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Development of LightCheck® Ultra

A NOVEL DOSIMETER FOR MONITORING LIGHTING CONDITIONS OF HIGHLY PHOTOSENSITIVE ARTEFACTS IN MUSEUMS

Anne-Laurence Dupont, Costanza Cucci, Claudine Loisel, Mauro Bacci and Bertrand Lavédrine

This paper presents the development and optimization of an ultrasensitive light dosimeter, especially designed for the preventive risk assessment of damage to highly photosensitive artefacts. This indicator, named LightCheck® Ultra, is composed of a photosensitive dye/polymer layer applied on a paper support. The indicator is characterized by a progressive colour variation as the exposure to light increases. Initially blue, the colour successively changes through purple and pink to white, under the effect of visible light. The colour variation was investigated under different environmental conditions. The light-induced alterations under study were evaluated using various light sources and illuminance levels. The impact of other factors unconnected to light (such as temperature, humidity, indoor air pollution – atmospheric and volatile organics – and oxygen) was also studied. The behaviour of the indicator in the field in selected museums and historic houses was assessed in parallel with the laboratory experiments at each development stage. Finally, the calibration was carried out and a reference colour scale was provided for fast and easy reading of the indicator. By means of this reference colour scale, the colour exhibited by the dosimeter after an exposure can be easily correlated to a quantitative indication of the light dose received. The phases of development and testing of this novel light dosimeter are presented in detail, from the early design to the final product.

INTRODUCTION

The assessment of the environmental risks of damage to museum artefacts is essential for establishing preventive conservation strategies. Passive sensors, like dosimeters or indicators, which indicate the level of risk that can potentially result in damage to objects on exposure, are thus very useful for implementing a risk management policy in museums and galleries.

Dosimeters work on the principle that their reaction to a given agent should follow a dose–response function similar to that expected for the objects under study. When this response is also much faster than that of the object, dosimeters can be used as an early warning for risk assessment. One requisite is that the specific property which changes upon exposure of the indicator, and which is measured, should be representative of the rate of decay, or related to the degradation phenomenon of the object and easily quantifiable. In the best case scenario, the alteration induced in the indicator should be clearly distinguishable by simple visual examination, so that a simple reading can be made on site with no specific measuring equipment.

Dosimeters usually provide a very simplified model and, in reality, their response function cannot be expected to reproduce exactly that of artefacts under the same conditions. Rather, the sensor should be used for semi-quantitative indications by ensuring that its behaviour can suitably simulate the reaction under analysis (e.g. discolouration or corrosion).

In this research, the environmental factor under examination was light, and the sensors developed were specifically tailored to respond to the damaging effects of light.

Light induces photochemical reactions in materials which may result in a change in the appearance of objects (e.g. fading or chromatic balance) [1]. It is well established that any unnecessary light exposure should be avoided for art objects and archival materials on display [2]. Moreover, it has to be taken into account that these reactions can also be modified or accelerated by other environmental parameters such as temperature, humidity, external atmospheric and indoor air pollution

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[3]. The complex mechanisms involved in these phenomena cannot be reproduced or foreseen for all the materials and objects of cultural interest. Nevertheless, the assumption was made and verified here, that the property measured (the light-induced colour change in the dosimeter) is a good parameter for establishing the possible risk of damage to works of art on exhibit due to the overall action of light and the micro-environment.

A well-known light damage indicator, which works on the same principle of light-induced colour change, is the International Standards Organization (ISO) Blue Wool Standards scale (BWS) [4–6]. BWS are adapted from the international classification of light stability for modern textile colours and comprise eight fabric based indicators with different levels (1–8) of light-fastness [7]. BWS are commercially available and their use in the museum field is quite widespread. Nonetheless, for low light doses (LDs), that is, exposures below 100 klux hours, even the most light-sensitive BWS (no. 1) clearly fails to respond promptly [8]. Several other attempts have been made in the past to monitor lighting conditions – and other environmental factors – with dosimeters in museums, but to date none has reached the level of a marketable product [9–16]. In addition, the sensitivity range of these indicators is not adapted to monitoring the low cumulative light exposures which are of particular interest when a preventive approach is favoured.

Based on these observations, research was initiated at the Centre de recherche sur la conservation des collections (CRCC) to develop a light dosimeter for estimating very low to moderate LDs, between 5 and 100 klux hours. The intention was to identify a combination of dye/matrix/support able to react, with a controllable colour change, to the LD received. This early work led to a patent, and the first prototype of dosimeter was later modified, optimized and fully validated within the frame of the European project LiDo [17, 18]. LiDo was aimed at achieving a marketable product for risk assessment of the light-induced damage in museums and galleries [19]. The consortium of partners in the project included three research institutes (Fraunhofer-Institut für Silicatforschung, coordinator of the project; CRCC; and Istituto di Fisica Applicata 'Nello Carrara'), two small and medium enterprises (SMEs; UV-Technik and Particle Technology) and two cultural heritage end-users (Victoria and Albert Museum and National Institute for the Preservation of Cultural Heritage of the Czech Republic). The goal of the project was the production of new competitive indicators that would complement the lack of sensitivity of BWS by responding to very low to moderate light levels over relatively short periods in a quantifiable fashion. The research encompassed the development of two new types of light dosimeter, which, upon transfer to the market at the beginning of 2004, were named LightCheck® Ultra (LCU) and LightCheck® Sensitive (LCS). LCU was projected as a highly sensitive indicator for surveying very light-sensitive artefacts (ISO Blue Wool Standards 1–3; e.g. colour photographs, watercolours, textiles and natural history specimens), which should be exhibited under low levels of light [20, 21], whereas LCS was aimed at monitoring lighting conditions of more durable objects (ISO Blue Wool Standards 4–6; e.g. oil paintings, tempera, polychrome sculptures, bone and ivory) [22]. As a whole, LCU and LCS constitute a light-monitoring tool known as the LightCheck® system, suitable for very different lighting situations.

The aim of the work presented here is to illustrate the methodological approach and the research process that were behind the release of the final product. This paper deals exclusively with the case of LCU, the more sensitive prototype, and reports in detail the main phases of the research accomplished for the fabrication, development and characterization of the dosimeter. Field exposures were also carried out in selected museums and historic buildings at every stage of the prototype development in order to examine the behaviour of the dosimeter and its suitability for monitoring lighting situations on site. The data obtained in the field were then cross-checked in order to validate the laboratory tests.

EXPERIMENTAL

Materials

The LightCheck® Ultra indicator is made of a light-sensitive coating prepared with two dyes: Toluidine Blue O (Colour Index No. 52040) and Eosin 225, a red dye preparation for histology based on Eosin Y (Colour Index No. 45380).

Two types of polymer were investigated to act as the matrix for the dyes: polyvinyl alcohol (PVAL) and polyvinylacetate (PVAC). Three PVALs were tested: Rhodoviol 25/140 (viscosity at 20°C of a 4% aqueous solution 24–30 mPas), PVAL 99% hydrolysed (average molecular weight, M_w 85000–146000) and PVAL 98–99% hydrolysed (average M_w 31000–50000). The concentrations tested were 150, 200 and 300 $g \cdot L^{-1}$ for Rhodoviol 25/140, and 150 $g \cdot L^{-1}$ for the two other PVALs. The PVAC emulsions tested were Savatex 18, 385 ST, 239 M, FS and EO1. The formulation was then

fine-tuned by adding two organic compounds which acted as plasticizer and photosensitizer.

Two papers were selected as possible supports: a) a highly calendered glossy non-absorbent paper made of bleached kraft pulp (100% cellulose), known under the commercial name of Bristol (B); and b) a resin-coated photographic paper Multigrade IV FB fiber (RC), laminated with a polyethylene film on the verso side, which was the side used for coating. Although, ultimately, only one support could be chosen, all the characterization experiments on the dosimeter were carried out on both supports in parallel. The sensitive coating mixture was applied on the support by spin-coating for small-scale production in the laboratory, whereas, in the final phase, for large-scale commercial production, bar-coating was used.

Light-ageing equipment and protocols

Series of accelerated light-ageing tests were performed using different equipment set-ups and varying the light source features and the overall ageing conditions. Two kinds of accelerated light-ageing tests were carried out: a) very high light intensity and short exposure time, used especially in the first phase of the research to investigate the responses of different formulations; and b) moderate intensities and long exposure time, designed to simulate, at least to a first approximation, real exposure conditions. The different equipment set-ups used were as follows.

A light-ageing chamber with adjustable temperature and humidity control was used for the exposures to very high intensity outputs. The chamber functions were controlled by SIRPAC 2000 v.2.26 software. The nominal illuminance at 0.8 m distance from the source was 1000 $W \cdot m^{-2}$ (1100 $W \cdot m^{-2}$ effective, i.e. approximately 193 klux). The light source was a metal halide lamp Solarconstant HMI® 2500 W (± 1%), colour temperature 5600 K (± 150 K). In accordance with ANSI Standard IT9.9-1990, a soda lime float glass filter, 6.5 ± 0.5 mm thick, was located below the lamp to simulate interior light through a glass window [23]. The temperature of the chamber could be adjusted in the range 10–100°C (± 1°C), and relative humidity (RH) in the range 25–97% (± 3%) within the temperature window 10–90°C. Unless otherwise specified, the conditions in the light-ageing chamber were set to 23°C and 50% RH. This light-ageing chamber could achieve complete bleaching of LCU samples after an exposure of 30 minutes.

The dosimeter prototypes were also tested under different light sources commonly found in indoor situations, such as tungsten-halogen, fluorescent, natural light and combinations thereof. Different light intensity ranges were investigated. To obtain an intermediate intensity output, a fluorescent light rack was used. The rack consisted of four daylight TFP Prestiflux 18 W JR/865 tubes, with nominal luminous flux of 1300 lm, enclosed in a wooden box. The output from the four tubes was 5 klux – and about 0.05 $W \cdot m^{-2}$ ultraviolet (UV) A component – as measured on the surface of the sample (50 cm vertically from the source) and was homogeneous across a 20 × 20 cm surface in the centre of the illuminated field. The box was placed in a controlled climate room (23°C, 50% RH), and the temperature inside the box was 27°C.

