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## Colorimetric sensor arrays: development and application to art conservation

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### ABSTRACT

Acceptable air pollutant concentration limits for sensitive artwork are generally at or below a few ppb: this is only ~1% of the permissible exposure limits for humans. Monitoring pollutants at such low levels is an exceptional challenge, especially to do so in a cost-effective fashion for a large number of locations and microenvironments (e.g., every display case in a museum). To meet this challenge, we have extended our portable “optoelectronic nose,” by using new sensor array chemistry to develop *cumulative* colorimetric sensor arrays with dosimetric sensitivities that are dramatically better than commercial sensor tubes. The color changes of each sensor in a disposable printed array produce a composite response to volatiles. Using cell phone camera imaging, we have made field trials to monitor pollutant exposure of artwork from the Walt Disney Animation Research Library during shipping to and exhibition in Beijing. This exhibition, “Drawn from Life: the Art of Disney Animation Studios,” featured animation drawings, story sketches, layouts, and concept art spanning the 90 years of the Disney Animation Studio’s history. Sensor arrays monitored exterior and interior environments of *pas-se-partout* artwork frames during exhibition and inside shipping crates during transport providing quantitative information on oxidant, aldehyde, and sulfide pollutant exposure.

### RÉSUMÉ

La concentration limite de polluant acceptable pour une œuvre d’art sensible est généralement plus petite ou égale à quelques ppb : ceci étant environ 1% de la valeur limite d’exposition (NIOSH PEL) fixée pour les humains. Surveiller les polluants à un niveau si faible est un défi scientifique exceptionnel, particulièrement lorsqu’il s’agit de le faire de façon économique pour un nombre élevé de lieux et de microenvironnements (ex. chacune des vitrines d’un musée). Afin de relever ce défi, nous avons amélioré notre « nez optoélectronique » déjà extrêmement sensible et portable : en utilisant une nouvelle gamme de capteurs chimiques, nous avons développé une matrice de capteurs colorimétriques dont la réaction est cumulative, possédant des sensibilités dosimétriques infiniment meilleures que celles des tubes capteurs commerciaux. En suivant numériquement le changement de couleur de chaque zone réactive sur une matrice imprimée et jetable, une mesure quantitative de la réponse du réactif au contaminant volatil est obtenue. Nous avons développé une plateforme utilisant le traitement d’image de l’appareil photo d’un téléphone cellulaire et avons fait des expériences pratiques de mise à l’essai en suivant l’exposition aux polluants des œuvres d’art provenant du Walt Disney Animation Research Library pendant leur transport et leur présentation à Pékin. Cette exposition, « Drawn from Life: The Art of Disney Animation Studios » mettait en valeur des dessins d’animation, des esquisses de scénario, des schémas, des arrière-plans et des concepts artistiques couvrant les 90 ans de l’histoire du Walt Disney Animation Studio. Les matrices de capteurs ont été utilisées pour suivre à la fois les environnements extérieurs et intérieurs des *pas-se-partout* des cadres lors de l’exposition, et l’intérieur des caisses de transport pendant leur déplacement, livrant de l’information quantitative importante sur l’exposition aux oxydants, aux aldéhydes et aux sulfures. Traduit par : Isabelle Cloutier.

### RESUMO

Os limites aceitáveis de concentração de poluentes atmosféricos para obras de arte sensíveis são geralmente iguais ou inferiores a algumas ppb (partes por bilhão): isto é apenas 1% dos limites de exposição permitidos (NIOSH PEL) exigidos para seres humanos. Monitorar poluentes em níveis tão baixos é um desafio científico excepcional, especialmente quando se quer fazer isso com uma boa relação de custo-eficácia para um grande número de locais e microambientes (por exemplo, todos os expositores de um museu). Para enfrentar esse desafio, ampliamos o nosso já extremamente sensível e portátil “nariz optoeletrônico”: utilizando um novo sistema de sensor químico, desenvolvemos matrizes de sensores colorimétricos cumulativos com sensibilidades dosimétricas que são dramaticamente melhores do que os tubos de sensores comerciais.

