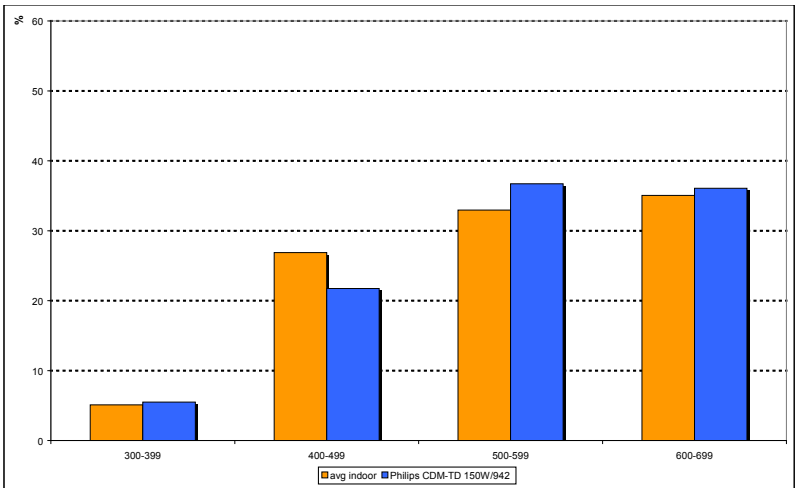

Figure 8: Histogram of the spectral distribution in nm bands, average home - Philips CDM-TD

Different types of filtration for xenon arc lamps are proposed and compared. These combinations may provide a better simulation of the average spectrum, however the filters have a main disadvantage. The optical and physical properties may change rapidly in high intensity fading tests which leads to high running costs.

Another important advantage of the installed light testing chamber is the available space for test specimen (approx. 90 samples of 10x15 size) and the possibility to take single charts out for measuring at any time without shutting

down the whole device.

The spectral distribution of the illuminants are controlled regularly by an Ocean Optics spectral photometer (HR4000), as well as the illuminance level on the sample area (using a Gossen Luxmeter).

The lifetime calculations for indoor light fading are based on the the following equation (3):

$$Light - Stability_{\,Indoor}[years] = \frac{Accelerated\ Exposure\ [klux - h]}{0,450\ klux * 12h * 365\ days/year} \qquad (3)$$

The accelerated exposure is the test time (until one stop criterion is reached) multiplied by the increased light intensity on the test surface. The average indoor conditions which are the basis for the calculation (450 lux per 12 h day, 365 days per year) are higher than found in the study mentioned above [8]. The residential home light intensities are 120 lux per 12 h day. This indicates additionally that test method should met the actual-use conditions as closely as possible. Because this might not be predicted, the future standard might claim to calculate two expected lifetimes to show to customers this effect. The one based on 150 lux/12h day for 'normal' domestic indoor homes or museums, were the archival aspect should be considered. And a second value according to equation (3), which corresponds to offices, for example.

3.2 Influence of aggressive gases

Considerations to stability issues concerning aggressive environmental gases mainly refer to influences of ozone, NO_2 and SO_2. The concentrations of these gases vary widely depending on the position (indoor- outdoor/ cities-industrial areas) or the time (time of the day, and period of the year). The average concentrations (German average) of NO_2 and SO_2 decreased in the past years (1995-2005) to an average 2005: NO_2: $55\mu g/m^3$, SO_2: $10\mu g/m^3$ [20]. These gases do "contribute to gas-fade in all printing processes at nearly the same magnitude, but always to a lesser extent than ozone " [2].

The technical committee therefore concentrates mainly on the effects caused by ozone as it is the most present gas (2005 average: $40\mu g/m^3$, tendency increasing) and proved to be the main factor originating image degradation compared to other gases which are present.

The accelerated ozone tests have found to correlate well with ambient conditions in real time measurements [2]. The maximum concentrations of the test are 5 ppm (parts per million). Again the test period until one above mentioned stop criteria is reached is essential for lifetime expectations.

The lifetime calculations are based on the following equation (4):

$$Ozone - Stability_{\,Indoor}[years] = \frac{Accelerated\ Exposure\ [ppm - h]}{40ppm - h/year} \qquad (4)$$

The base for the lifetime calculation is the accelerated exposure time, the time until one patch of the test chart reaches one of the stop criteria listed in table 1. As the ozone concentrations vary widely during geographical position and time of the year, the nominal indoor yearly concentration is suggested to be 40 ppm-h.

The accelerated test is conducted at 23°C and 50 % relative humidity as these

thermal conditions represent average indoor conditions. The temperature shall be clearly under the level of 50 °C due to the fact that trioxygen diassociates under this condition. Further comparisons might be done in test series with 60 % or even higher RH, as the humidity was found to have a high impact on the fading behavior in combination with ozone. [5]

Test equipment: The device used for the ozone fading at Image Engineering is a Satra-Hampden Ozone Chamber, Model No. 903, which is able to control different ozone concentrations up to 20 ppm (see figure 9). The humidity system is able to control the relative humidity within the chamber between 50 and 80 % at temperatures of 23 - 70°C. The chamber features a recirculating air supply system which provides an airflow between 40 and 400 liters per minute.

Figure 9: Ozone Test Chamber, Satra-Hampden Model 903 and internal test chamber with rotating test piece carrier

The process of ozone generation is going on in a closed loop system like displayed in figure 10.
The spent ozone leaves the testing chamber at the top and gets de-humidified before entering the carbon purifier, where the ozonized air is passed through an activated charcoal filter. The purified air is returned to the housing of the UV lamps, passing a circulation blower, whose speed may be adjusted by a volumetric flow meter. The internal volume of the testing chamber is about

150 Liters, so that a setting of 150 L/min represents one air change per minute. During the test series, the airflow was kept between 180 and 200 L/min dependent on the ozone output of the UV lamps. Fresh ozone is generated from the oxygen in the air supply, mixed with the adequate amount of humidity from the steam bath and blown into the chamber through the bottom.

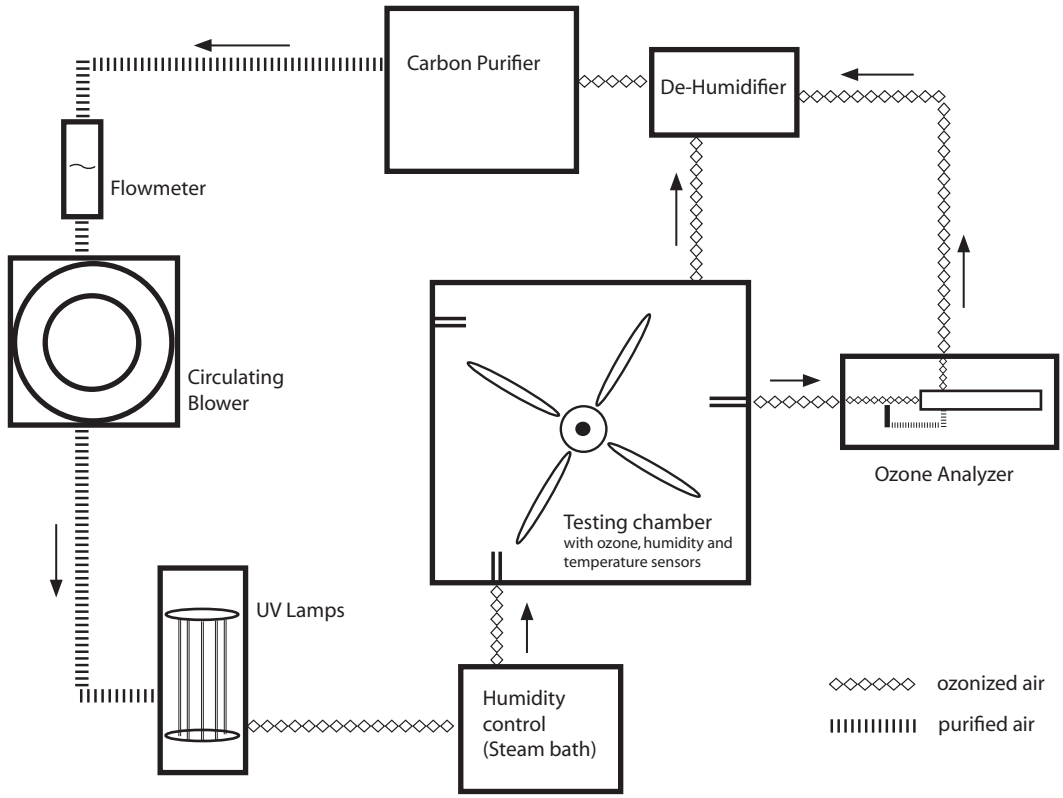

Figure 10: closed loop system of ozone generation

The concentration of ozone is controlled by the variation of the lamp current of the five cold cathode low pressure mercury lamps. One lamp can be driven automatically to compensate fluctuations in airflow, temperature/humidity or reactions of the samples under test, while the other lamps remain at a certain static level. The custom made lamps are cold cathode low pressure mercury lamps with a quartz envelope and allow emitting of short wave ultraviolet radiation at wavelengths below 200 nm. Oxygen molecules from the air flow in the system are dissociated by this radiation and produce the unstable allotrope

ozone.[17]

The concentration of ozone in the testing chamber is determined by a Horiba ambient ozone monitor APOA-360 which is a UV absorption analyzer. Ultraviolet light is absorbed by ozone at a frequency of 253.7 nm, therefore the ozone, passed through a sealed tube, is measured based on the attenuation of light. A low-pressure UV-lamp (mercury vapor) is fitted on the one end and a photo-detector, properly filtered to this frequency, on the opposite side. The concentration is evaluated in comparison to filtered airflow.