The response to indoor illuminance levels was also a very important part of the testing. For these tests, focused on lower intensity outputs, tungsten-halogen lamps were selected (Halostar longlife, 12 V, 20 and 35 W, colour temperature 3000 K). The lamps were made of doped quartz crystal that blocks UV radiation totally in the range 300–350 nm, and partially (50%) in the range 350–380 nm. Tungsten-halogen with UV filtering was chosen as the reference light source for the complete characterization and calibration of the dosimeter, as well as for the verification of the reciprocity law, because it is one of the light sources most commonly used in museums. It should be remembered that LCU was envisaged to be used to monitor very light-sensitive artefacts that would not normally be exhibited in a museum context under high-intensity lighting, and should be protected from UV radiation [24, 25]. Taking into account this intended application, the response of LCU was carefully investigated in the visible range, whereas its response to UV radiation was less fully explored. Likewise, preliminary spectral characterizations showed that the dosimeter did not exhibit absorption bands in the near-infrared region. Based on these findings, the use of dichroic mirrors to block the infrared component of the radiation of the tungsten-halogen bulbs was deemed unnecessary. Bare lamps were preferred, to ensure optimal homogeneity of the illumination field in which the samples were exposed. Luminous flux intensities tested with the tungsten-halogen sources were set in the range I = 50–700 lux. The intensity value was moderated by using circular glass metallic neutral density filters (homogeneous filtering in the range 400–2000 nm), with optical density (OD) = 0.7, 1 and 1.5. All the exposures were carried out in controlled climate conditions (23°C, 50% RH), either in the open air or in tailor-made wooden boxes in which the lamps were mounted. In the light boxes a fan for air ventilation ensured no overheating at the sample surface.

Table 1 *Environmental conditions of the various exposures to which LCU samples were subjected, collected with data loggers*

Illuminant		Luminous flux intensity (lux)	UVA	T (°C)	RH (%)	Exposure conditions	Exposure location
(1) metal halide	mean	193000	18000[a]	25	50	controlled & stable	laboratory
(2) tungsten-halogen	mean	100	0	23	50	controlled & stable	laboratory
	mean	200	0	23	50	controlled & stable	laboratory
	mean	500	0	23	50	controlled & stable	laboratory
(3) fluorescent	mean	5000	47[a]	23	50	controlled & stable	laboratory
(4) tungsten-halogen	min	33 (day)	0	20	26	varying (T, RH)	field
	max	40 (day)	0	34.5	71.5	constant light (day)	
	mean	40 (day), 15 (day/night)	0	26	50	day/night cycles	
(5) natural	min	0	0	16.5	23	varying (T, RH, light)	field
	max	1407	506[b]	27.5	62.5	day/night cycles	
	mean	117	59[b]	20	45	no direct sunlight	
(6) mixed (natural,	min	0	0	19	16	varying (T, RH, light)	field
halogen, fluorescent	max	1010	165[b]	29.5	65	day/night cycles	
components)	mean	42.5 (day/night)	3.5[b]	24.5	36.5	no direct sunlight	

The values of luminous flux, UVA, T and RH were monitored with data loggers during the entire exposure periods. a: $\mu W \cdot m^{-2}$; b: $\mu W \cdot lm^{-1}$.

Table 1 summarizes the environmental conditions of the various exposures to which LCU samples were subjected during the tests.

In each experiment, data logger units were placed near the samples in order to monitor continuously the different environmental parameters (illuminance, temperature, RH and UV radiation). Data were sampled every 10 minutes through the entire duration of each exposure. Based on the illuminance data acquired, the LD received by the samples was calculated, to establish the quantitative relationship between LD and the colour change occurring on the LCU. The other environmental data (temperature and RH) were used to characterize the average microclimatic conditions to which the dosimeter was subjected and their possible impact on the colour variation.

In the final phase of the project, once the dosimeter was fully characterized, a set of tests was performed to build the calibration and a reference colour scale (RCS) intended to provide a rapid assessment of the degree of fading of the LCU. Three sets of colour scales were prepared, which included different stages of progressively aged LCU samples covering the LD range 0–100 klux hours, considered that of practical interest. These colour scales were prepared by exposing the LCU samples under fixed illuminances (I = 100, 300 and 500 lux), varying the exposure time in order to obtain increasing LD values. This also served to demonstrate the reciprocity principle.

Finally, the light-ageing programme also included a series of natural exposures in the field, made in selected museums. These tests were used systematically to corroborate and interpret the results obtained with the accelerated light-ageing tests in the laboratory. These field tests were all carried out according to the same protocol, to ensure comparability among the data from samples exposed in different museums and, thus, in different conditions. Typically, each field trial included LCU samples from the same batch, as well as BWS no. 1, which were simultaneously exposed next to selected artefacts at different sites for a given period (variable from one week to a few months).

Optical measurements

Methodology

As the dosimeter response was based on a light-induced colour change, colorimetric analysis was the main tool adopted for the characterization. Diffuse reflectance spectrophotometry was selected as the main technique. Indeed, the reflectance spectrum in the visible region is the basic information for calculating the colorimetric coordinates [26]. At the same time, the raw reflectance spectral data provides complementary information in the study of the fading process characteristic of the dosimeter.

As the samples to be characterized were highly light-sensitive, it was a concern that they could undergo a colour modification during the measurement. Non-invasiveness was therefore another criterion considered in the choice of the characterization technique. The experimental set-up and the measurement protocol selected guaranteed that no colour alteration due to the

instrumental light source was induced in the samples. A standardized measurement protocol was established in order to guarantee reproducibility in the measurements and comparability between data sets collected on different samples (LCU and BWS have different textures and surfaces) and/or at different times.

Instrumentation

The reflectance spectra were collected in diffuse mode (i.e. excluding the specular component of the reflected signal). A Lambda 19 (Perkin-Elmer) double beam ratio spectrophotometer, with ± 0.2 nm resolution in the UV-visible range, was used. The spectrophotometer, configured in reflectance mode, was equipped with a 60 mm integrating sphere, in the geometry 8°/D (diffuse). A light-trap accessory in the sphere was used to exclude the specular component of the reflected beam. The calibration (both wavelength and signal) was repeated before each measurement session. A 99% reflectance calibrated Spectralon® standard was used as white reference for the diffuse reflectance measurements, and a Holmium Oxide Spectralon® standard was used for the wavelength calibration.

The reflectance measurements were performed in the 350–860 nm spectral range, with 1 nm steps. This spectral resolution was considered a satisfactory compromise to provide a fairly highly resolved spectrum and spectral data suitable for colorimetric calculations.

A hand-held X-rite SP 64 spectrophotometer, equipped with integrating sphere was occasionally used for on-site colorimetric measurements when the bench instrument could not be used. Again the configuration adopted was in reflectance mode in the 400–700 nm spectral range, with 10 nm steps, with 5 mm diameter aperture; in this case the specular component was included.

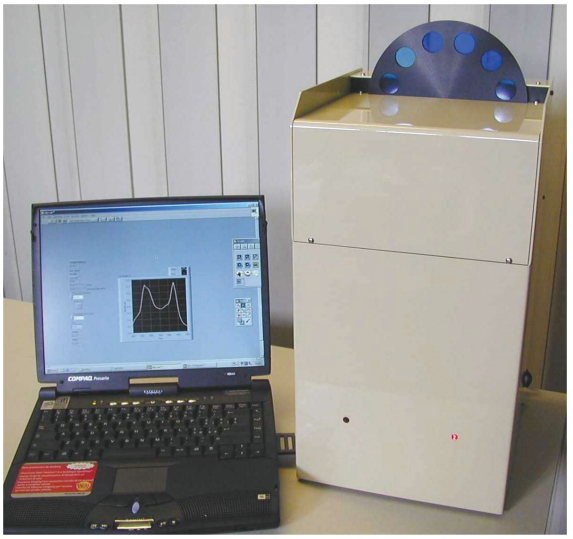
A novel instrument, specifically designed for light-monitoring in museums, was also used for tests aimed at investigating the effects of light/dark (day/night) cycles on the fading features of LCU. The instrument is based on a miniaturized spectrophotometer equipped with optical fibres (see Figure 1) [27]. The system is conceived to follow spectral and/or colour changes in specific samples in a given environment. Samples housed in the upper half of the wheel holder are exposed to the external environment, whereas the other half faces the fibre-optics acquisition system within a dark case. A periodic rotation of the wheel allows measurements to be taken from the exposed samples at the desired frequency so that their light-induced changes can be monitored in time. The instrumentation performs reflectance measurements and specific software produces the corresponding colorimetric data. Initially this instrumentation was designed to be used with the ISO Blue Wool Standards [28].

Experimental and data-processing protocol

As mentioned earlier, a fixed experimental protocol was followed for spectral characterization of all the samples used during the project. The protocol included standardization of specimens' shape and size, which allowed reproducibility of the geometrical conditions in the reflectance measurements as well as exact repositioning in subsequent measurements. The standardized procedure involved the collection of reflectance spectra for each sample, before (t = 0) and after (t = x) any light-ageing test or field exposure. For each set of aged samples an unaged specimen, stored in the dark, was retained as control and measured in the same context as the other samples at t = 0 and t = x. The spectral data were processed following two approaches which provided complementary information:

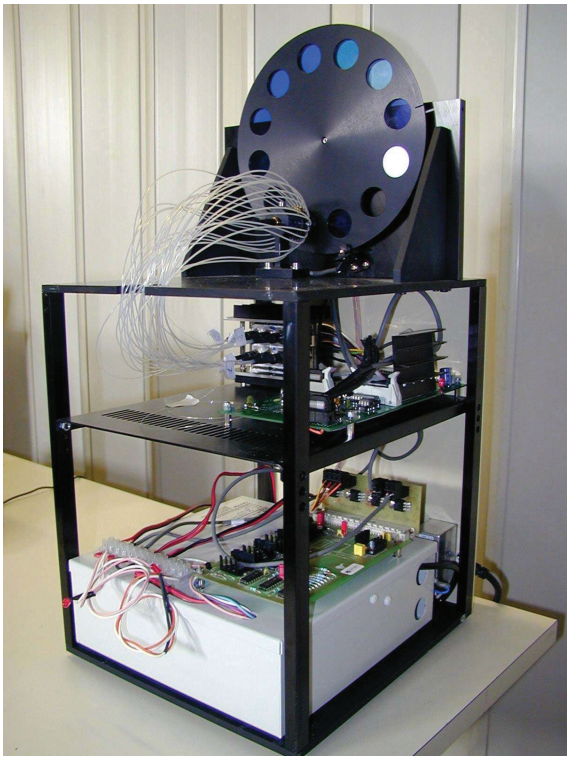
- *Colorimetric analysis* was used for the quantitative assessment of the colour variation induced in LCU samples by the light-ageing tests (natural and artificial). The colorimetric coordinates values (L,a,b)* were calculated using the CIELab76 Colour System[1], with the D65 standard illuminant and 10° standard observer [26]. Based on the (L,a,b)* values measured before and after light-ageing, the total colour change (ΔE*) that had occurred on a given sample was calculated and was related to the LD provided by the data logger.
- *Spectral bands analysis* was used to investigate the fading mechanism of the dosimeter, the rates of decay of the dyes in the photosensitive coating, the sensitivity threshold lower limit and the saturation light levels of the dosimeter.