### KEYWORDS

Colorimetric sensor array; preventive conservation; air pollution; pollutants; sensors

Monitorando digitalmente as mudanças de cor de cada ponto do sensor em uma matriz descartável impressa, é obtida uma medida quantitativa da resposta composta aos voláteis. Desenvolvemos uma plataforma que se baseia em imagens de câmeras de telefones celulares e fizemos testes de experimentos de campo para monitorar a exposição poluente de obras de arte da *Walt Disney Animation Research Library* (Biblioteca de Pesquisa de Animação da Walt Disney) durante o envio e a exposição em Pequim. Esta exposição, "Extraída da Vida: A Arte dos Estúdios de Animação da Disney", contava com desenhos de animação, esboços de histórias, layouts, planos de fundo e arte conceitual que abrangem os 90 anos da história do *Walt Disney Animation Studio* (Estúdio de Animação de Walt Disney). Conjuntos de sensores foram usados para monitorar tanto ambientes externos quanto internos de molduras na exposição e dentro das caixas de remessa durante o transporte, oferecendo valiosas informações quantitativas sobre exposição a poluentes oxidantes, aldeídos e sulfuretos. Traduzido por Cristina Antunes, e revisado por Beatriz Haspo.

#### RESUMEN

Los límites aceptables de concentración de contaminantes atmosféricos para obras de arte sensibles son generalmente de unos pocos ppb o menos: esto es solo ~ 1% de los límites de exposición permisibles (NIOSH PEL) requeridos para humanos. Monitorear los contaminantes a niveles tan bajos es un desafío científico excepcional, especialmente para hacerlo de una manera rentable para una gran cantidad de ubicaciones y microambientes (por ejemplo, cada vitrina en un museo). Para hacer frente a este desafío, hemos ampliado nuestra "nariz optoelectrónica" ya extremadamente sensible y portátil: mediante el uso de nueva química de matriz de sensores, hemos desarrollado matrices de sensores colorimétricos acumulativos con sensibilidades dosimétricas que son dramáticamente mejores que los tubos sensores comerciales. Controlando digitalmente los cambios de color de cada mancha de sensor en una matriz desechable impresa, se obtiene una medida cuantitativa de la respuesta compuesta a volátiles. Hemos desarrollado una plataforma que se basa en imágenes de cámaras de teléfonos celulares e hicimos experimentos de campo de prueba para monitorear la exposición de las obras de arte de la Biblioteca de Investigación de Animación de Walt Disney durante el envío y duración de una exhibición en Beijing. Esta exhibición, "Dibujado desde la vida: el arte de los estudios de animación de Disney" presentó dibujos de animación, bocetos de historias, diseños, fondos y arte conceptual que abarca los 90 años de la historia del estudio de animación de Walt Disney. Las matrices de sensores se usaron para monitorear tanto el ambiente exterior como el interior de los marcos paspartú en la exhibición y dentro de las cajas de envío durante el transporte, ofreciendo valiosa información cuantitativa sobre la exposición a oxidantes, aldehídos y sulfuros contaminantes. Traducido por Amparo Rueda.

## 1. Introduction

One of society's most important cultural responsibilities is the preservation of the past for the future (Whitmore 2005). The surest way to protect cultural heritage objects from damage is to control the environment in which they are displayed; both physical and chemical factors, such as light, temperature, relative humidity (RH), and so on, can have a profound impact on these objects' lifetimes (Brimblecombe 1994; Camuffo et al. 2001). The discussion of slowing the deterioration of museum materials through environmental control was pioneered by Thomson (Thomson 1965; Thomson 1986) on the issue of artwork exposure to pollutants and by Oddy on exposure to local environmental conditions, including the microenvironment of the display case itself (Oddy 1973). In conservation, the focus over time began to shift from restoration to prevention, and the field of preventive conservation as it is known today was born (Whitmore 2005).