3.3 Thermal and humidity stability

In the center European area the thermal conditions are normally less problematic, but particularly museums or other places were images are stored or archived have a major interest on the 'dark stability'. Major problems are increase or stain formation, which can be indicated by a color imbalance in Dmin patches, usually because of rapid increase of blue color density. Other samples may be vulnerable to color shifts, because of dye migrations in the colorant-bearing layer.

High temperature and high humidity can lead to loss of sharpness and color balance depending on paper and ink type. Particularly ink jet prints on swellable paper printed with dye-based inks suffer from higher humidity. Prints made with pigment-based inks are usually less sensitive to high temperature and humidity.

The test series for thermal (dark-) fading consists of four cycles at different increased temperatures (range of 20 °C) and the same level of humidity (55,65,75,85 °C; 50 %RH). One sample is tested for each temperature until one endpoint (table 1) is met. In contrast to light and gas fading the calculation of the expected lifetime is done by using Arrhenius analysis. The four exposure times (to reach one stop criterion) scaled in log times are plotted versus the converted test temperatures (reciprocal of the absolute temperature (1/K)). The resulting fitting curve can be used to derive a time estimation for nominal temperature of 23 °C.

To test the stability of the print result at higher humidity it is kept at 30°C and 80 %RH for a time of 4-14 days. Color shift (Delta E) can be measured with a spectral photometer in color and neutral patches. Both test series are realized individually in a Heraeus-Voetch climate chamber.

3.4 Special test sample types

The base for the measurement of degradation is the densitometric measurement, which follows the geometric conditions of ISO 5-4:1995. For a specific kind of presentation method printed images are laminated between two glass plates with a thickness of approx. 4 mm. Lifetime predictions for these material combinations are of special interest because the additional cover by the laminate is known to support degradation mechanisms of the dyes in the image which results in rapid fading or color shifts. [15]
In general the lifetime of the specific test samples decreases because of the additional coating or laminate. These adhesive films contain solvents which affect the paper and dye compound in negative way and support degradation mechanisms.

Because of the thickness of the final product the densitometric measurements can not be executed by commercially available devices due to the fact that they do not meet the geometric requirements following to the ISO standard mentioned above.

Making lifetime predictions possible was made by developing a method to compare RGB data of the scanned glass samples with 'real' measured density values of test samples of the same type and print process not embedded in glass. The glass samples were digitized using an Epson V4990 scanner and the Lasersoft SilverFast Scan Software v. 6.5.0r3c , profiled by an IT8 chart and with all color correction or image enhancement switched off. The scanner's ICC-profile was embedded to the 3*8bit RGB data. Readout of the data was done by IE-Analyzer Software, Custom Tool, using ICC media whitepoint. The transformation from Lab to XYZ prepared the data readable for Profilemaker as measurement data for the profile creation. The corresponding test prints to every glass sample were measured using a gretagmacbeth spectral photometer and Profile maker's MeasureTool to derive Status A densitometric color values. To receive a correlation of measured densities to RGB values of the glass samples the densitometric values were rescaled to a range of 0...255. Therefore the density data was converted to reflective values and scaled ($Reflexion_{Max}$ to

255). These data sets represent the reference files for the ICC-profile creation.

Look up tables (LUT) were generated to transfer scanned RGB values to the densitometric measurement values. These LUTs were stored in an ICC-profile and therefore easy to handle for all the measurement files during the test. For each type of test sample, matching ICC- profiles were created with the comparison data before the light fading test was performed. At starting time, the test calculations comparing measured to 'calculated' densities showed good correlation for all test types at density values < 1.0 (see figure 11). Higher deviations between 'calculated' and measured densities with values above that point may be due to the little number of sampling points. This is why it is suggested to future tests of that kind to receive a higher bit-depth when scanning the image to increase the accuracy of the rescaling process. In this test series we decided to use 0.6 and 1.0 initial density as base for controlling the stop criteria instead of including 1.5 OD like intended in the revision of the standard.

Analysis of the scanned samples during the test was performed by a Matlab based software tool allowing to cut the image files, store the RGB values and evaluate proper rescaling using the matching ICC-profile. An excel spreadsheet calculated percentage density loss and reported current time and illumination level.

To evaluate an error estimation we carried out a comparative test. We performed the light fading test with four test samples not embedded between glass by measuring real densities and at the same time scanning and calculating the values by above mentioned method throughout the test cycle. There was no difference in the stop criteria which was reached first. For all specimen lifetime evaluation by scanned image data indicates shorter lifetime than the density measurement does. Average deviation of the calculated life expectancy is about 10% (see table below). This amount is assumed to be a realistic rate of error tolerance for the indicated lifetime values.

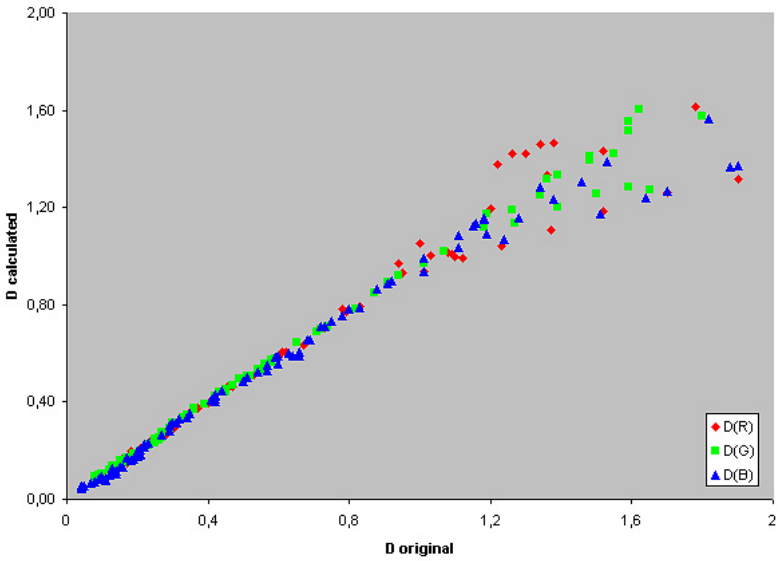

Figure 11: Correlation between measured and calculated density values for sample 1

Sample No.	Scanner method Lifetime [years] for 450 lux /12h day	Density measurement Lifetime [years] for 450 lux /12h day	Average (years)	Deviation [%]
1	14	16	15	-10,9
2	20	23	21	-15,1
3	21	25	23	-13,4
4	44	45	44	-2,2

Table 2: Test samples for error estimation overview

4 EVALUATION AND RESULTS

4.1 Calculation software

To perform an automatic evaluation and overview over the fading measurements a software tool with a graphical user interface (GUI) was designed. The platform of Mathworks Matlab Version R2006a was used to develop the LIfE-TimeCalculator. The complete program and the source code can be found on the attached CD. For the description of the GUI and handling aspects, see the application's user guide in the appendix (page 55).

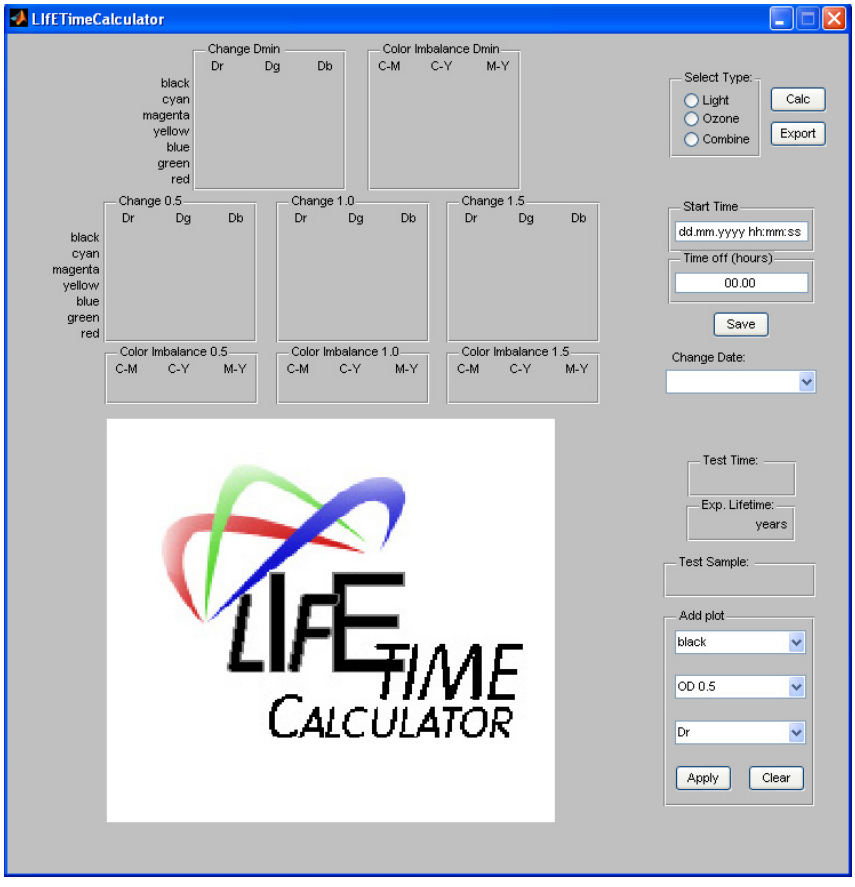

Figure 12: LIfETimeCalculator start screen

The application runs automatically the evaluation of data sets by reading out the optical densities listed in the text files from the spectral photometer and

calculates percentage change. As most test samples show different behavior to light and gas fading which results in different stop criteria reached first, the program provides a 'combi'-mode to compare the fading results of light fading with the proper corresponding gas fading data, and the other way around.