[1] *Following the introduction of the CIELab76 Colour System, further improved and more sophisticated formulas – namely the CIE94 and CIE00 Systems – have been proposed by the CIE for more accurate colour difference evaluations, and especially to calculate very small colour differences. In the present context it was considered more appropriate to use the CIELab76 Colour System, since it remains the most commonly used system in many areas of research and it is absolutely suitable for applications in which sizeable colour differences are measured [29, 30].*



(a)

(c)



(b)

Figure 1 *LCU indicator: (a) the fibre-optic based spectrophotometer instrument; (b) detailed view of the optoelectronic unit normally enclosed in the protective case; and (c) the instrumentation in operation on-site, in the Geographic Maps Room at the Uffizi Gallery, Florence (January 2002).*

RESULTS AND DISCUSSION

Formulation and optimization

The main selection criteria in the formulation of the LCU indicator were related to how the fading evolved with light exposure (i.e. how the colour changed with increasing LD, as well as the speed of fading). The sensitive coating had to be responsive in the desired range of LD and to display a characteristic and clearly distinguishable colour variation.

The same dyes were used as in the patented version of the dosimeter [17]. The dyes were dissolved separately in water. Small volumes of each solution were mixed together with the required additives to form a new solution, which was slowly poured into the polymer viscous phase. This resulted in a homogeneous coating, presenting no phase separation or aggregation of the dye.

Besides the nature of the dyes, other chemical and physical factors are involved to various extents in the

fading of the sensitive coating layer. These factors depend on the polymer, the additives and the support. For instance, the viscosity of the coating mixture had to be such that an optimal coat thickness would be achieved after drying. The thickness of the active layer was important, as the time necessary to reach total fading is proportional to the dye concentrations in the medium [31]. Other aspects to take into account were the flexibility of the coating layer upon drying (in order to avoid cracks) and the inertness of the support.

Besides technical aspects, the materials chosen in the formulation also had to suit economic and market requirements. For large-scale production, the polymer chosen should be available in the long term at moderate cost. A raw polymer base with no additives was thus preferred. This would also provide the autonomy to determine the most appropriate chemical additives (plasticizers and photosensitizers). Another important aspect to be considered was the innocuousness of the materials used to human health and to the environment.

Based on all these aspects, two classes of polymers were considered initially: PVAL and PVAC. This choice was based on the fact that both are synthetic polymers commonly used by industry for a variety of applications. PVAC emulsions are the most widely employed adhesives on the market and are also used in paints, textile sizing and paper coatings. PVAL is water-soluble and can also be used as an adhesive, for sizing and finishing of textiles and for paper coatings. PVAL is commercialized in a wide range of molecular weights (resulting in a wide range of viscosities). Among the PVALs tested, the best results were obtained with Rhodoviol 25/140. Among the PVACs, Savatex EO1 was found to be the most suitable as it presented optimal working viscosity and, in contrast to the other PVACs, it was made exclusively with the homopolymer. However, both Rhodoviol 25/140 and Savatex EO1 resulted in somewhat brittle coating films upon drying (at the optimized concentration of 150 $g \cdot L^{-1}$), and it was found necessary to add a plasticizer. A common additive used in the polymer industry was tested at several concentrations ranging from 5 to 40% (vol/wt matrix).

In order to enhance the sensitivity of the coating, several chemicals were tried in various concentrations as potential photosensitizers. Organic acids were tested because numerous dyes are sensitive to pH [32]. Acetic acid (1 M), citric acid (5×10^{-2} M) and oleic acid (72% purified, assay of fatty acids 97%) were added to the polymers at three different concentrations: 2.5, 5 and 10% (vol/wt polymer). Unfortunately, none of the acids tested had a significant influence on the fading. Surfactants, both cationic and anionic were tried next, at 2.5, 5 and 10% (vol/wt polymer). The anionic surfactant was found to be the most effective, as not only did it accelerate the fading, but additionally, in combination with the plasticizer, it produced a significantly enhanced quality in the colour span obtained upon exposure to light.[2]

Concerning the support, the two papers (B and RC) provided a layer thickness (spin-coated) of 100 ± 5 μm as measured by optical microscopy on cross-sections. Five micrometres was thus considered the threshold tolerance for thickness variability. However, the fading rate was faster when the coating was applied on RC than on B. The drawback of RC was that the coating layer took longer to dry and was more sensitive to surface abrasion. However, as sensitivity was the chief criterion, RC was chosen as final support.

The outcome of the optimization phase led to the following formulation for preparing a small batch of the LCU coating: a solution containing the dyes and the additives was slowly incorporated into 20 g PVAC Savatex EO1. The solution was prepared by mixing Toluidine Blue O, certified (separate solution at 5 $g \cdot L^{-1}$ in water), Eosine 225 (separate solution at 3.97 $g \cdot L^{-1}$ in water), the plasticizer (10% vol/wt matrix) and the photosensitizer (2.5% vol/wt matrix) [17]. A batch of this mixture was sufficient to spin-coat one piece of RC of 20 cm diameter, from which about 10 LCU test samples can be made. The drying took place in the dark for four days at ambient conditions. The experiments reported in the following sections were carried out on samples prepared according to this final formulation, unless stated otherwise.

Reproducibility of preparation of the coating

The light indicator was designed to become a common tool for the assessment of the light environment and light damage to cultural heritage artefacts and to be widely available to art preservation professionals. As such, it had to be a robust system: precise, accurate, selective and reproducible. An important aspect of the testing was the colour reproducibility of the coating. At the initial stage (t = 0), all the dosimeters prepared had to display the same colour in order to ensure the same behaviour towards light.

[2] *The identities of the plasticizer and the sensitizer cannot be revealed here because of the existing patent FR2784458 [17].*

The reproducibility of the colour encompasses two main aspects: a) the sample-to-sample variation within a given batch, which is related to the colour difference between different locations on a single coated surface; and b) the colour reproducibility from batch to batch, which is an evaluation of the tolerance in the colour difference between samples prepared from different batches at different times.

Depending on the volume of the batch needed, different coating procedures were used to prepare LCU (spin-coating for laboratory tests, and bar-coating for industrial preparation), so the reproducibility tests were performed with both small-scale (laboratory) and large-scale batches. The reproducibility was evaluated on the basis of the colorimetric analysis.

Laboratory-scale batches

First, the reproducibility in the preparation of the dosimeter using small quantities of the coating base was investigated. Several samples were prepared on different days, using different coating batches, and the sample-to-sample variability was evaluated by calculating the colour differences $\Delta E^\star$ between the various samples. It was found that even small variations in the preparation of small batches of coating base could lead to a wide dispersion in the colorimetric parameters, with $\Delta E^\star$ varying from 5.8 to 13.6.

The colour homogeneity within a single sample was investigated, and it was found that the average $\Delta E^\star$ between different points within a single sample was very small ($\Delta E^\star<2$). Thus, it was concluded that the homogeneity of the sensitive coating base on the support for separate samples (i.e. the coating thickness) was very good. Conversely, the robustness in colour reproducibility from batch to batch required further improvements.

Large-scale batches

Based on the observations above, it was supposed that the problem of poor small-batch reproducibility could be circumvented in the commercial-scale preparation, where a large number of dosimeters are obtained from a single, large coated paper sheet. Therefore, special attention was given to the homogeneity of the colour in the final phase of the project when the preparation of the samples was transferred to Particle Technology[3], a commercial partner in the LiDo project and, at present, distributor of the product.

Two large coating batches (10 L) were prepared, one to be used in the laboratory spin-coating procedure and the other to be used with the industrial bar-coating procedure at Particle Technology. The sets of laboratory-coated and industrially coated samples were evaluated by colour measurements. Ten randomly selected locations were measured on each sample and the average $(L,a,b)^\star$ values were taken as the mean colour of the sample. The homogeneity within a single sample was assessed by evaluating the colour difference $\Delta E^\star$ between each of the ten spots and the mean value.

Tables 2 and 3 gather the results obtained with the industrially coated samples. $\Delta E^\star < 3$ was found in all cases (Table 2). The sample-to-sample differences were also evaluated based on $\Delta E^\star$ (Table 3). The results showed that using the same (large-scale) coating base

Table 2 *Industrially coated LCU samples: differences within samples (6 samples, 10 measurements per sample)*

Sample	ΔE^* mean	ΔE^* min	ΔE^* max
1	0.8	0.1	1.6
2	1.3	0.5	1.7
3	1.5	0.2	3.3
4	0.4	0.1	1.0
5	0.8	0.1	3.0
6	0.8	0.2	1.3
average	0.9	0.2	2.0

Table 3 *Industrially coated LCU samples: sample-to-sample differences (6 samples, 10 measurements per sample)*

Sample	ΔE^* mean	ΔE^* min	ΔE^* max
1	1.5	1.1	3
2	1.1	0.2	2.6
3	1.4	0.7	3.4
4	1.8	0.7	4.1
5	1.1	0.2	2.4
6	2.3	1.1	4.1

[3] *Particle Technology Ltd, Station Yard Industrial Estate, Hatton, Derbyshire, DE65 5DU, UK; www. particletechnology.com.*

[4] *As a general indication, for the human eye, a barely perceptible colour change has $\Delta E^\star \sim 1$, even though the differences appreciable by the human eye also vary according to the hue and other complex parameters [26, 33]. In the present context, the acceptability of the tolerance in $\Delta E^\star$ was established by considering that LCU is designed to be evaluated visually by the end-user, who compares it with a reference colour scale. For that reason, and also because colour perception depends on other parameters such as the lighting conditions, a less strict criterion for the tolerance on $\Delta E^\star$ is acceptable in this case.*

batch, the reproducibility in the production of LCU was acceptable, always resulting in $\Delta E^{\star} < 3$.[4]

These tests demonstrated that the formulation proposed for the preparation of LCU was suitable to guarantee reproducibility and stability in colour at t = 0, once the production process was standardized and carried out on a large scale.

Stability during storage in the dark

LCU samples kept in the dark under ambient conditions were regularly measured. LCU proved to be very stable with $\Delta E^{\star} = 1.3$ at t = 28 days with respect to t = 0, and showed almost imperceptible change after one year ($\Delta E^{\star} < 2$). LCU can thus be safely stored in the dark under ambient conditions for several months.

The behaviour of LCU in the dark was also tested under severe temperature and humidity conditions (70°C, 70% RH). Figure 2 shows the gradual yet significant change in colour from t = 0 to 22 days ($\Delta E^{\star} = 5.3$ after 6 days and $\Delta E^{\star} = 23$ after 22 days). Temperature and humidity can thus play an important role in the colour alteration path, even though this effect could be observed only under severe conditions which fall outside of the targeted application range of LCU. Therefore, in typical museum environments, temperature and RH can be considered factors of secondary importance compared with light in determining the LCU response curve.