The intention of preventive conservation is to extend an object's lifetime by controlling environmental conditions (microclimate, microbiology, and chemical pollution)

around a work of art. Over the last 50 years, the importance of measuring air pollution exposure has become clear for preservation of cultural heritage materials. Work in preventive conservation has revealed the deteriorative effects of even very low levels of specific pollutants (Feng 2016; Grzywacz 2006; Hatchfield 2002; Lucchi 2018; Smielowska, Marc, and Zabiegala 2017). Improvements in detection methods, museum materials, and HVAC environmental control have begun to improve the environment to which artwork is exposed (Blades et al. 2000; Camuffo et al. 2001; Ferdyn-Grygierek 2014; Hisham and Grosjean 1991; Marć et al. 2015; Schieweck and Bock 2015). In order to continue such progress, it is necessary to develop cost-effective techniques for continuous control and monitoring of pollutants for a large number of locations and microenvironments (e.g. every display case in a museum) at the low levels needed to minimize the effects of common pollutants (Table 1).

For this reason, a collaborative effort between the University of Illinois, the Getty Conservation Institute (GCI), and the Walt Disney Animation Research Library

**Table 1.** Important pollutants found inside museums and suggested concentration limits for cultural heritage materials (Grzywacz 2006).

Major pollutants	Suggested limits for sensitive materials (ppb)	Suggested limits for other collection materials (ppb)
NO <sub>2</sub>	0.05–2.6	2–10
O <sub>3</sub>	<0.5	0.5–5
SO <sub>2</sub>	0.04–0.4	0.4–2
H <sub>2</sub> S	<0.1	<0.1
Acetic acid	<5	40–280
Formic acid	<5	5–20
Formaldehyde	<0.1–5	10–20
Acetaldehyde	<1–20	

(ARL) has explored and developed, as a proof of concept, a colorimetric sensor array for the monitoring of air pollutants in situ during shipping and exhibition of artwork. This work builds on the “optoelectronic nose” that has been developed by the Suslick group at the University of Illinois (Askim and Suslick 2015; Askim, Mahmoudi, and Suslick 2013).

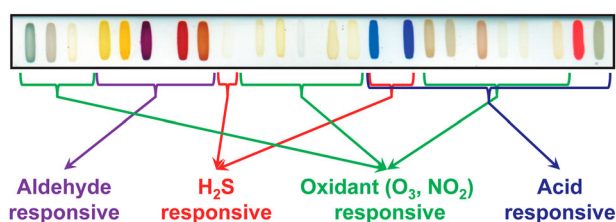
Colorimetric sensor arrays have proven to be a broadly applicable technology that can successfully differentiate among volatile organic compounds (Janzen et al. 2006; Lin, Jang, and Suslick 2011), toxic industrial chemicals (Feng et al. 2010a, 2010b; Lim et al. 2009), explosives (Askim et al. 2016), foods and beverages (Li and Suslick 2016; Musto, Lim, and Suslick 2009; Suslick, Feng, and Suslick 2010; Zhang and Suslick 2007; Zhang, Bailey, and Suslick 2006), and bacteria and fungi (Carey et al. 2011; Zhang et al. 2014). The core of past colorimetric sensor array technology is an array of chemically responsive dyes. These dyes serve as sensors by changing colors due to dye–analyte interactions, e.g. Brønsted and Lewis acid–base, hydrogen bonding, dipolar, and  $\pi$ – $\pi$  interactions (Askim et al. 2013). These sensors are essentially “chemical fuses.” They are generally reversible for most analytes (especially at lower concentrations), but do need to be replaced after exposure to very high concentrations or to an aggressive analyte. Thus, the interactions between gaseous analytes and colorants in our earlier arrays mostly represent equilibrium interactions, and for this reason, previous colorimetric sensors must be imaged during exposure in real-time. Many of the dye–analyte interactions are relatively strong, and so sensitivities are often in the ppb regime, especially for chemically reactive analytes (Askim et al. 2013; Askim et al. 2016; Feng et al. 2010b). Importantly, the colorimetric sensor arrays are relatively unaffected by changes in RH or changes in temperature (Askim, Mahmoudi, and Suslick 2013; Janzen et al. 2006; Rakow and Suslick 2000).