The diagram in figure (13) provides an insight into the main functions of the program which are described in detail below. The overview illustrates the program's structure and chain of the evaluation.

The main program is divided into two main blocks:

First task is to read in all text files and save the density data as well as the date entries of the header. The density values for red, green and blue color density are stored in a three dimensional matrix, color densities listed in each column, therefore each row represents a single patch and the third dimension is the chronological list of times.

The optical densities, which represent the steps to analyze the stop criteria have to be found within the color scales, if not a linear interpolation of two neighbored patches is done in the same portion for the three color densities (see figure 14). Percentage changes are calculated.

The second task is to check the results and compare if any of the proposed stop criteria (like listed in table 1) is reached yet. If this is the case, the expected lifetime according to the equations (3) & (4) should be evaluated and displayed. Therefore the accelerated exposure time is calculated by a polynomial fitting curve of second degree (higher degrees have found to be less accurate due to alternating overshoots)

The future standard will establish to use the major color densities for each color patch:

Cyan:	D_R	Red:	D_G, D_B
Magenta :	D_G	Green:	D_R, D_B
Yellow:	D_B	Blue:	D_R, D_G
Black, D_{min}:	D_R, D_G, D_B		

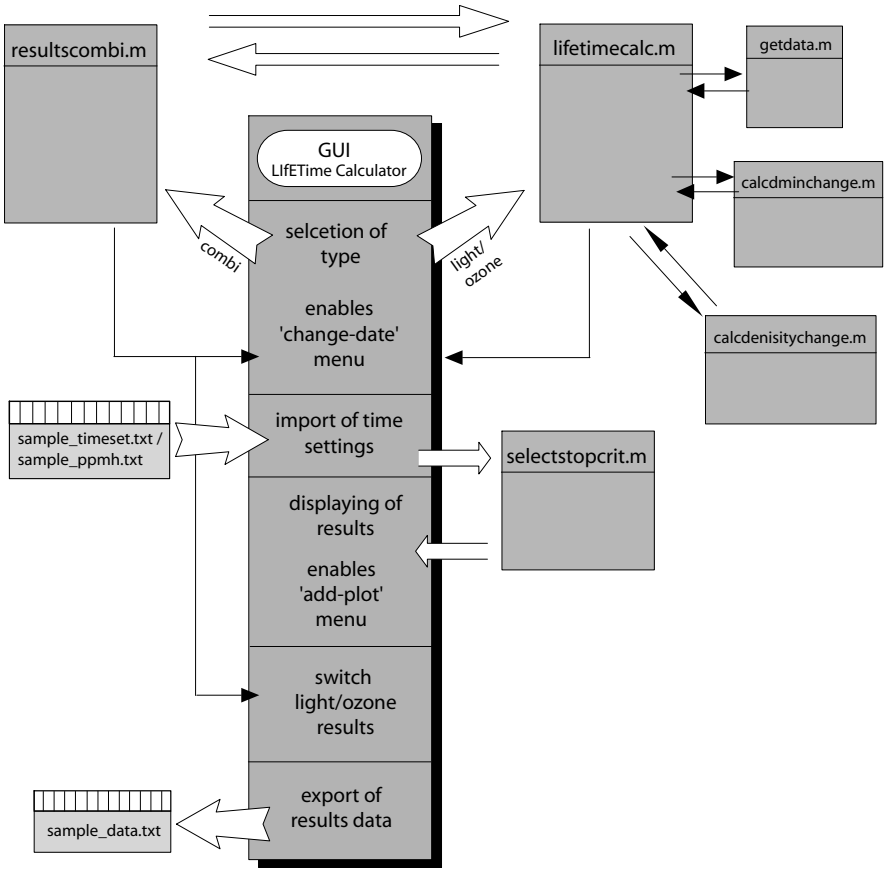

Figure 13: LIfETimeCalculator: Function structure and networking between GUI and most important functions

Overview of the most important functions of the LIfETimeCalculator:

GUI: gui-lifetime.m: The graphical user interface (and its corresponding gui-lifetime.m function) is the 'main' function which creates and handles all figures displayed. Figures are all different types of buttons, text fields, pop-up menus and diagram axes. These are bundled in a so called 'handles'- structure that can be edited and used everywhere in the main- and sub-functions. The GUI is shown in figure 12. The start configuration and all intended buttons and text fields are loaded in the background before the figure is displayed.

Several options are provided and administrated by the GUI-function:

- selection of evaluation type: light/ ozone/ combi

- editing the start time and optional 'offhours' for light fading calculations, or alternatively find and read in data, stored before in the current directory (sample_timeset.txt or sample_ppmh.txt)

- export function: stores all results and current stop criterion with its polynomial fitting data to a txt-file

- all results are displayed in the upper part: absolute D_{min} changes and D_{min} Color Imbalance, percentage change and Color Imbalance of color patches of OD 0.5, 1.0 and 1.5

- enables the 'change date' pop-up menu: the selection of a date represents the position in the third dimension of the endresults-variable which will display the data for this point of time to the results block

- axes figure displays current data, where any stop criterion is reached and the corresponding fitting curve
additional results may be added to the plot by selection of color, OD, and type of criterion with the help of the three pop-up menus on the right side;
the clear button deletes all additional selections and leaves (if reached) the stop criterion data to the plot

- shows expected lifetime and total exposure time (klux-h/ ppm-h)

- in case of 'combi' evaluation: toggle button appears to switch light and ozone results

lifetimecal.m: receives the data output of getdata.m:

reference file and measurement files density data, measurement dates stored in 'timeline', mainsettings (current directory etc.)

- calls calcdeminchanges.m and calcdensitychange.m for evaluation of the results

- clears results for not evaluated data : due to the fact that not every color of the density- triple is used for evaluation (see section above), the deleted values are set to zero

- provides all settings and endresults to the GUI for displaying the results

getdata.m: requires user selection of reference file t_O

- reads in all text files and stores date information from header into one time line, density values ($D_R D_G D_B$) in 91 x 3 x No.-of-measurements matrix

calcdeminchanges.m: - absolute D_{min} change as a difference of $D_{R,GorB}$ and the corresponding $D_{reference}$,

-D_{min} Imbalance as a color shift

(Δ D$_{min}(R-G)$, Δ D$_{min}(R-B)$, Δ D$_{min}(G-B)$) - the seven D_{min} patches of one sample are averaged to compensate measurement variations

calcdensitychange.m: for each color:

- selects the color patches with OD 0.5, 1.0 and 1.5 +/-0.04 from the density data matrix for each measurement time: searches for last value (patch) lower as the low threshold the current OD and observes the following patch & decides:

value in range:

use $D_R D_G D_B$ triple of current patch for calculation: calculate % density loss $time_{1..X}$ and t_0

additional for black patches: calculate Color Imbalance (R-G, R-B, G-B)

value out of range:

use last patch smaller than the lower threshold and use this ($patch_x$) and

the following $patch_{x+1}$ for linear interpolation of the desired OD following equation (5) as found in [8] and also intended for the new standard.

$$OD_{desired(t)} = patch_{1(t)} + [patch_{2(t)} - patch_{1(t)}] * \frac{OD_{desired(t_0)} - patch_{1(t_0)}}{patch_{2(t_0)} - patch_{1(t_0)}} \tag{5}$$

this is done at the value of one color triple, where a second requirement is met: $patch_x < OD_{desired} < patch_{x+1}$, the other color densities are interpolated balanced on the difference to the desired OD (see figure 14)

OD 1.0 or 1.5 not reached:
use maximum measured density for evaluation

selectstopcrit.m - uses the time line to calculate the test period as 'expected lifetime' in years as an universal scaling for light or gas fading using formula (3) and (4)
- checks the results for the proper stop criterion reached and finds correct stop time by adding a polynomial fitting curve (second degree was found to be the best approach)
- finds minimum stop time reached by any of the patches and displays data and fitting curve

resultscombi.m - reads in two sets of data, one for light and one for ozone fading of the same sample type
- evaluates each set of data independently and adds polynomial evaluations for the values of the stop criterion reached by the other parameter
- displays current stop criteria of light fading and the same values reached so far for gas fading to compare both properties of one test sample type
- toggle button appears on the right side of the results block to switch light and ozone data