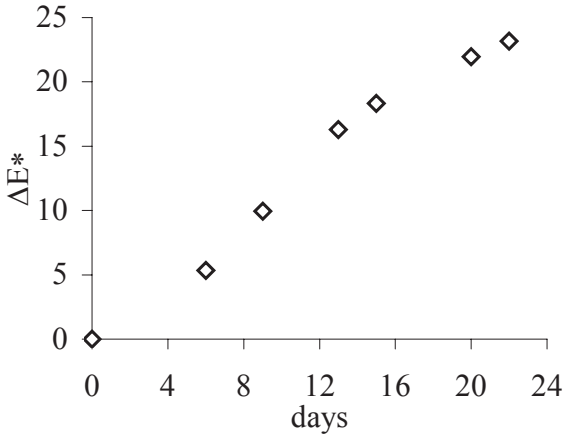


Figure 2 *Colour change ΔE* of LCU as a function of time of exposure to a temperature of 70°C and 70% RH in the dark.*

Light-induced effects

Fading process, sensitivity, and definition of operational LD range

The investigation of the fading mechanism of the dosimeter was fundamental for the determination of its characteristic operational range (i.e. the range of LD values over which a reliable and quantifiable response can be expected). The operational range is highly dependent on the formulation of the active coating layer. Slight variations in the composition can give rise to different (faster or slower) photochemical reactions. Thus, the final formulation was selected taking these aspects into account and, in particular, the fact that the response of the dosimeter to very low and moderate LDs had to be progressive.

The reflectance spectra of LCU colour scales were obtained with accelerated light-ageing tests. The different stages of colour corresponding to increasing LDs undergone by the LCU samples were analysed with a focus on the light-induced spectral variations. The spectroscopic behaviour underlying the progressive change of colour was analysed; details of this study are reported in a previous publication by the authors [31]. In particular, it was shown that the rate of fading of the blue dye was approximately twice that of the red dye. This explains the characteristic colour change, which progresses from blue to purple, then pink. The fully bleached white stage can be reached upon prolonged exposure beyond the operational range of the dosimeter.

The study of the decay rates of the dyes also determined the typical LD range over which each dye accomplishes its photochemical fading. It was calculated that to extinguish the active dye mixture in the LCU an exposure to a LD of about 100 klux hours was needed. This was therefore defined as the maximum LD, beyond which the dosimeter reached saturation.

The lowest detectable LD (i.e. the minimum LD required to initiate the fading process) was also explored. The value of LD required to produce a detectable colour change was extremely low (5 klux hours), confirming the high sensitivity of the light sensor. Based on this characterization, it was established that the dosimeter was suitable to respond over the operational LD range of 5–100 klux hours.

Besides the spectroscopic study, a complementary evaluation of the operational range of LCU was carried out by considering the colorimetric curve. In Figure 3 the plot of $\Delta E^{\star}$ versus LD shows the typical behaviour of LCU upon exposure to increasing LDs. A progressive

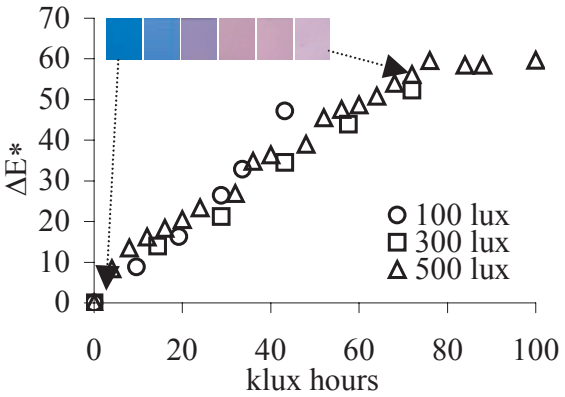


Figure 3 Colour change ΔE* as a function of LD for three sets of LCU samples aged at different illuminance values (light source: tungsten-halogen lamp). The colours displayed by LCU as the LD received increases are also shown on the graph.

colour variation occurred as luminous exposure increased, until the saturation (plateau) was reached corresponding to the final colour of the dosimeter. The colorimetric response confirmed the results of the spectral bands analysis and the same operational range was obtained.

Another aspect to be investigated was the reciprocity principle fulfilment [24], at least in the typical exposure conditions of the museum environment. In other words the LCU colour change should depend only on the product of the source illuminance and the exposure duration (I × t) [1, 2].[5] Several tests were performed to verify this aspect by varying the illuminance of the source in the range of interest for practical applications. Figure 3 shows the results obtained with I = 500, 300 and 100 lux.

Since the final goal of the research was the development of a new light-monitoring tool, the performance of the dosimeter was also compared with that of BWS. The higher sensitivity of LCU with respect to BWS no. 1 was verified with a comparative light-ageing test, using the same exposure conditions. This test also included the other sensor developed within the LiDo project, LCS. The results showed that the sensitivity range covered by the two dosimeters, LCU and LCS, was perfectly complementary to that of BWS, leading to their being named the 'LightCheck System'. In particular, the LCU was found to be the more sensitive indicator, suitable to respond clearly to LD levels undetectable by BWS no. 1. As an example, a colour variation of ΔE* ≈10–12 could be obtained by exposing LCU to LD ≈12 klux hours, whereas LD ≈ 250 klux hours was needed to obtain the same effect on BWS no. 1 [22, 28]. This fundamental achievement was corroborated by the natural ageing tests performed during the field exposures in selected museums.

Influence of light source and lighting conditions

The photochemical reaction involved in the LCU discolouration could depend, in principle, on the spectral power distribution of the light source impinging on the indicator. This aspect was taken into account by testing the LCU response to different light sources (selected from those most typical for lighting in museums), as well as considering natural lighting conditions, and dark/light cycles.

Table 1 lists the different light sources and climate conditions to which LCU samples were subjected. Figure 4 shows clearly that the overall luminous exposure was the main factor determining the colour variation of LCU. Indeed, with the exception of fluorescent and metal-halide sources, the curve of ΔE* as a function of LD was the same upon exposure to various light sources and luminous flux, whether under continuous or cycling light and/or different climate conditions (see below). As shown in Figure 4, the exposure under fluorescent light led to a slightly different curve shape, with a lower slope and a plateau (corresponding to the saturation regime) reached upon higher LDs than with the other light sources tested. Nevertheless, this may not be a critical point when fluorescent light is present as a minor light source. Indeed, under mixed light sources – where a small proportion of fluorescent light was present – the response of LCU was identical to that under 100% tungsten-halogen light and 100% natural light. Thus a minor component of fluorescent radiation in the light source can be neglected in the colour path variation of LCU.

Upon exposure to the metal-halide lamp (in accelerated ageing tests under very high illuminance, I = 193 klux) the colour variation path was also rather different from that obtained with lower light outputs,

[5] *The reciprocity principle is based on the assumption that 'the factor responsible for change is the total amount of energy absorbed by the object rather than the rate at which the energy is absorbed' [2]. Hence, with a given light source, the colorimetric response of the sample should depend on the total LD received and should be independent of the illuminance.*

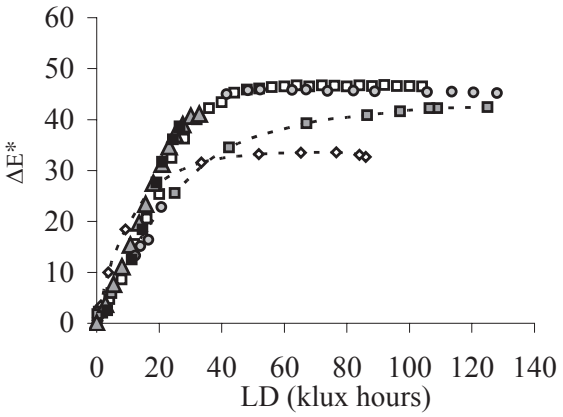


Figure 4 ΔE* as a function of LD of LCU samples upon exposure to different light sources and diverse climatic conditions. Legend: --◇-- metal-halide 193 klux (1), □ tungsten-halogen 500 lux (2), --▩-- fluorescent 5 klux (3), △ tungsten-halogen 40 lux (4), ● natural (5), ■ mixed natural + tungsten-halogen + fluorescent (6) (the numbers correspond to those in Table 1).

showing a faster discolouration and a lower value of ΔE* corresponding to the saturation regime (Figure 4). The most likely explanation is the discontinuous spectral distribution of the metal-halide lamp and the very high value of illuminance used. These effects were consistent with other tests performed under severe conditions. As fluorescent light sources are also characterized by a discontinuous spectral distribution, it can be speculated that the differences in the behaviour of LCU under these lights, compared with that under continuous light sources, may be due to the generic difference between black body spectral responses and responses to sources with discontinuous spectra. The metal-halide lamp was used mainly in the screening phase for the formulation and optimization of the indicator, but it was not the main tool for the characterization of the light-induced effects on LCU. For the practical use of the dosimeter, the response curve studied was that simulating typical museums lighting conditions (tungsten-halogen source, with moderate illuminance values).

In principle, another factor that can affect the rate of the photochemical reaction in LCU is UV radiation. As LCU is targeted to very light-sensitive collections, displayed in UV-protected environments, the sensitivity to UV of the dosimeter was not investigated in depth. However, at an early stage in the project, exploratory spectral analyses were carried out. The accelerated ageing tests were performed with very high illuminances in order to check roughly the reactivity of the light sensor formulation to UV radiation, but certainly do not represent real lighting situations in museums. The results showed that the behaviour of the blue dye was partially affected by the UV portion of the spectrum while the red dye was unaffected. Considering that UV levels in a museum are very low, when not totally absent, it can be supposed that under practical in situ conditions the reactivity to the visible portion of the spectrum of the blue dye overrides its sensitivity to UV. This working hypothesis was retained in all the tests made.

Colour regeneration in the dark

Since a slight tendency to post-exposure colour recovery was observed when the indicator had been exposed to very high illuminances (> 100 klux) for short periods of time, the possible effects of colour regeneration in the dark were investigated [34]. Obviously, any colour recovery effect might compromise the reliability of quantitative LD evaluations of the dosimeter. First, the effect of light/dark variations simulating daily cycles was investigated. The behaviour of LCU samples exposed in the fluorescent light-ageing box (5 klux) under both continuous and intermittent luminous flux (cycles of six hours light and six hours dark) was studied (Figure 5). The two curves, essentially identical, confirmed that no colour recovery occurred in the dark after a moderate-intensity light exposure.