For monitoring of artwork, there are two significant disadvantages for any equilibrium-based sensor: (1)

there is no improvement in sensitivity with increased dosage (i.e. exposure time) and (2) as mentioned, arrays must be imaged in real-time. The first disadvantage is critical where long-term, but very low concentration, exposures need to be monitored, i.e. the long-term sensitivity of artwork to pollutants (Table 1). The second disadvantage, real-time imaging, complicates real-world use of the sensor array during exhibition conditions.

To overcome these disadvantages, we have altered the dye–analyte chemistry to make the reactions *irreversible* and thus the analyte–dye reactions become *cumulative* (dosimetric) sensors where the color change is monotonic (and generally linear) as a function of dose until saturation (i.e. when all dye has reacted). Pollutant concentration measurements are therefore representative of a time-weighted average (TWA) in units of concentration multiplied by exposure time (e.g. ppb-days). Essentially, our approach draws on colorimetric irreversible reactions already in use with current direct-reading passive sampling devices (Grzywacz and Stulik 1992), such as Draeger tubes (Draeger Co. 2016), but miniaturizes the reactive media dramatically, includes dozens of analyte-specific sensors (i.e. each spot), and substantially improves sensitivity and accuracy by use of digital imaging. Given the relatively high cost of individual sampling tubes, using a printed disposable array also has the promise of a very large decrease in operating expenses.

Each sensor includes a dye-indicator whose reactions with the target analyte are essentially irreversible, i.e. have enthalpies greater than  $\sim$ –150 kJ/mol (e.g. metal sulfide precipitations, strong coordination to metal ions, irreversible oxidations of dyes or bleaching, redox reactions with large  $\Delta E^\circ$ ). In addition, to de-convolute the response to the total environment into responses from individual analytes or classes of analytes, each chromophore is chosen to react more specifically with a class of analytes (i.e. oxidants, sulfides, aldehydes, acids), which is a departure from the cross-reactive equilibrium sensor spots of our earlier arrays. These sensor spots were compiled into a first-generation array for detection of multiple museum pollutants; the array incorporates into one cohesive system a series of chemically responsive dyes that respond specifically to the main museum pollutants (Figure 1 and Supplemental Material (Table SM1)). Digital images are taken of the sensor array before and during exposure to pollutants, and color difference maps (Figure 2) are generated by digital subtraction, pixel by pixel, of the center portion of sensor spots in the array before and after exposure. The resulting changes in red, green, and blue values are used for quantitative analysis of the data, as described below in Section 2.3.



**Figure 1.** Photographic image of a colorimetric sensor array for artwork monitoring, 5×30 mm. The array incorporates, into one disposable strip, 24 cumulative chemically responsive dyes that are dosimetric for specific classes of analytes relevant to the museum environment. These include redox indicators for oxidants (ozone, NO<sub>2</sub>), precipitatory metal ions (for sulfides), pH indicators (SO<sub>2</sub>, acetic, formic acids), and Brady and Schiff reactants (aldehydes).

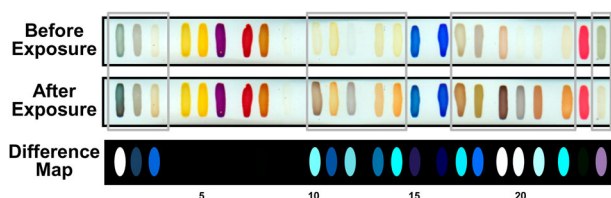
## 2. Materials and methods

### 2.1. Materials, formulations and array printing

Certified, premixed gas tanks were obtained from Matheson Tri-Gas Corp. through S. J. Smith, Co. (Urbana, IL). Gas streams were prepared by mixing the analyte stream with dry and wet nitrogen gas. MKS mass flow controllers were used to achieve the desired concentrations and RH. Importantly, gas stream concentrations and RH were confirmed by real-time, in-line analysis using an MKS multigas analyzer (model 2030, a dedicated Fourier transform infrared spectrophotometer).