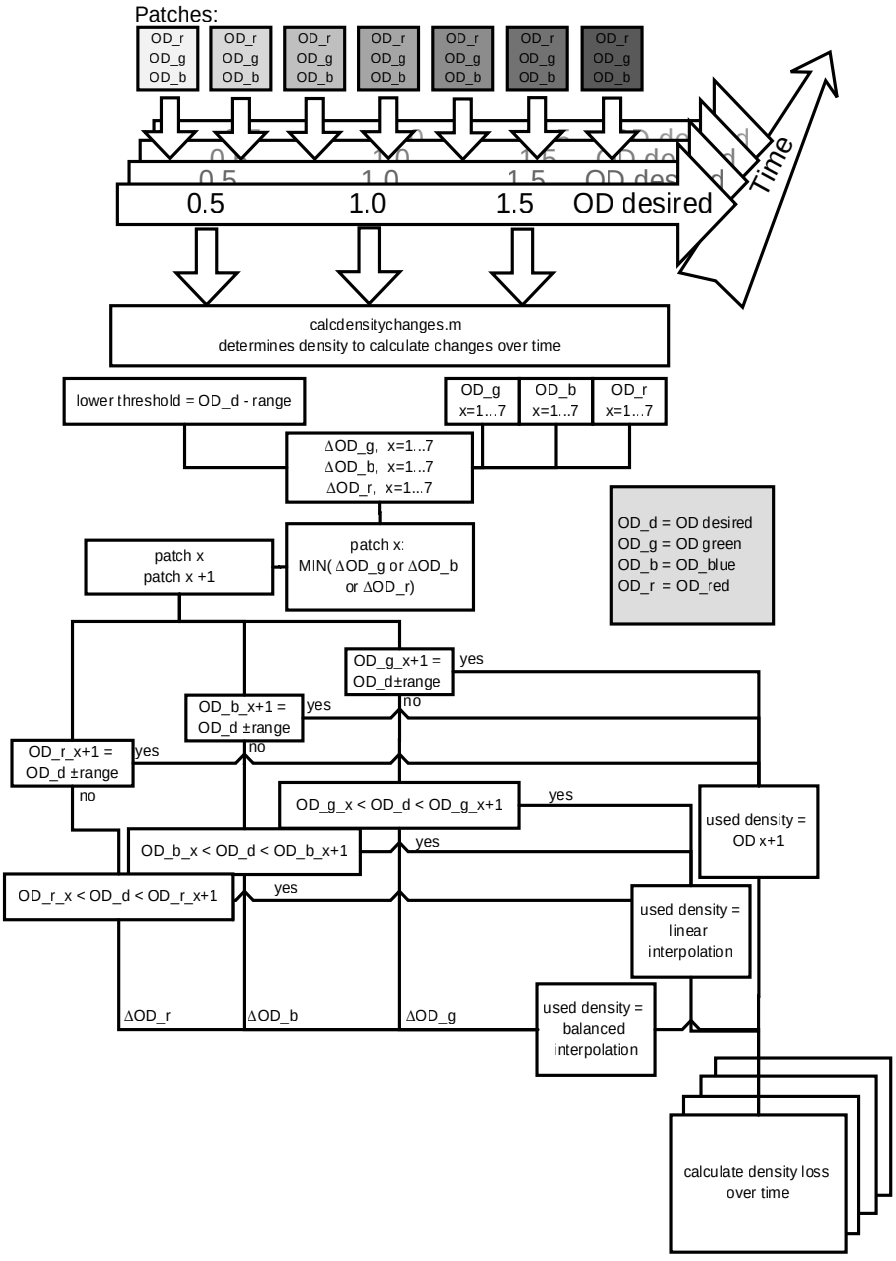

Figure 14: main function calcdensitychange.m - function principle to detect desired OD's from the patch densities

4.2 Test results

The permanence tests have been executed like mentioned before according to the current status of the corresponding ISO standards mentioned before.
The following samples have been chosen for the graphical comparison: (Colorant/Media)

a) pigment/glossy
b) dye/glossy
c) pigment/microporous
d) photographic
e) dye diffusion

These test samples have been chosen to represent a variety of commonly used print substrates and inks including one sample of photographic paper and a consumer 'mobile' small format printer. Sample a) and b) are printed on the same type of photo glossy paper, the first with a new pigmented ten-ink set and the latter with a set of five dye based inks. The third sample represents a high-quality set of 8 pigmented inks and microporous paper. Additional samples are one silver halide photographic paper (d), and one sample printed by a 10x15 thermal dye diffusion printer (e), where the three color layers are covered by an additional protective layer.

Tables 3 and 4 report the results for light and gas fading tests for these samples.

Sample Type Ink/Paper	**Light fading Lifetime [years] for 450 lux /12h day**	**Stop Criterion reached first Patch/org. OD**	**Exposure [klux-h]**
pigment/glossy	30	Yellow-Db 0.6	59225
dye/glossy	13.1	C-Y 0.6	25903
pigment/microporous	135.3	Blue-Dr 0.6	266752
photographic	16.9	Cyan-Dr 0.6	33221
dye diffusion	4.8	C-Y 1.0	9397

Table 3: Light fading - Results overview

The results approve the assumption that the different ink-media combinations

Sample Type Ink/Paper	Gas fading Lifetime [years] for 40 ppm-h/year	Stop Criterion reached first Patch/org. OD	Exposure [ppm-h]
pigment/glossy	>108	—	4360
dye/glossy	2.6	Yellow-Db 1.0	102
pigment/microporous	57.5	Black-Dr 0.6	2300
photographic	>108	—	4360
dye diffusion	>108	—	4360

Table 4: Gas fading - Results overview

can show totally different behavior to the influence of either light or ozone. The most extreme example in this test series is the dye diffusion print, which shows a high resistance to ozone but a weak light stability. A similar tendency can be determined for the photographic paper. The colorants are completely surrounded by the gelatin, so that they are well protected to degradation due to aggressive gases. The light stability is moderate but exceeds the expectancy for the dye based sample, which is additionally faded by ozone much quicker than all the other test samples. The overall highest resistance to light has the pigmented ink on microporous paper with more than 130 years lifetime. Also the ozone stability is with a lifetime > 50 years good for this sample. The comparison of both samples printed with pigment based ink shows the dependence of ink and media compound. Both samples reach highest results for both test parameters. But as the weakest part of the chain determines the lifetime, the paper is an also important key to resistance of the image. So the important factor for degradation is in the one case (photo glossy paper) the influence of light, and in case of the microporous paper the environmental gas.

One example comparison of a faded test sample is attached to the appendix. The original test chart, one sample which passed the light and gas fading test have been scanned. The seven color scales have been reassembled to make direct comparison of each patch possible.

The following chapter shows the graphical plot of the density changes of the selected samples. For comparison purposes not necessarily the data of the reached stop criterion are listed, but expected lifetime versus percentage density change for pure Cyan, Magenta, Yellow patches and Black patches (OD

0.6) for all samples. For lower densities the inks are usually diluted and more vulnerable to environmental impacts. In few cases negative interactions between the inks of the higher concentrations may prevail and lead to more rapid fading (as seen in dye diffusion sample for light fading). The results are shown for light and gas fading test series as polynomial fitting curves based on the measurement data sets.

4.2.1 Light fading

Sample a): The yellow ink is the weakest to the high intensity of light and is also the one reaching the first stop criterion. The neutral patch fades constantly for all three color densities, slightly faster for the yellow part (Db) as well.

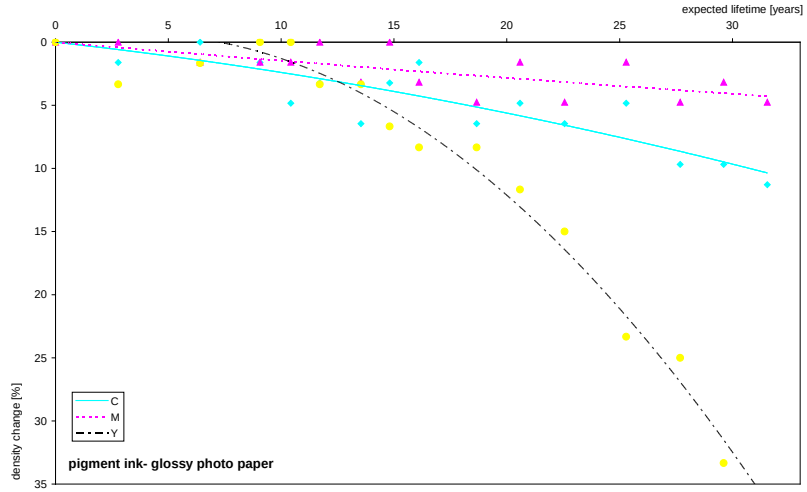

Figure 15: light fading: pigment/glossy, C M Y patches

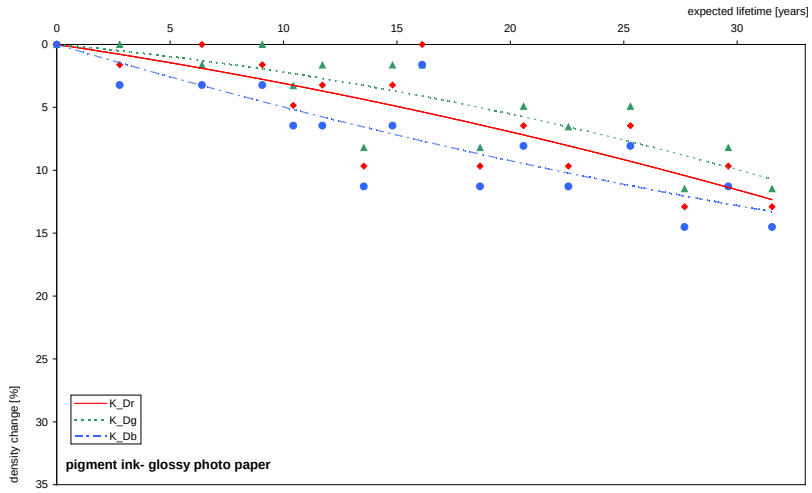

Figure 16: light fading: pigment/glossy, Black patch

Sample b): Magenta and yellow ink fade nearly linear while the cyan ink remains stable. A color imbalance can be seen for the neutral patch, due to the yellow color density fading much quicker than the others.