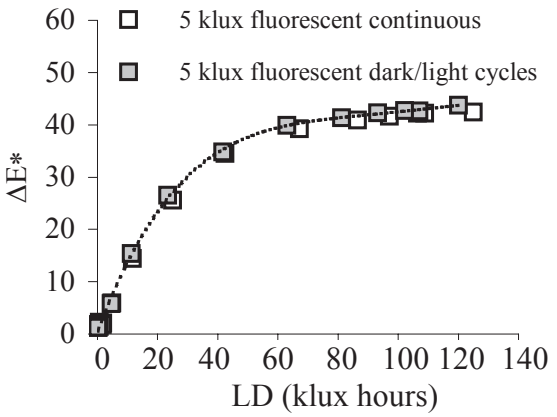


Figure 5 ΔE* as a function of LD of LCU samples upon exposure to fluorescent light source with continuous exposure and discontinuous exposure dark (six hours)/light (six hours).

Another experiment in which samples were exposed to tungsten-halogen light under both continuous and cycling conditions was carried out (Figure 4 and Table 1). In this case the situation was more complex, since it included samples exposed in the laboratory (continuous fixed exposure) and samples exposed in situ in a museum (Musée Cognaq-Jay, Paris). The continuous exposure was carried out in the light box, at illuminance I = 500 lux and under stable temperature and RH conditions. Conversely, in the museum room the samples underwent light cycles due to day/night periods and public opening/closing hours, as well as daily variations in temperature and RH since the room was not climate-controlled (Table 1). Moreover, the lighting was provided by a tungsten-halogen source, as daylight from a nearby window was fully blocked by a screen. The average light intensity during the day was 40 lux. Despite the variability of these factors, the two types of exposure led to a similar colour variation of LCU as a function of LD, as shown in Figure 4. This confirmed that even under complex environmental situations, such as in situ exposures, the LCU colour changes were mainly driven by the LD received.

These results were corroborated by a more in-depth study carried out using the rotating instrument for light monitoring in museums (Figure 1). Thanks to the automated acquisition system, it was possible to follow the colour changes of the LCU samples exposed to cycles of natural light. An exposure of 18 days at room conditions was performed (natural light exclusively from a window, no direct sunlight). The spectral acquisition on the LCU was performed twice a day: after sunset and before sunrise. The results are given in Figure 6, where ΔE^* of the exposed sample with respect to a reference is reported as a function of the exposure time. An identical value of ΔE^* was recorded before and after the night, indicating that, under usual daylight/night cycles, no regeneration of the colour was observed.

Obviously, these results do not exhaustively tackle the complex topic of colour regeneration. Nevertheless, they were sufficient to conclude that under normal exposure conditions (ambient RH and temperature) and in natural daylight/night cycles, LCU did not undergo colour regeneration.

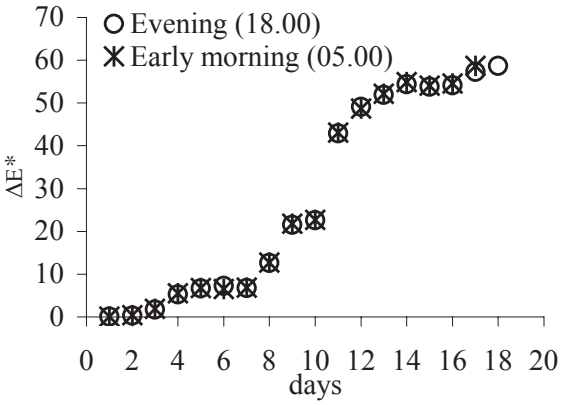


Figure 6 *Experimentation with the fibre-optic-based instrumentation shown in Figure 1 designed to verify the absence of colour regeneration phenomena on LCU. The cumulative colour change ΔE* of an LCU sample upon intermittent exposure in the Geographic Maps Room at the Uffizi Gallery was monitored twice per day, at 18.00 and 05.00.*

Impact of the support on colour alteration

In an ideal situation, the support should be physically and chemically inert and only the photosensitive layer should have a role in the fading process. In order to verify this, the responses of two LCU samples coated on the two different supports, namely polyethylene coated paper (RC) and calendered Bristol paper (B), were studied. RC provided a slightly higher dosimeter sensitivity than B, with a slower progression towards the saturation plateau. A close examination under the microscope of cross-sections of the two LCU samples showed evidence that with the B support cellulose fibres on the surface had slightly absorbed the dyes, while RC was totally impervious to the polymer mixture. The difference in behaviour was thus explained by the fact that the dyes could bind to cellulose fibres and become less sensitive to alteration by light. This finding confirmed that the support can play a role in the colour change.

It was thus decided that RC was a more appropriate support than B. Nevertheless, the possibility of using B was not totally discarded. Its lack of total inertness was considered a potentially useful feature, which could eventually be exploited to add versatility to the system without affecting the quality of the light-induced colour variation.

Impact of the matrix on colour alteration

Unlike the support, the matrix was of course not deemed to be inert. The matrix primarily acted as the embedding material, but practically also played a role in the fading process. Light-ageing tests were performed with samples prepared with the two matrices, Savatex EO1 PVAC and Rhodoviol 25/140 PVAL (150 $g \cdot L^{-1}$),

with all other components identical to the final formulation. As the response to tungsten-halogen (I = 300 lux) was similar in both cases, PVAC was selected for the final formulation of LCU for practical reasons (ease of the preparation).

Other environment-induced effects on colour alteration

It is well established that several photo-deterioration mechanisms are affected by the synergistic action of various environmental parameters, and that the overall damage to an object on display cannot be correctly evaluated by considering all these factors independently [24]. For instance, it has been shown that thermo-hygrometric conditions can significantly influence the rate of fading of BWS no. 1 [28]. Based on this fact, a set of tests was performed to study the role of some environmental factors other than light – such as temperature, RH and selected pollutants – on the LCU fading process.

Temperature and RH

The influence of temperature and RH on the colour variation of LCU upon exposure to light was investigated using the light-ageing chamber with fixed illuminance (I = 193 klux). Figure 7 shows that an increase in temperature in the range 20–40°C (at stable 50% RH) resulted in a faster discolouration, as well as the saturation regime being reached at higher ΔE*. At constant temperature (20°C), a variation in RH in the range 30–70% had no visible impact on the colour span with increasing light exposure.

Oxygen

The use of oxygen-free environments in museums is most often relevant to pest control and management [35]. Recent trends in preservation management have also explored the possibility of storing or exhibiting very fragile or special artefacts in anoxic environments [36]. Candidate objects for such measures are those consisting, partly or totally, of materials that are particularly prone to oxidative degradation, such as lignin-containing papers, cellulose nitrate and a large number of colorants. It is known that numerous dyes and pigments are sensitive to oxidation/reduction processes [3]. These reactions can be initiated by photons (photo-oxidation, photo-reduction), and are often accompanied by different colour alteration phenomena.

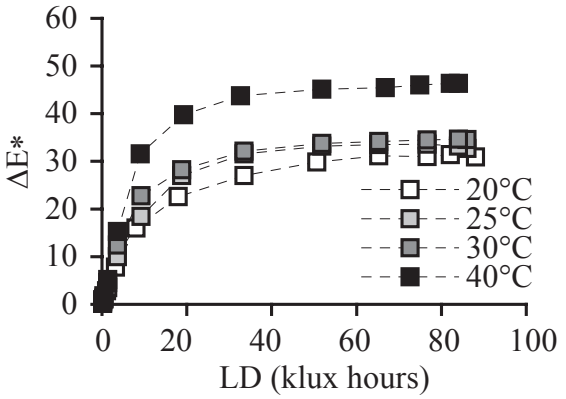


Figure 7 *ΔE* as a function of LD of LCU samples at varying temperature and constant RH upon exposure in the light-ageing chamber (I = 193 klux, light source: metal halide lamp).*

These considerations suggested an exploration of the effect of an oxygen-free environment on the fading process of LCU. This would also allow some insight to be gained into the degradation mechanism of the dyes, and thus specific recommendations to be made concerning the limits of use of LCU. In addition, the high fragility of artefacts exhibited in anoxic conditions would be a major reason for low light exposure and special care, which would justify the introduction of LCU in a light-monitoring programme of such artefacts.

The LCU samples were packaged in sealed transparent plastic film bags with low oxygen permeability, both with and without oxygen absorbers and indicators, and their colour variation was evaluated. The plastic film was a high-barrier laminated film made of oriented polypropylene/vinyl alcohol-ethylene/polyethylene (thickness 20/15/40 μm), with a permeability to O_2 in the range 0.5–3 $cm^3 \cdot m^{-2} \cdot atm^{-1}$ per day (at 23°C, 85% RH). After packaging and before exposure to light, the samples were left standing for several hours in the dark in order to ensure that all the oxygen was effectively removed from the package containing the oxygen absorbers. The samples were exposed in the light-ageing box, under the fluorescent light rack, at I = 5 klux for four hours (LD = 20 klux hours). The data point of the reference sample, packaged without oxygen absorbers (unsealed plastic bag) fell exactly on the corresponding colour variation curve, which validated the experimental protocol. The sample exposed to light in an oxygen-deprived atmosphere presented a larger colour variation

than expected for the equivalent received LD in standard conditions, with ΔE* = 41.8. By projecting the abscissa onto the ΔE* reference curve, an 'equivalent light dose' (ELD) of 100 klux hours (i.e. saturation level) was obtained. The ELD corresponds to the LD that produces, in a given material and environment, the same effect as that measured on the same material exposed to light under controlled environmental conditions [28]. Figure 8 shows the graph of the typical evolution of (a,b)* of a LCU sample exposed under 5 klux fluorescent light, on which the data points of the two LCU samples are plotted. Both the coordinates +a* (red component) and –b* (blue component) for the LCU appeared highly affected by the absence of oxygen. This observation suggests that both the red and the blue dyes are sensitive to the oxygen concentration in the surrounding environment. It was concluded that the fading mechanism of both dyes is accelerated in the oxygen-free atmosphere. Although a more in-depth study would be needed to confirm and understand the underlying mechanism, the results suggest that for both dyes the leading fading process is most likely a photo-reduction mechanism. Figure 8 also shows that the blue dye has a higher sensitivity than the red dye (different rates of fading), as previously established by spectroscopic analysis.