All other reagents were an analytical-reagent grade, purchased from Sigma-Aldrich and used without further purification. Each sensor spot was optimized for best chemical compatibility and sensor performance (formulations given in Supplementary Material (SM), Table SM1).

Sensor arrays were robotically printed in a linear geometry using custom-designed rectangular pins (Figure.

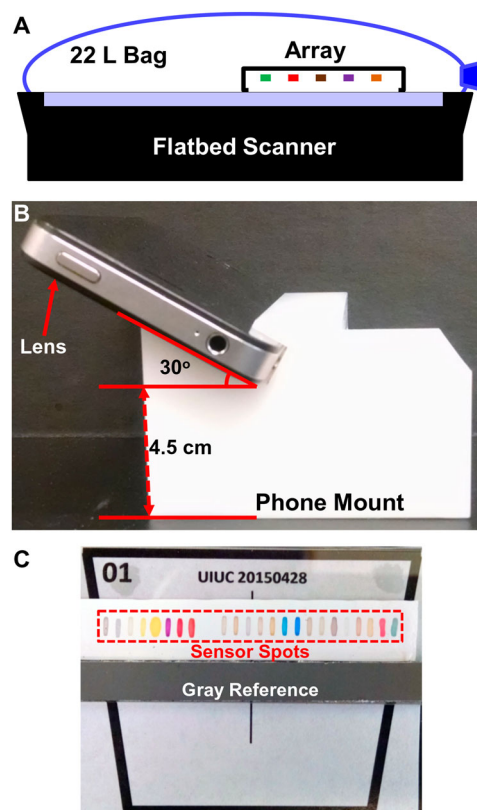


**Figure 2.** Images of a 24-dye colorimetric sensor array, (Top) before exposure and (Middle) after exposure to 1.5 ppm ozone for 2.0 min; images obtained with an ordinary flatbed scanner. (Bottom) Subtraction of the two images yields a difference vector in 3N dimensions (i.e. N changes in red, green, and blue color values, where N is the number of sensor spots); the difference vector is represented by a difference map that shows the absolute values of the color changes of each spot. The gray boxes indicate the sensor spots that are most responsive to ozone. For purposes of visualization and display, the color range of difference maps is usually expanded; in this case, the RGB color values were expanded from 6 bit to 8 bit color; all quantitative analysis, however, uses only the raw measured differences unaltered.

SM1) installed in an Array-It NanoPrinter (Askim et al. 2016). Printing of arrays was usually on a white polypropylene membrane (Sterlitech Corporation; thickness: 130–170 μm, pore size: 0.22 μm) and attached to an imaging platform (described in detail in Section 2.2) using a solvent welding method (dichloromethane). Further experimental details are available elsewhere (LaGasse 2016).

### 2.2. Passive sampling techniques

Array response has been explored for passive sampling environments, in which air and pollutants diffuse to the surface of the sensor spots without active circulation or pumping. There are two different modes of exposure and imaging used in this study (shown schematically in Figure 3): (1) passive sampling and real-time imaging using a flatbed scanner and (2) passive sampling and



**Figure 3.** (A) Passive sampling setup using a flatbed scanner for array imaging. Injection of a calibrated concentration and volume of the analyte was used to prepare a fixed gas concentration passive sampling environment. (B) Cellphone imaging platform and holder for an iPhone 4S. (C) Sensor array for imaging by cellphone camera. To generate reproducible images of the sensor array, the cellphone in the custom holder was positioned so that the trapezoid filled the camera's rectangular field of view; the trapezoid was sized so that the perspective through the cellphone camera appeared as a rectangle.



periodic imaging using an iPhone 4S camera. The former was used primarily for calibration purposes and the latter primarily for field-testing during shipping and exhibition. In general, we observe significantly better S/N in the difference maps generated with the flatbed scanner compared to the iPhone camera, due primarily to higher noise in the iPhone images.