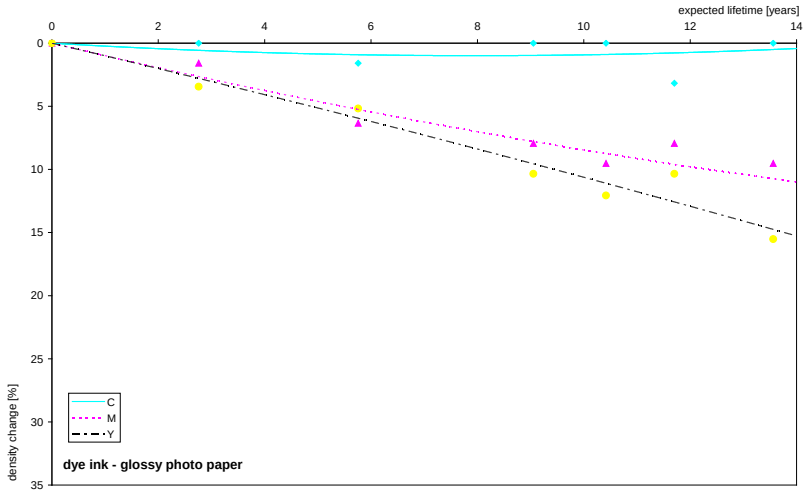

Figure 17: light fading: dye/glossy, C M Y patches

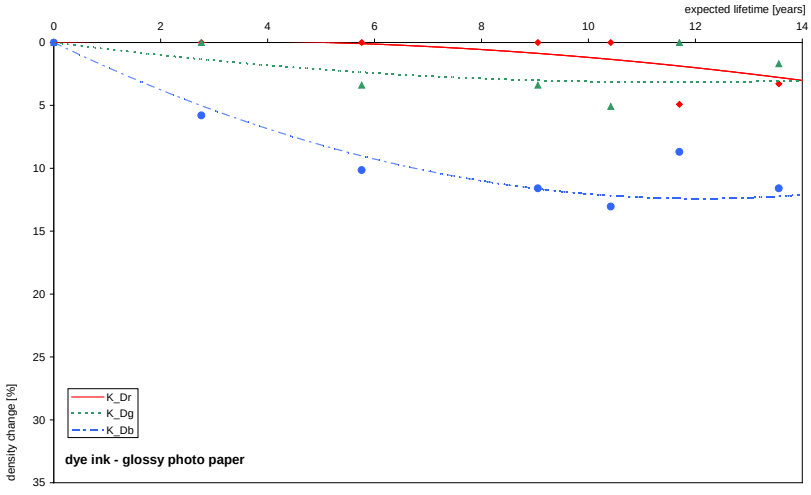

Figure 18: light fading: dye/glossy, Black patch

Sample c): The overall best results for light fastness reaches this pigmented ink, where finally the cyan ink decreases slightly faster than the others.

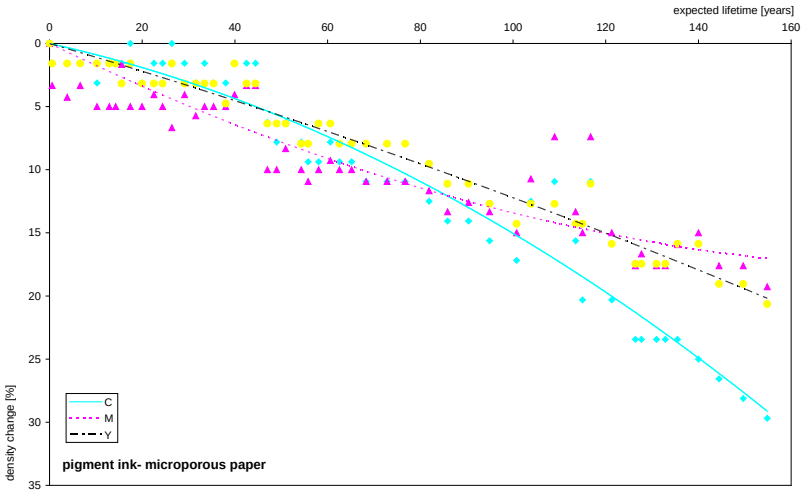

Figure 19: light fading: pigment/microporous, C M Y patches

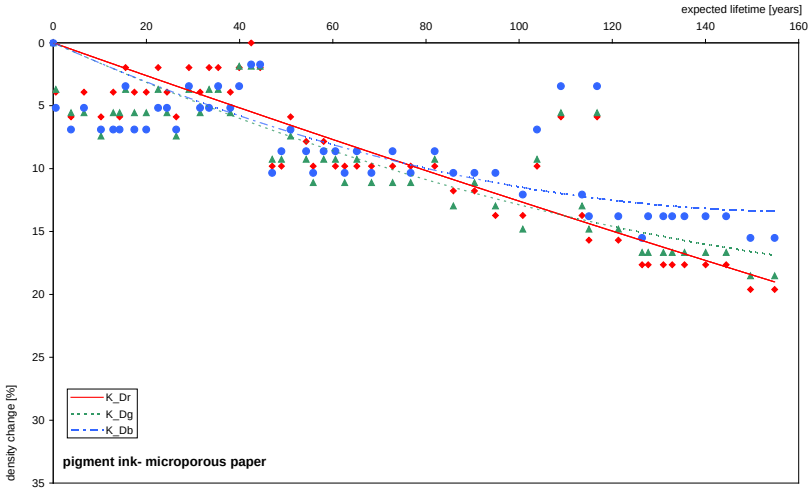

Figure 20: light fading: pigment/microporous, Black patch

Sample d): The magenta and yellow colorant of the photographic print shows nearly identical fading behavior for either pure and composite patches. Both tendencies are exceeded again by the cyan colorant. In the pure color patch it is even more unstable than in composition.

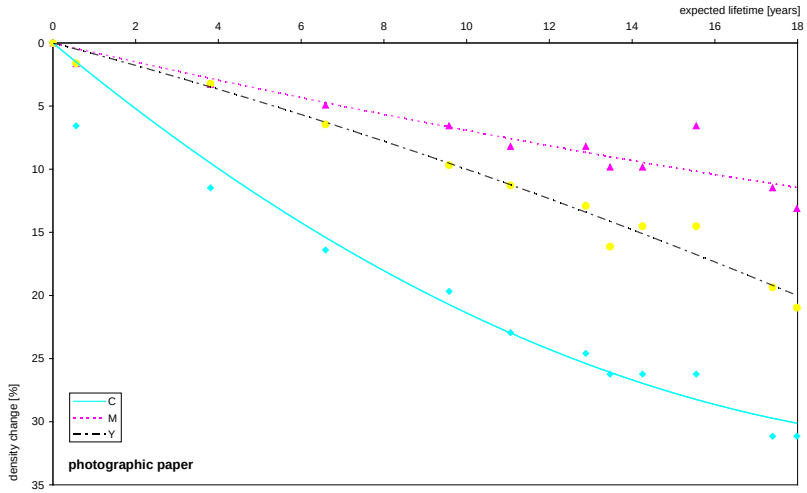

Figure 21: light fading: photographic, C M Y patches

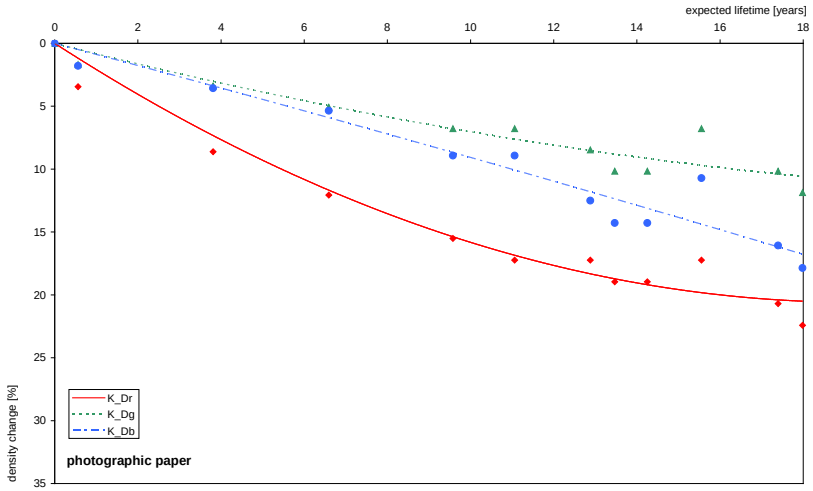

Figure 22: light fading: photographic, Black patch

Sample e): The cyan colorant is the one most problematic for the dye diffusion sample, while magenta and yellow fade slowly at the same rate. This tendency gets even more clearly for the black patch data, since the black is a 'real' composition of CMY and not a combination with a black ink.