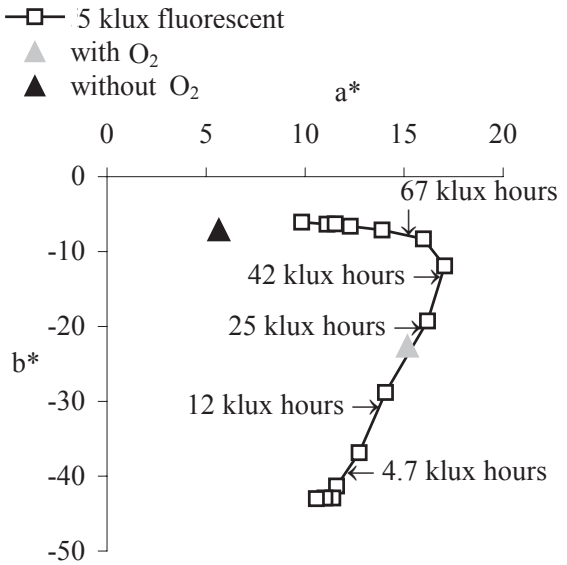


Figure 8 *Effect of oxygen and oxygen-deprived atmosphere on the colour alteration. (a,b)* graph obtained at I = 5 klux (fluorescent light source). Plotted on the curve are the LCU samples exposed to 20 klux hours (same conditions as for black line) in the presence of oxygen (▲) and without oxygen (▲).*

Atmospheric pollutants (NO_2 and SO_2)

Indoor air pollution is a matter of concern in the museum environment. It was thus decided to investigate the impact on LCU of selected atmospheric pollutants among the most commonly considered in museum environments (NO_2, SO_2). The timeframe of the project did not permit a full assessment of the entire range of polluting compounds occurring within indoor environments (including ozone and common indoor carbonyl compounds such as formaldehyde).

LCU sensor strips were prepared (2 × 15 cm) and exposed in the dark in a temperature- and humidity-controlled pollution chamber. The flow of NO_2 and/or SO_2 was laminar and controlled at both ends by chemiluminescence analysers AC 31 M for NO_X and AF 21 M for SO_2. The exposures of the samples to the two gases were carried out separately. The concentration of SO_2 was 53 mg·m^{-3} and the exposure lasted seven days at 23°C and 54% RH. The exposure to NO_2 was 10 mg·m^{-3} (the lowest concentration that could be accurately measured with the detection system), and lasted 10 days. It was carried out at 23°C and 50% RH. In Figure 9 the positions of the data points of the polluted samples are plotted on a reference graph of ΔE* versus LD (linear portion of the curve only) of a LCU sample exposed in the light box at I = 500 lux (tungsten-halogen light).

The more or less pronounced discolouration effect due to pollutants was evaluated on the basis of the reference curve. SO_2 had almost no effect, with ΔE* = 4.5, compared with the reference unpolluted sample. Unlike SO_2, NO_2 was found to be more effective in modifying the LCU colour. As an indication, with a colour change ΔE* = 32.5, a corresponding ELD = 24.3 klux hours was calculated. Thus, NO_2 seemed to have a considerable impact on the dye/matrix system. Nevertheless, it must be borne in mind that the levels used in the experiment were far higher than the levels typically found in an urban atmosphere, and probably even more so than an indoor atmosphere. In large cities, such as Paris for instance, the concentration of SO_2 in the air is typically around 0.02 mg·m^{-3}, and that of NO_2 about 0.05 mg·m^{-3}. Thus, in normal indoor conditions it can be assumed that the impact of atmospheric pollution gases on the LCU dosimeter would be negligible, especially as the LCU's extreme sensitivity to light would ensure that, in a typical timeframe of use, any pollutant's effect on

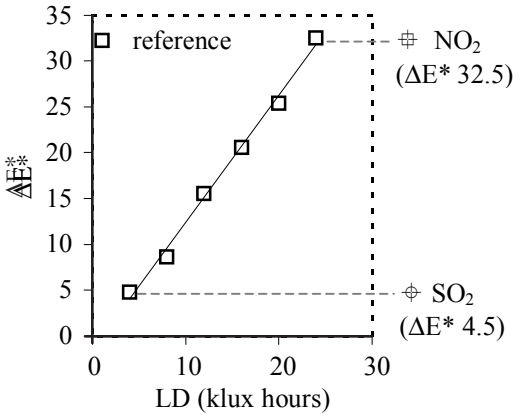


Figure 9 *ΔE* as a function of LD of LCU samples (I = 500 lux, light source: tungsten-halogen lamp). ΔE* of the samples exposed to SO_2 (⊕) and to NO_2 (⊕) are plotted on the graph. An ELD was calculated using the corresponding mathematical function.*

the colour of a LCU is likely to be overridden by light-induced effects.

Acetic acid vapour

In the museum environment, volatile organic compounds (VOCs) can be found in specific, yet not uncommon circumstances. Acetic acid is among the most ubiquitous of VOCs. It is present in concentrations that can reach up to 250 $mg \cdot m^{-3}$ in the air of freshly painted exhibition rooms, in wooden showcases and showcases containing silicone-based sealants [37–39]. In other situations, such as in archival boxes assembled with VOC-emitting adhesives, the concentration of acetic acid can range from 5 to 12.5 $mg \cdot m^{-3}$ over prolonged periods of time before levelling off [40]. Being fairly representative of typical indoor acidic VOCs, acetic acid was selected for investigation of its impact on the colour changes of the LCU sensor.

A three-component system (acetic acid/water/salt) was used to generate a humid acidic environment in enclosures at room temperature [41]. An equilibrium vapour acetic acid concentration of 49.9 $mg \cdot m^{-3}$ at 23°C and 54% RH was generated by mixing 100 mL of different concentrations of acetic acid solution with 500 g of magnesium nitrate hexahydrate in airtight 3 L Pyrex vessels, in the dark. Acetic acid concentrations in the head space of the vessels were measured, at the beginning and at the end of the exposures, with 81 and 81L specific detector tubes using the GV-100S pump set. The exposures lasted 10 days. Reference samples, exposed in the same climate conditions in vessels where no acetic acid had been added to the salt/water mixture, were used to validate the experimental protocol. For these reference samples, the values of ΔE* calculated compared to a normal sample at t = 0 were below 1.5.

The exposure to acetic acid led to a very slight effect on the colour of the sample (ΔE* = 2.2). In conclusion, in a first estimate analysis, it can be supposed that, although not null, the effect of acetic acid on the discolouration of the LCU is very small and probably negligible in the normal cases of application of LCU. It is generally advised that in practical use, in parallel with the LCU exposed in a museum room, a reference LCU should be placed in a dark, but well-ventilated, control package in the same environment as the exposed LCU, so that the preventive conservator may have higher confidence levels in the response of LCU with respect to anthropogenic pollutants.

Calibration and reference colour scale

It has been clearly established that the level of precision of the dosimeter was not intended to compete with electronic devices. Rather, the concept of preventive risk assessment was considered a high priority and the dosimeter was designed to provide a novel early warning tool capable of giving reliable, although only indicative, assessments of the LD received. In the final calibration of the dosimeter, the ease of visual interpretation was preferred over precise indication of the LD value. For these reasons, it was decided to deliver the LCU together with an easy-to-use calibration chart, that is a reference colour scale (RCS), rather than provide the whole colorimetric response curve. The calibration of the dosimeter, aimed at creating the RCS, was an important step in the characterization of the final LCU prototype. In developing the RCS many aspects were taken into account, considering that a commercial, user-friendly product had to be delivered.

LCU varies continuously from blue through purple to white during its fading process, each colour being related to a given value of the LD received. Since a subjective colour evaluation (observer dependent) is involved in the interpretation of the LCU response to the LD received, an unambiguous and simplified scale was preferred over a finer scale with multiple steps covering all the nuances spanned by LCU. Hence, the RCS was made with only a few colours, easily distinguishable to the naked eye.

Another important aspect considered when defining the RCS was the possible influence of factors other than light in the discolouration of LCU. From the numerous tests carried out on LCU, it was shown that although light was the leading factor, the colorimetric response could be slightly affected by some other environmental parameters. In other words, under particular exposure conditions, the colour of the LCU would have to be related to a synergistic action of light and other environmental agents. Thus, the choice of giving a broad range of possible LDs corresponding to each step in the RCS also responded to the need to take into account an intrinsic uncertainty in the LD value, due to a possible additive environmental impact.

Five easily distinguishable colour steps (named 0U to 4U) were isolated for the RCS. Light-aged samples were prepared under controlled lighting conditions, with different illuminance values within the range of practical interest (I = 500, 300 and 100 lux); the exposure time was varied to obtain an assortment of aged samples covering the range of LDs of 0–100 klux hours. Several sets of each were prepared in order to ensure statistically meaningful data for establishing the colorimetric calibration of the dosimeter. The five colours singled out for the RCS, in steps of $\Delta E^{\star} = 15$, were thus associated with a relatively broad range of LDs. The colour displayed by an exposed LCU has to be compared to the closest matching colour on the RCS – but not necessarily be coincident. Correspondingly, a broad range of LDs was attributed to each colour step of the RCS, so as to include the whole LD range which could produce all the possible colour nuances close to each colour stage. In this manner, the colour exhibited by LCU after exposure can be correlated to a quantitative indication of the LD accumulated during the exposure time. The final RCS and the tabulated values of LDs corresponding to each colour are given in Figure 10.

As the RCS was established at the final stage of the project and was based on LCU samples prepared by Particle Technology, using large-scale coating procedures, it was possible to provide a reliable colorimetric calibration based on samples produced in the industrial chain, rather than on laboratory prototypes. The tolerance in the LDs attributed to each colour stage allowed slight differences in the colour rendering of the final RCS to be taken into account. A difficulty which had to be overcome in the final stage of the project was the production of good quality printing for the RCS, to perfectly match the real colours exhibited by the LCU prototypes. An 'Instruction for use' sheet was prepared to be included in the commercial kit of the LCU and RCS,

(a)

LCU level	Light-Dose interval (lux hours)
0U	0 - 5000
1U	5000 - 30000
2U	30000 - 45000
3U	45000 - 75000
4U	75000 - 100000

(b)

Figure 10 *(a) Practical reading of an LCU dosimeter using the reference colour scale (photograph by Maja Kardum, reproduced with permission of the Trustees of the V&A Museum, London). (b) LCU reference colour scale table with correspondence between colour stages (0U to 4U) and LD, and colour chart corresponding to the defined fading stages (as reproduced in the commercial kit). (The reproduction of the colour here is not necessarily correct.)*

to provide practical guidelines and suggestions for the correct use and interpretation of the dosimeter.

Field exposures

In general, the accelerated light-ageing tests were aimed at simulating an ideal and simplified exposure situation, with stable conditions of temperature, RH and illuminances, in a range of typical values for museums (I = 50–500 lux). Nevertheless, in real situations unavoidable variability in display conditions and micro-environment are encountered, and it is difficult to cover all the possible circumstances with only laboratory tests. The comparison between the dosimeters' responses under controlled conditions and their actual behaviour in the field was therefore considered a necessary phase of the LCU testing. The field LCU exposures were carried out in parallel with the laboratory experiments in every phase of the research.

Several museums were selected for the field trials so as to cover a broad range of typical situations. The selected sites were the Uffizi Gallery, Florence; the Victoria and Albert Museum, London; the Musée Cognac-Jay, Paris; and the National Library and Lobkowicz Palace, Prague. In the last phase of the LiDo project, samples were also prepared for end-user testing. They were distributed to the conservation department staff of the Jewish Museum in Berlin, and to the organization staff of a travelling temporary photographs exhibition at venues in Rome and Paris,[6] which lasted about two months in each city. Only the results from the Rome exhibition are presented here.