### 2.2.1 Passive sampling with flatbed scanner

For passive sampling with flatbed scanner imaging, sensor arrays were imaged face down on the flatbed scanner (Epson V600) through a clear zip-top polypropylene bag (6 Gal., filled using mass flow controllers to a total volume of 22 L) with a rubber septum and 3-way valve attachment (Figure 3A). The array was placed in the bag facing downward to the scanner surface, the bag was closed and excess air removed. The bag was then filled with a fixed volume (7.8 L) of 50% RH filtered air. After 3 min, a “before-exposure” image of the array was taken. Analyte gas was then drawn out of another bag using a syringe through a rubber septum attachment and injected into the experimental bag containing the array to create an environment with the desired concentration of analyte. After analyte addition, images of the arrays were taken at set time increments during exposure.

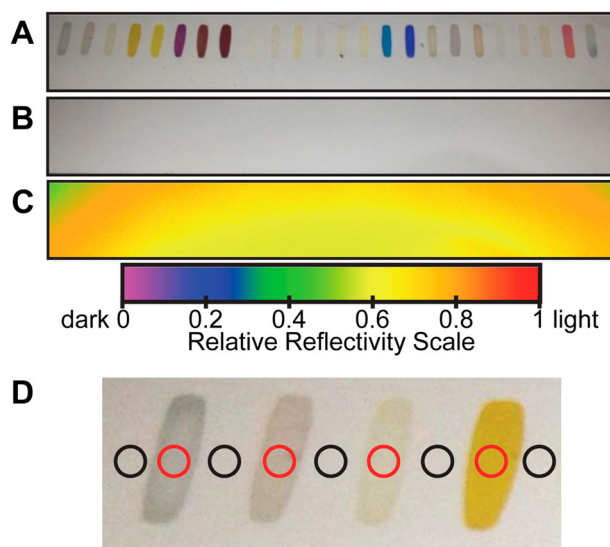
### 2.2.2. Passive sampling with cellphone imaging

For passive sampling with cellphone imaging, images were acquired using an iPhone 4S held in a custom mount machined to hold the iPhone at a fixed 30° angle and at a fixed height above the array surface (Figure 3B). This mount, when used in conjunction with the trapezoid outline printed on the array-imaging platform (Figure 3C) allowed images of each array to be taken from the same position. To obtain an image, the phone-mount setup was moved towards the imaging platform until the sides of the trapezoid perimeter aligned with the edges of the camera display. Due to perspective, the trapezoid perimeter when viewed through the camera becomes the rectangular outer edge of the camera’s field of view. Triplicate images were taken each time and later averaged to minimize noise.

The imaging platform consists of sensor spots printed on a white polypropylene membrane with a neutral gray reference strip mounted 1 cm below sensor spots. The polypropylene and gray reference strip was then attached to an impact resistant polycarbonate film the size of a glass microscope slide (McMaster-Carr; 1”x3”x0.040”) and mounted on acid-free paper (HP Premium Choice). Printed on the acid-free paper was (1) an identifying number unique to each array, (2) the date the array was printed, and (3) a trapezoid outline that was sized to aid in image alignment (as discussed above).

To make field use as easy as possible for the curator, no special effort was taken to isolate the system from ambient lighting. To reduce the effect of changes in ambient lighting, each image was taken with the camera flash illumination turned on. Illumination of the array, as expected, was not uniform even with the use of the camera flash. There are inevitable differences in the illumination across the field of view, in both horizontal and vertical directions. As shown in Figure 4, these differences are significant, and lighting differences across the sensor array surface or from image to image can deviate as much as  $\pm 20\%$  from the center of the array outwards.