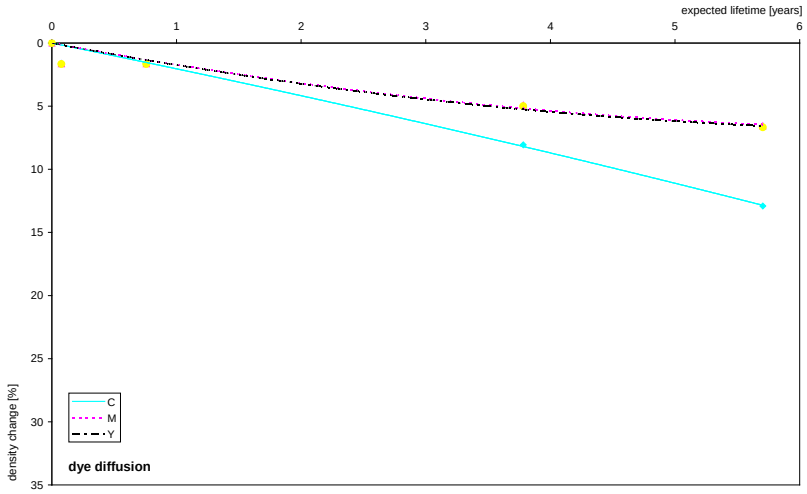

Figure 23: light fading: dye diffusion, C M Y patches

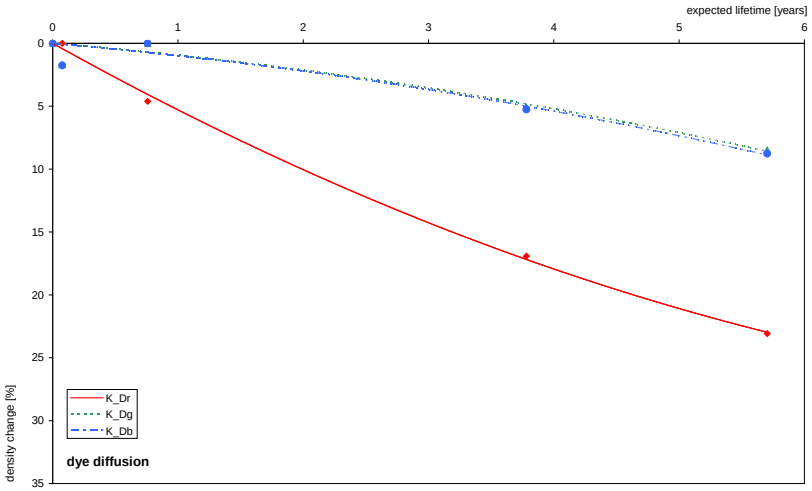

Figure 24: light fading: dye diffusion, Black patch

4.2.2 Gas fading

Sample a): The pure color patches show a more or less linear decline for the first half of the test period, magenta and cyan patches decrease more slowly during the second half. Any of the patches do reach a stop criterion during the test. Green and red color density of the black patch fade more quickly than the blue part.

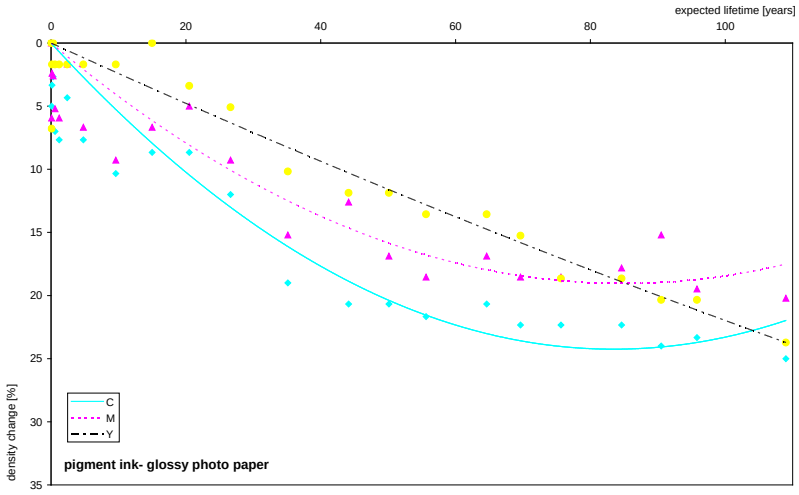

Figure 25: ozone fading: pigment/glossy, C M Y patches

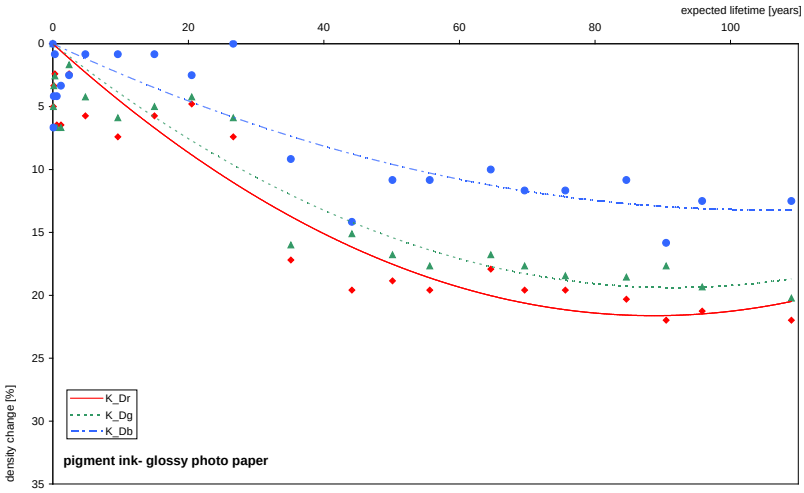

Figure 26: ozone fading: pigment/glossy, Black patch

Sample b): The most problematic ink for this sample is the yellow ink, which decreases during the short test period of approx. 2 years rapidly to the stop criterion of 35 %. At the same time magenta and cyan reach at max. 10% loss.

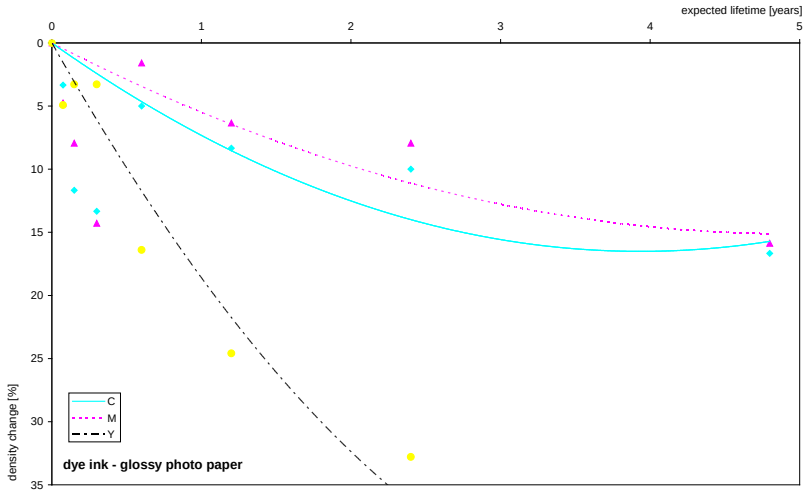

Figure 27: ozone fading: dye/glossy, C M Y patches

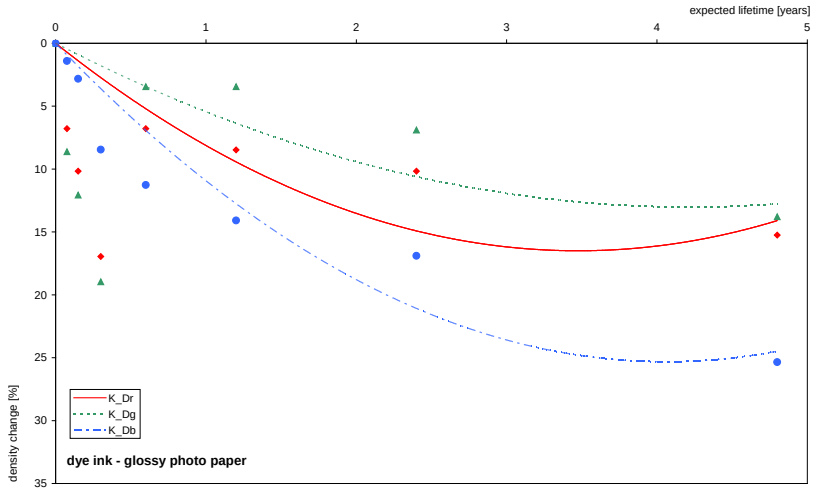

Figure 28: ozone fading: dye/glossy, Black patch

Sample c): Second highest lifetime in the test was reached by sample c). Compared to the light fading of this sample, the cyan ink is again the weakest part.