In the natural exposure trials, different aspects in the behaviour of LCU were studied, such as the importance of fluctuations in lighting conditions (due to seasonal factors, day/light cycles, type of illumination, etc.), the impact of the overall micro-environment (synergy of light with temperature, RH and air pollutants) and the possible role of factors other than light in accelerating the fading. LCU samples were exposed simultaneously with other dosimeters (LCS and BWS no. 1) in the selected sites for a fixed period of time from one week to three months. The field data were projected onto the calibration curve built with laboratory experimental data points.

Four field trials were organized during the project. The most meaningful results are reported below. The first example concerns the first two field trials held in August 2001 and January 2002. These were mainly exploratory, and were aimed at investigating the qualitative colorimetric response of the dosimeters. Specifically, this earlier phase was focused on establishing the average lighting conditions/levels under which the dosimeters could be profitably used. Dosimeters were exposed in summer and in winter, in exactly the same locations of the selected rooms. The aim was to investigate the extent to which seasonal lighting changes could influence the rate of fading of the dosimeters. Both summer and winter exposures lasted one month. Figure 11 shows an overview of the colorimetric changes ΔE^* registered in all the samples exposed in the four sites (each set included a reference sample stored in dark). In winter, after four weeks, all the exposed LCUs exhibited moderate colour changes, but a completely different effect was observed in summer, although dosimeters were exposed in the same position and for the same period. Indeed, almost all the samples reached a colour change close to the saturation level ($\Delta E^* \approx 40$), regardless of the museum room considered. This result indicated that in museums rooms the average lighting levels were highly variable and could be strongly affected by seasonal fluctuations. In this experiment, only one sample exhibited a very small colour change, comparable

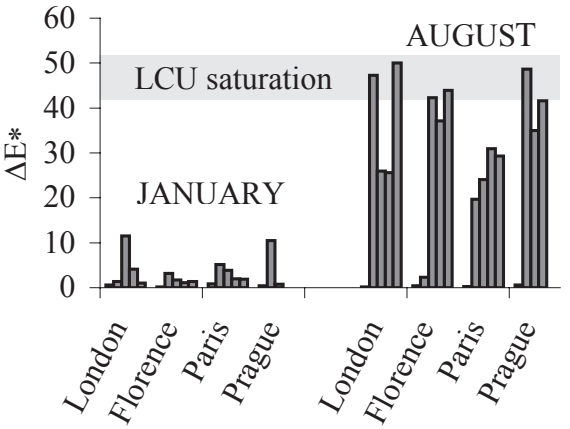


Figure 11 *Museum exposures of LCU (first and second LiDo field trials) in the selected sites. The seasonal (summer and winter) effects on lighting conditions in museums can be visualized by comparing the colour changes ΔE* occurring in winter and summer. In summer several LCU samples underwent complete bleaching after one month's exposure. (cf. Figure 3).*

[6] *'Rome 1850: The Circle of Artist-Photographers at the Caffè Greco', Capitoline Musei, Rome, 29 November 2003–25 January 2004; Maison Européenne de la Photographie, Paris, 11 February–18 April 2004.*

for both winter and summer exposures: the sample exposed in the Giotto Room of the Uffizi Gallery (LCU no. 1). This room houses a major masterpiece, *The Maestà* by Giotto, and it is therefore strictly controlled, with very low lighting levels – exclusively artificial light – and stable climate conditions.

These field trials also showed that in order to use the dosimeter effectively it is important to keep in mind its operational range and the average features of the luminous environment to be controlled. Indeed, when the colour change reaches values of ΔE^* that are too high (over saturation level) a correct assessment of the LD value cannot be made. The LD can only be reliably estimated if the LCU has not reached the stage of bleaching.

Based on this kind of experiment, a series of useful directions and guidelines for end-users were formulated: a) the LCU has to be periodically checked with a frequency adapted to the average lighting conditions; and b) the dosimeter should exhibit a promptly noticeable ΔE^*, upon a LD range of practical interest for the objects under control. This is known to be highly dependent on the kind of collection under survey [24, 38], and the more sensitive the collection, the lower the LD range over which irreversible changes can occur.

The third and fourth field trials were aimed at tackling more specific aspects. As a marketable product had to be delivered, it was of the utmost interest to ascertain the novelty of LCU with respect to BWS. As mentioned above, this aspect was primarily studied in the laboratory tests, which showed that the operational range of LCU was considerably lower than that of BWS no. 1, and that LCU and LCS together had a complementary operational range to BWS. The field trials corroborated this result. The fourth LiDo field trial was held in the summer of 2003. The exposure lasted three months, and intermediate checks of the samples were made monthly. The LCU samples were exposed together with LCS and BWS no. 1. The results from the exposure made at the Uffizi Gallery are reported in Figure 12 and Table 4. In this museum two sites were selected: the Giotto Room, described above, and the Geographic Maps Room (see Figure 1c), which has an environment that is completely different, yet quite typical of commonly encountered situations. Owing to the presence of large windows and almost completely natural lighting, this room presents highly variable environmental conditions. Moreover, the Geographic Maps Room is often closed to the public, while the Giotto Room is highly frequented.

In Figure 12 the colorimetric variations are plotted against the exposure time for the three types of dosimeter (LCU, LCS and BWS no. 1), simultaneously exposed, and the environmental data for the two rooms are reported in Table 4. The results showed that the LightCheck dosimeters (LCU and LCS) are much more reactive to light than BWS no. 1. Thus, the different sensitivities established in the laboratory tests were confirmed by the field tests. In particular, as projected, LCU was the more sensitive prototype, exhibiting an already appreciable colour variation ($\Delta E^* = 10$) after a short exposure period (one month) in the dimly lit Giotto Room. This fact indicated that LCU was particularly suitable to be used as a preventive tool for surveying light-sensitive objects, usually kept in closely

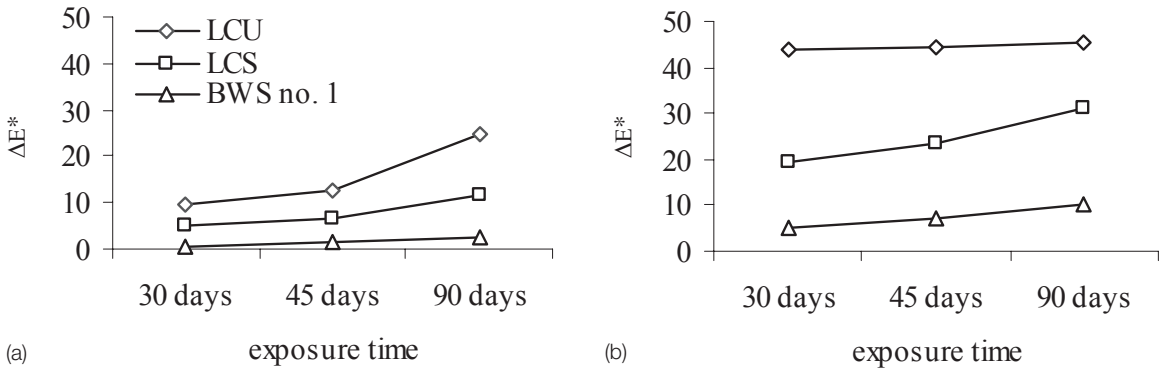


Figure 12 Responses of LCU, LCS and BWS no. 1 samples (ΔE^*) over time when exposed at the Uffizi Gallery during the fourth LiDo field trial: (a) in the Giotto Room; and (b) in the Geographic Maps Room.

Table 4 *Environmental data for the two exhibition rooms at the Uffizi Gallery, collected with data loggers during the entire exposure time (3 June–1 September 2003)*

	Illuminance (lux)	UV (μW·lm⁻¹)	T (°C)	RH (%)
Giotto Room				
mean	11	0	28	53
max	50	0	29	67
min	0 (dark)	0	26	35
Geographic Maps Room				
mean	100	6.4	30	53
max	830	42	32	90
min	0 (dark)	0	27	42

controlled environments and exhibited under very low illuminance. On the other hand, the same experiment showed that LCU was too sensitive to be used profitably in uncontrolled environments, such as the Geographic Maps Room, where natural light and high peaks in the illuminance levels led to a very rapid bleaching of the dosimeter. To survey this type of environment, in which only durable objects can be exhibited, the most useful dosimeter was found to be the less sensitive LCS [22]. In both types of exposure the BWS no. 1 showed a very small colour change, even at the end of the exposure time, performing poorly as a preventive light control tool in any of the situations tested.

The final topic related to field exposure that was studied was the impact of the entire micro-environment (i.e. the synergistic action of light with other environmental factors). The laboratory tests showed that the speed of the LCU fading could be slightly affected by factors other than light. Nevertheless, the field trials indicated that, in practical conditions, LCU was not significantly affected by the micro-environment, provided it was used properly (no UV light, compliance with the operational range).

All the field trials reported above were designed and carried out by the LiDo project team. In the last stages of the project, when the final prototype had been developed, LCU sensors were distributed to a group of end-users in order to gather a wider range of experience on the various uses of the sensor. This was carried out in the framework of the photography exhibition 'Rome 1850: The Circle of Artist-Photographers at the Caffè Greco'. Twenty LCU sensors were given to the Italian and French organization staff to be spread in the exhibition rooms in Rome, and placed adjacent to selected photographic items. The target illuminance for the rooms was 50 lux or lower, in compliance with recommendations for the lighting of very light-sensitive artefacts [24]. The exhibition lasted 51 days, and the opening time was six days per week, from 10.00 to 19.00 (nine hours per day). Assuming that the average light level was indeed 50 lux, a total maximum LD of about 23 klux hours was expected for the exposed samples.