For this reason, the RGB values obtained for each spot have been normalized using the white surface of the polypropylene membrane on which the sensor spots were printed as a reference. The areas on both sides of each sensor spot were used to obtain an average white reference RGB value for each spot; this reference value was used to normalize the lighting illuminating each spot (Figure 4C). Each spot across the array for each image at each time point has a measured R, G and B value (i.e.  $X_{meas}$  where  $X = R, G, B$ ) and a corresponding value for a white standard (i.e.  $X_{white}$ ) averaged from both sides of the spot for the white reference (Figure 4C). A corrected RGB value,  $X_{corr}$  can be defined as  $(X_{meas}/X_{white}) * 255$ . This simple correction produces roughly a twofold



**Figure 4.** Sensor array imaging with cell phone camera. (A) Digital image of the array sensor spots taken with an iPhone 4S camera. (B) Digital image of an unprinted polypropylene membrane (i.e. no sensor spots) to show the non-uniformity of lighting. (C) False-color gradient map of relative reflectivity of the unprinted membrane showing non-uniformity of lighting; deviation is  $\pm 20\%$  over the array; false color reference scale also shown. (D) Enlarged view of several sensor spots showing the areas averaged for each of the dye spots (red outline,  $X_{meas}$ ) and for the illumination normalization (black outline) on both sides of each dye spot, averaged to give  $X_{white}$ .

improvement in the reproducibility of RGB values obtained for the sensor spots over a wide range of external illumination (e.g. fluorescent vs. halogen lamp at 5000 K vs. halogen at 3000 K). Attempts to make a more sophisticated correction to the RGB values using an adjacent gray standard were unsuccessful due to the non-uniformity of illumination in the vertical direction.

### 2.3. Data processing and analysis techniques

From digital images of the sensor array before and after exposure to pollutants, a color difference map (Figure 2) is easily generated by digital subtraction, pixel by pixel, of the center portion of each of the sensor spots in the array: red value after exposure minus red value before, green minus green, blue minus blue. The resulting data is inherently digital (simply a vector of  $3N$  dimensions where  $N$  = total number of spots), and all quantitative and statistical analysis is done directly from the digital difference vectors, i.e. the  $\Delta RGB$  values. The color difference maps are useful primarily for convenient visualization of color changes of the dyes in the array; note that the color values shown in the difference maps are the absolute values of the differences and that expansion of the color space is useful for visualization of small color changes.

The changes in red, green, and blue values ( $\Delta RGB$ ) from each sensor element at a given time point can be combined into a Euclidean distance at any given time, ( $ED_t$ ), defined by Equation (1):

$$ED_t = (\Delta R_1^2 + \Delta G_1^2 + \Delta B_1^2 + \Delta R_2^2 + \dots + \Delta B_n^2)^{1/2} \quad (1)$$

where  $n$  is the number of spots under consideration and  $t$  is the time of exposure. To generate a response profile for a given analyte, the average Euclidean distance for multiple trials ( $\overline{ED}_t$ ) is plotted with respect to time. From this response profile, reaction kinetics can be determined. A dosimetric sensor gives a nearly linear response until saturation, and the slope of this response profile as a function of analyte concentration determines the dosimetric sensitivity (DS) and limit of detection (LOD) (discussed in greater detail in Section 3.2).

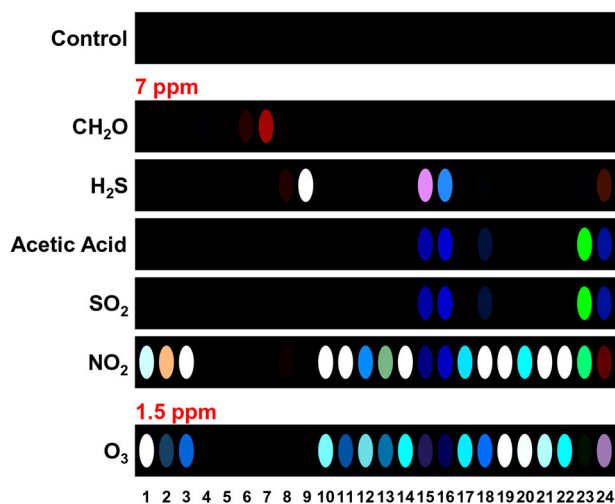
## 3. Results and discussion

### 3.1. Pollutant discrimination by the colorimetric sensor array

The sensor array was evaluated for its ability to discriminate among pollutant classes (i.e. oxidants, sulfides, aldehydes, and acids). As shown qualitatively by the difference maps (Figure 5), the response patterns to oxidants (ozone, nitrogen dioxide), sulfides (hydrogen sulfide), and aldehydes (formaldehyde) are substantially different.