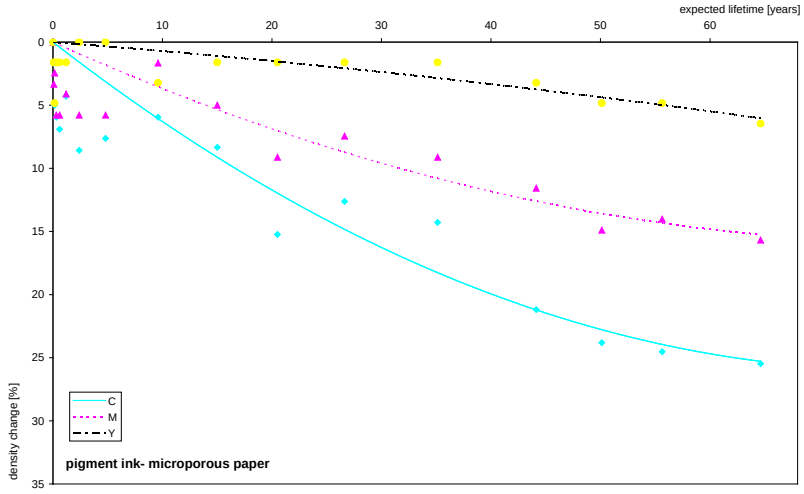

Figure 29: ozone fading: pigment/microporous, C M Y patches

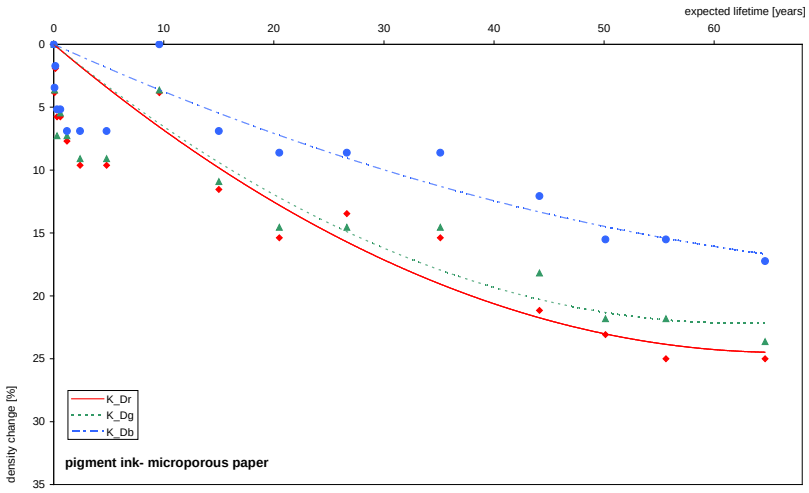

Figure 30: ozone fading: pigment/microporous, Black patch

Sample d): As supposed the photographic test sample is not notably affected by ozone, for the color densities of the black patch, a slight decline can be determined but till the end of the test period all values do not exceed 7 percent loss.

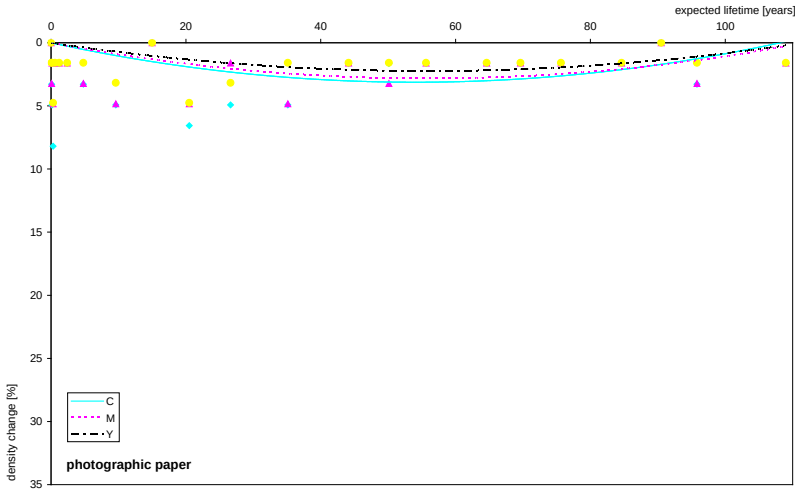

Figure 31: ozone fading: photographic, C M Y patches

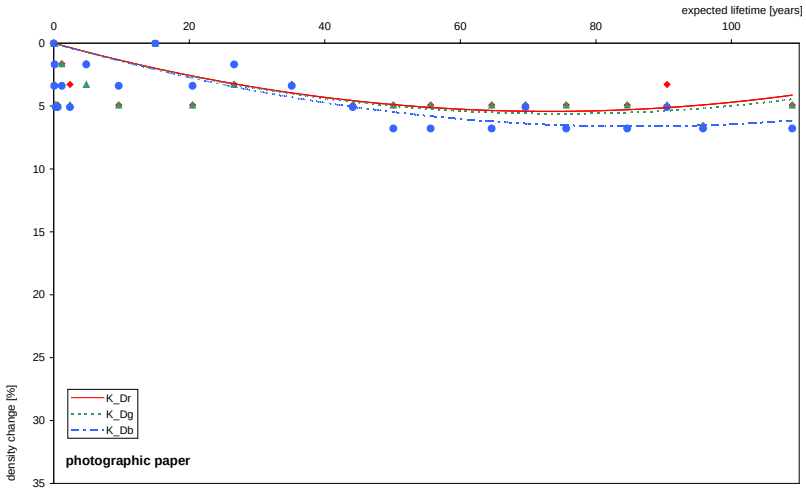

Figure 32: ozone fading: photographic, Black patch

Sample e): The individual colors of cyan, magenta and yellow show marginal changes (less than 6%) during the test period of more than 100 years of lifetime. The combination of the colorants results in a moderate decline for red and green color density, and slightly more for blue color density, but no stop criterion is met.

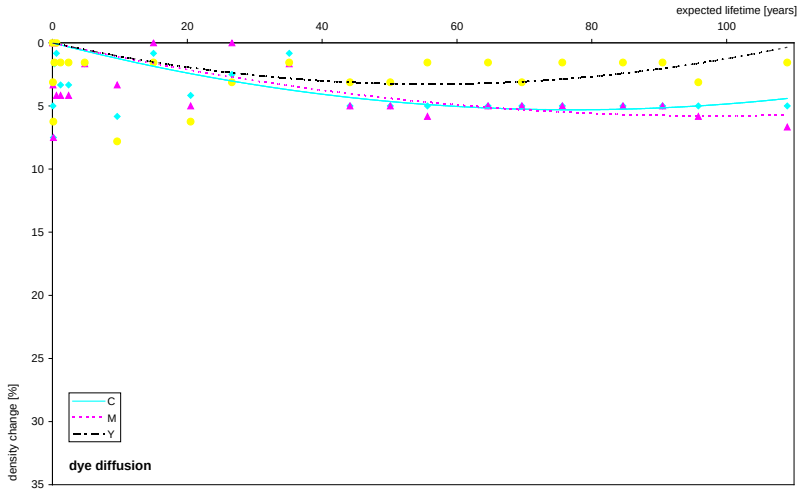

Figure 33: ozone fading: dye diffusion, C M Y patches

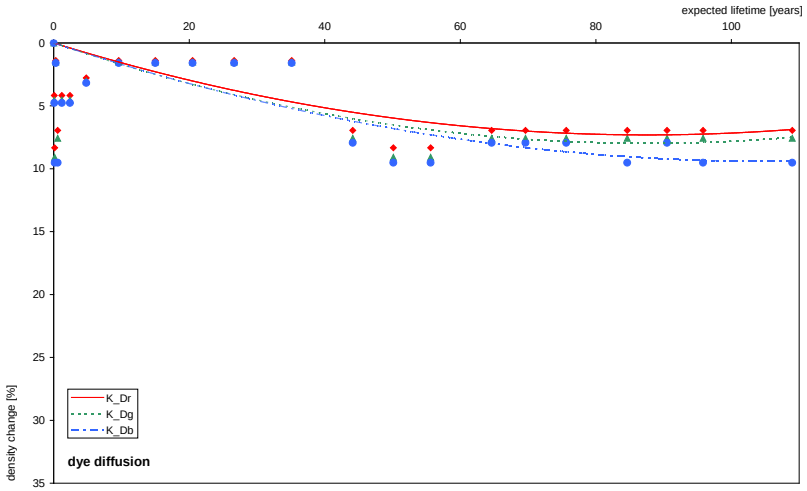

Figure 34: ozone fading: dye diffusion, Black patch

4.2.3 Thermal stability and humidity fastness

Due to a lack of time and available test equipment there was no investigation of the stability effects caused by increased humidity and the test series concerning thermal stability are currently in process. Having finished nearly two of four intended temperature settings (highest temperatures of 85 and 75°C) some assumed tendencies can be shown: Staining is the most severe problem for nearly all tested samples, which is shown by an increased Dmin density, especially in the blue density. Some samples might exclusively be investigated at the Dmin patches due to dye migration and colorant-bleed which is problematic with some photographic papers due to swelling of the gelatin at higher temperatures.

4.2.4 Sources of errors:

Different sources of errors are known which occur and uncertain the listed results:

- measurement error of the spectral photometer as given by the manufacturer in Delta E: (inaccuracy 0,3, repeatability: 0,02)
They do mainly effect the measurement of the Dmin patches (which range between 0 and 0.1 OD). Therefore the target consists of seven minimum patches to minimize this effect by creating a mean of these values for evaluation.

- inequalities in the light distribution:
For all tests two samples of each type are measured as a mean and additionally to the rotation of the test area, the samples change their position on the board itself regularly.

- fluctuations of ozone concentration:
To rely on the test chamber, the ozone analyzer needs regularly recalibration. During the test series the constancy of the ozone concentration lies between +/- 5 % of the set concentration.