Figure 13 shows the calibration curve (established with I = 500 lux, tungsten-halogen source) and the experimental data for each of the 20 LCU samples measured at the end of the exposure period. The chromatic coordinates were measured with the X-Rite SP 64 hand-held spectrophotometer. In order to obtain the LD received by each LCU sample, the data point was projected onto the calibration curve. As shown in Figure 13, two data points with ΔE^* of 39.6 and 44.1 appeared to have a LD above the maximal value expected, of 32 and 40 klux hours, respectively, as projected on the calibration curve. Thus, in the two corresponding locations the average illuminance could be supposed to be greater than 50 lux. All other LCU samples were exposed to expected light levels (≤ 50 lux). This experiment clearly showed that during an exhibition, even if specifically and carefully designed to expose very

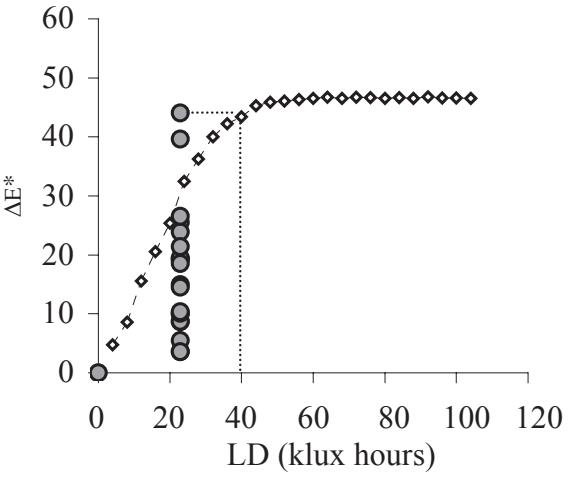


Figure 13 *Calibration curve ΔE* versus LD (I = 500 lux, light source: tungsten-halogen). The experimental data of each of the 20 LCU samples of the temporary exhibition in Rome are superimposed at the theoretical LD received of 23 klux hours (◯). The data points below the curve correspond to LCU samples that have effectively received LDs below 23 klux hours. The two data points above the curve correspond to two LCU samples that have received higher LDs than the calculated theoretical LD (LDs of 32 and 40 klux hours).*

fragile artefacts and with tightly controlled lighting, there can be a large variability in the lighting situations.

CONCLUSIONS

This work presents the exhaustive testing carried out for the development of the ultrasensitive light dosimeter, called LightCheck® Ultra (LCU), within the framework of the LiDo project. The LCU dosimeter can be used to provide an assessment of the risk related to light in museums and galleries, as it works as an early warning system for light damage to very light-sensitive artefacts.

The operational range of application of LCU was established as the LD range 5–100 klux hours, which is not covered by BWS no. 1. The LCU dosimeter functions on a colorimetric principle, and the assessment of the light dose received can be obtained promptly by a visual comparison with a calibration card (reference colour scale). By simple visual inspection of the hue, the end-user can determine reliably the range of light doses that has affected the dosimeter and, hence, the light dose the artefact on display has received. In this manner, the information provided by this novel device is significant, quick and easily extracted. Moreover, for more precise evaluations, the colour coordinates can be measured.

All the phases of the preparation and development of the final prototype were tested in anticipation of its final commercialization and large-scale production. All the characteristics of LCU were evaluated and fine-tuned by means of a structured programme of controlled light-ageing tests in the laboratory, corroborated by field trials.

The indicator has been shown to be a robust device which responded primarily to light (i.e. to the radiation in the visible spectral range) and could be stored for several months in the dark without undergoing any alteration of its properties. It was also found that the colour of the sensor could be slightly affected by environmental factors, but only secondarily. Indeed, only by adopting very severe environmental conditions (high values of temperature, RH or pollutant concentrations) could a measurable effect in the dosimeter response be observed. Conversely, in standard exposure conditions, the dosimeter response was completely correlated to the LD received and fully complied with the reciprocity principle.

In-depth laboratory tests fully characterized the dosimeter's response and provided directions for its correct use in practical situations. For instance, LCU promptly responded to very low and moderate light levels (I = 50–500 lux), but its reliability fails upon prolonged exposures in strongly illuminated environments. Moreover, LCU is not suitable for monitoring objects stored in anoxic or partially oxygen-depleted conditions. Unlike most other environmental parameters tested, oxygen was found to have a non-negligible impact on the LCU light-induced response, since its presence or absence in the surroundings of the sensor directly affected the fading mechanism of the dyes. Therefore, LCU reacts to global environmental conditions and thus can be considered as an integrative system. However, it can be safely concluded that, if correctly used, LCU principally records the LD received and the effect of light overrides all other ambient factors. This was further proven by the field tests carried out.

As stated in the introduction, a parallel study within the LiDo project resulted in the characterization and realization of a second light sensor prototype, LCS, which responded to visible light in the LD range 90–400 klux hours. Although it was not within the scope of the present article to cover the development of LCS, it is important to highlight that LCS and LCU have been designed as complementary sensors with their application ranges aimed at medium-to-low and low-to-very-low exposures levels, respectively, where BWS no. 1 lacks sensitivity [22]. The two sensors developed are currently commercialized under the name LightCheck®.

LightCheck® is expected to have a significant impact in the community of conservation professionals, in several applications. For instance, the concept of making art and cultural heritage available to the wider public has expanded extremely quickly in recent years and many museums operate very active loan policies. LightCheck® sensors are simple and low-cost tools which allow monitoring of the exhibition history, in terms of lighting, of a particular artefact. LightCheck® sensors are small and discrete and do not disturb the exhibition theme or the aesthetics of a display. Beside its relevance as a novel light-monitoring tool, LCU also has a great potential as a learning tool, to increase awareness of the fragility of many artefacts with respect to light. Indeed, as reported by end-users, the discolouration of the dosimeter is a very tangible and instructive representation of how light acts and reacts on artistic materials.

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SUPPLIERS

Toluidine Blue O; PVAL 99% and 98–99% hydrolysed: Aldrich, www.sigmaaldrich.com

Eosin 225: Réactifs RAL, www.reactifs-ral.fr

Rhodoviol 25/140 PVAL: Merck Eurolab, www.merckeurolab.com

Savatex PVAC emulsions: Raoul Labord, www.labord.com

Bristol paper: Clairefontaine, Rhodia, www.clairefontaine.com

Multigrade IV FB fiber photographic paper: Ilford, www.ilford.com/fr

Light-ageing chamber and SIRPAC 2000 v.2.26 software: Servathin, www.servathin.com

Soda lime float glass filter: Securit, Saint Gobain, www.saint-gobain.com

Daylight TFP Prestiflux 18 W JR/865 lamps: Mazda, www.mazdaeclairage.com

Halostar longlife tungsten-halogen lamps: Osram, www.osram.fr

Neutral density filters: Oriel Instruments, www.lot-oriel.com

Data loggers IrLog: Elsec, Littlemore Scientific Engineering, www.elsec.co.uk

Spectrophotometer Lambda 19: Perkin-Elmer, www.perkinelmer.com

Spectralon® standards: Labsphere, www.labsphere.com

Hand-held SP 64 spectrophotometer: X-Rite, www.xrite.com

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Résumé — *Cet article décrit la mise au point et l'optimisation d'un dosimètre de lumière ultrasensible, spécialement conçu pour l'évaluation préventive des risques de dégradation des objets hautement photosensibles. Cet indicateur, appelé LightCheck® Ultra, est composé d'une couche de polymère et de colorant photosensible appliqué sur un support papier. L'indicateur est caractérisé par une variation progressive de la couleur lorsque l'exposition à la lumière augmente. Initialement bleue, la couleur passe successivement au pourpre et au rose, puis au blanc, sous l'effet de la lumière visible. La variation de couleur a été étudiée sous diverses conditions environnementales. Les altérations dues à la lumière faisant l'objet de l'étude ont été évaluées au moyen de diverses sources de lumière et de divers niveaux d'éclairement. L'impact d'autres facteurs non liés à la lumière (comme la température, l'humidité, la pollution de l'air à l'intérieur – pollution atmosphérique et composés organiques volatils – et l'oxygène) a également été étudié. Le comportement de l'indicateur sur le terrain, dans des musées ou maisons historiques sélectionnés, a été évalué en parallèle avec des mesures en laboratoire à chaque étape de la mise au point. Finalement, l'étalonnage a été mené à bien et une échelle de couleur de référence a été établie, permettant une lecture rapide et aisée de l'indicateur. Au*

moyen de cette échelle de référence de couleurs, la couleur affichée par le dosimètre après une exposition peut être aisément corrélée à l'évaluation quantitative de la dose de lumière reçue. Les phases de mise au point et d'essai de ce nouveau dosimètre de lumière sont présentées en détail, depuis la conception initiale jusqu'au produit final.

Zusammenfassung *— In dieser Arbeit wird die Entwicklung und Optimierung eines ultrasensitiven Lichtdosimeters beschrieben, welches insbesondere für Abschätzung der Empfindlichkeit photosensitiver Materialien im Rahmen der Präventiven Konservierung konzipiert ist. Der Indikator LightCheck® Ultra besteht aus einer photosensitiven Farbstoff/Polymer – Schicht auf einem Papierträger. Er ist durch eine fortschreitende Farbveränderung bei steigender Lichtexposition gekennzeichnet. Anfänglich blau, verändert sich die Farbe unter sichtbarem Licht über purpur nach rosa und schließlich weiß. Die lichtinduzierten Veränderungen wurden unter Verwendung unterschiedlicher Lichtquellen und Beleuchtungsstärken evaluiert. Der Einfluß anderer Faktoren (Wärme, Feuchtigkeit, Luftverschmutzung, flüchtige organische Materialien sowie Sauerstoff) wurde ebenfalls untersucht. Das Verhalten des Indikators wurde parallel zu den Laborexperimenten auch in ausgewählten Museen und historischen Häusern getestet. Zum Schluß wurden eine Kalibrierung und eine Farbreferenzscala für eine schnelle und einfache Auswertung entwickelt. Mit Hilfe dieser Farbreferenzscala kann die Lichtdosis nach der Lichtexposition einfach abgelesen werden. Die Entwicklungsphasen dieses neuen Lichtdosimeters werden detailliert vom Frühstadium bis zum fertigen Produkt gezeigt.*

Resumen *— Este artículo presenta el desarrollo y optimización de un dosímetro ultrasensible de luz, especialmente diseñado para valorar, de manera preventiva, los riesgos de daño en objetos altamente fotosensibles. Este indicador, llamado LightCheck® Ultra, se compone de una capa de colorante fotosensible/polímero aplicado sobre un soporte de papel. El indicador se caracteriza por una variación de color progresiva según va aumentando la exposición a la luz. Inicialmente azul, el color va cambiando sucesivamente hacia el púrpura, y del rosa al blanco, bajo el efecto de la luz visible. Se investigó la variación del color bajo diferentes condiciones ambientales. Se evaluaron las alteraciones inducidas por la luz utilizando diferentes fuentes y niveles de iluminación. El impacto de otros factores no relacionados con la luz (como temperatura, humedad, polución en interiores, compuestos orgánicos atmosféricos volátiles – y oxígeno) también fue investigado. El comportamiento del indicador en su localización proyectada (para edificios históricos o museos) se valoró en relación con los experimentos llevados a cabo en el laboratorio en las diferentes etapas del proceso. Finalmente, se llevó a cabo la calibración, y se suministró una carta o escala de color con el fin de conseguir una lectura rápida y fácil. Por medio de esta escala de color de referencia el color exhibido por el dosímetro después de la exposición puede ser fácilmente correlacionado con una indicación cuantitativa de la dosis de luz recibida. Se presentan en detalle las fases de desarrollo y prueba de este novedoso dosímetro de luz, desde el diseño inicial hasta el producto final.*