- Temperature and humidity fluctuations:
They are measured regularly and controlled to minimize fluctuations. In practice the temperature for light fading ranges between 23 an 25 °C, humidity between 43-50 %. The ozone chamber can be kept between 25 and 28°C and 45 and 55 % RH.

5 SUMMARY

The results approve the assumed fading rates according to the image properties. Due to the fact that the reliability of the accelerated tests depend largely on the actual-use conditions that are normally not known before, it is not possible to simulate all influences that weaken a print in one test.

The test results can never give a guaranteed lifetime of a paper-ink combination, because the real time conditions of storage and displaying will vary widely. However, these accelerated test series can provide a valid classification concerning the specific environmental impact and may show possibilities of improving the storage conditions to have stable images for a longer time. The lifetime predictions will not lead to 'one number', for consumers it is important to easily compare the information provided. Anyhow, consumers may decide on the intended storage conditions: prints framed under glass may not be affected by pollutions, or samples like thermal diffusion print may be a choice for prints that are stored in albums where less light can degrade the prints.

Efforts have been made in providing test methods and equipment more reliable to average indoor conditions. This is important for manufacturers to receive a comparable overview of the different substrates with a variety of possible colorants.

Further improvements could be made for the evaluation software in the future by adding a function which evaluates two equal samples simultaneously and creating the mean, due to the fact that normally two samples of one test type are measured. Current discussions try to further define the precise properties of the equipment for the tests, especially concerning the light source used in light fading tests.

These new approaches try to find a way of describing the energy properties of the light source and abosorbance capabilities of the dyes. The current standard simply suggests different types of illuminants which was found to be less precisely compared to the average home spectrum.

6 APPENDIX

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LIfETime Calculator's User Guide

The Mathworks Matlab based Software tool was designed to evaluate lifetime predictions according the current ISO standards drafts based on density measurement files created by the gretagmacbeth I-One spectral photometer. The charts for the permanence tests with a size of approx. 10 x 15 cm, are exposed to a high intensity of different environmental impacts and measured regularly. They contain seven different colors (K, C, M, Y, R, G, B) in twelve increasing intensities and additional Dmin patches without any color application. Automatically they are measured by the spectral photometer and interpreted as Status A densities by the evaluation software. The values are stored in a .txt file, the charts are controlled regularly so that each test sample is represented by a set of data.

System requirements for the tool is the Matlab component runtime routine which can be found on the attached CD and has to be installed before (MCRInstaller.exe).

The start screen of the LIfEtime Calculator is shown in figure 12. First selection has to be done in the upper right part.

Light: For first evaluation of one data set the corresponding start time should be typed into the 'starttime' editing field. If longer periods of interrupting the tests should occur, the 'offhours' can be set accordingly and saved. Pushing the 'Save' button will directly start the calculation and create a file named sample_timset.txt to the current directory where the date entries and off-hours are stored. The dates of measurement are important to calculate the current test time and the simulated lifetime as a difference from the typed start time. For the light fading calculations these dates are adopted from the .txt file header which lists date and time. Due to the fact that the samples can easily be taken out of the test chamber for measuring, these dates represent the current measurement point. For following evaluations of the same data set, the time set file is recognized automatically.

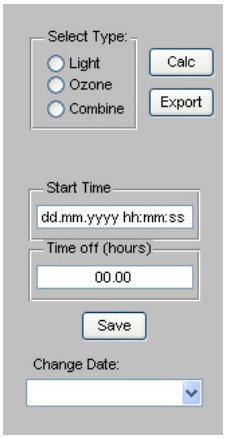

Figure 35: Start selections

Gas: In terms of gas fading, the whole test device has to be shut down to open the chamber and measuring all files can endure approx. one hour. Therefore a list of ppm-h (sample _ppmh.txt) of the exposures during the test period is attached to each set of gas fading data. Examples can be found in the attachment as well.

Combi: The 'Combi' mode tries to give an impression on how the prints will behave when exposed to both light and environmental pollutions. The degradation effects of light and ozone are supposed to be cumulative and therefore both results will be displayed for one sample type. The user has the possibility to look at the light fading results and the results for gas fading of the same type of stop criteria at the same time. Usually there will be a difference in which stop criteria is reached first by light or gas fading. On the left side o the results block will appear a toggle button which enables the user to alternatively display either the light or gas fading results and display the equivalent data for the same stop criteria of the second component in the diagram.

The 'Calc' button will start the evaluation. The user is requested to select the reference file of the current test sample, named sample_TO.txt. For Combi-Evaluation select first the light fading and second the gas fading data. First aim of the software is to select the patches with OD 0.5, 1.0 and 1.5 for every

color and calculate the percentage density change (or absolute change for Dmin patches). The results are presented in the upper part.

The 'Change dates' pop-up menu shows a chronological list of the measuring dates, each can be selected and the result values for the selected date will be displayed.

To save the results and the data sets for the calculation of the expected lifetime to a .txt-file in the current directory, 'Export' has to be clicked on.

The diagram will be filled as soon as a stop criteria is reached automatically, if not, the user can select specific data sets by the 'Add plot' menu to have a view on the speed of decline for one color patch. The diagram shows percentage density change versus expected lifetime of the current patch (+) and the polynomial fitting curve (-). See the legend for the first reached stop criteria. The menu contains three pop-up menus for color, OD and color density selection.

The 'Apply' button will display the current selection, the Dmin data will refer to the right side Y-axes due to scaling properties. To clear the graph press the so-called button, this will not effect the first reached stop criteria, which will be excluded.

Warning massages that may occur are listed in table 5.

Warning:	**Problem:**
'Status A not met'	The named text file was not saved as Status A density, which will be indicated in the file header. The measurement information can not be used, remove the file.
'Check ppm-h list'	Either there exists no list in the directory of the sample files or the program has problems in reading the data. Compare to example file on the attachment.
'Check start time'	Check if _timeset.txt file exists and if the date format is correct.
'Problem reading data'	Check .txt files for any inequalities
'No stop criteria reached yet'	The results data will be displayed so far and it is also possible to use the plot function to get an impression how rapid the color loss might be.

Table 5: LIfETime Calculator: waring dialogs

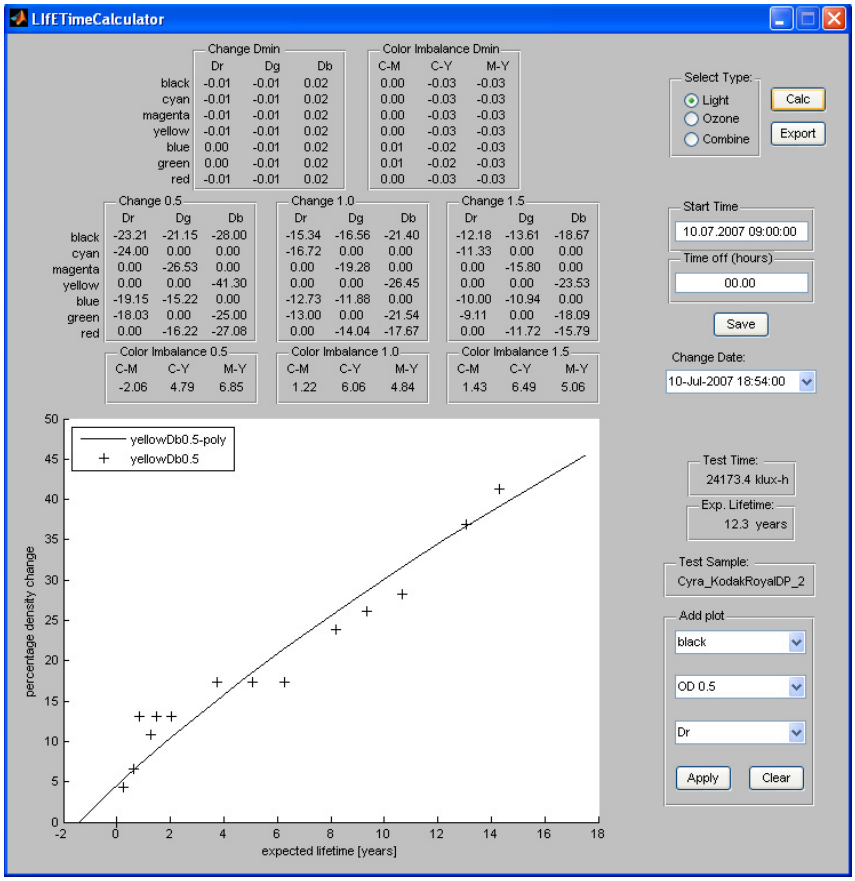

Figure 36: graphical display of the results

Eidesstattliche Erklärung

Ich versichere hiermit, die vorgelegte Arbeit in dem gemeldeten Zeitraum ohne fremde Hilfe verfaßt und mich keiner anderen als der angegebenen Hilfsmittel und Quellen bedient zu haben.

Köln, den 23.10.2007

Agnes Etzkorn

Sperrvermerk

Die vorgelegte Arbeit unterliegt keinem Sperrvermerk

Weitergabeerklärung

Ich erkläre hiermit mein Einverständnis, dass das vorliegende Exemplar meiner Diplomarbeit oder eine Kopie hiervon für wissenschaftliche Zwecke verwendet werden darf.

Köln, den 23.10.2007

Agnes Etzkorn