Finer discrimination among analytes of the same class using colorimetric sensor arrays is generally possible using standard pattern recognition techniques, most notably hierarchical cluster analysis (HCA) (Askim, Mahmoudi, and Suslick 2013; Lim et al. 2009). This finer discrimination was not a major goal of the work described here. Nonetheless, in order to obtain a rough sense of the discrimination ability of the cumulative sensor array developed in this work, we examined discrimination among each of the six analytes and controls (difference maps shown in Figure 5) and also among different concentrations of a single analyte (e.g.  $\text{NO}_2$ ) using standard pattern recognition clusterification (Figure SM2). The resulting HCA dendrograms showed excellent discrimination among all analytes tested with the exception of acetic acid vs.  $\text{SO}_2$ .

In the version of the cumulative sensor array used for the Disney Exhibition, detection and identification of acid analytes was imperfect in another sense as well. Over the prolonged exposure time from artwork framing to shipping to exhibition to reshipping, that the acid sensitive sensor elements were exhausted by continual exposure to atmospheric  $\text{CO}_2$  (~400 ppm), which is, after all, an acidic gas. As mentioned, the array was also difficult to differentiate the two acidic analytes of primary interest ( $\text{SO}_2$  and acetic acid) from one another

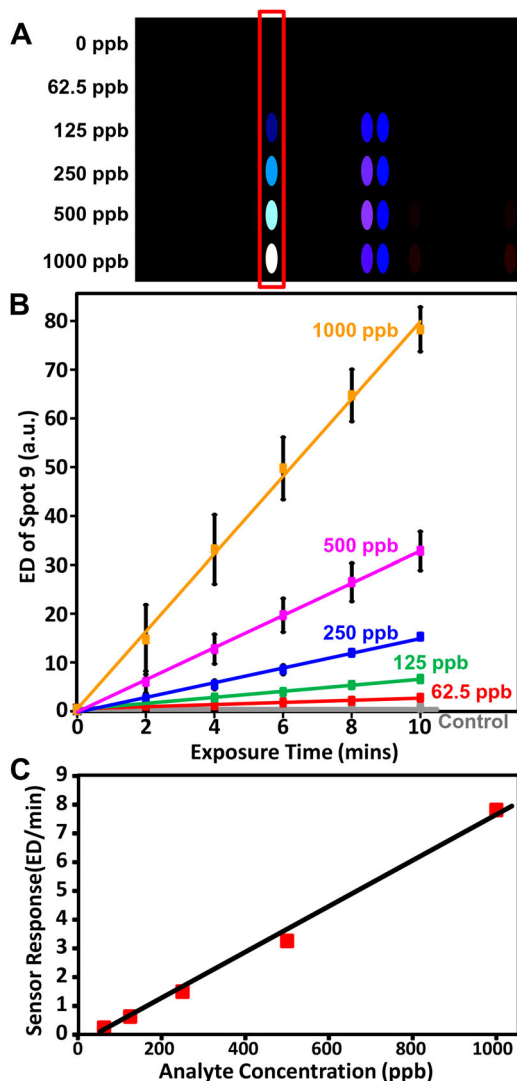


**Figure 5.** Color difference maps of arrays exposed to the major museum pollutants imaged using a flatbed scanner. All pollutants are shown at 7 ppm except ozone, which is shown at 1.5 ppm due to its higher responsivity. The sensor spot numbers (as in Table SM1) are provided at the bottom of the figure. Exposure utilized a two-minute exposure with a flow rate of 500 sccm. For visualization purposes, the RGB color values were expanded from 6 bit to 8 bit color. For quantitation, aldehyde response was measured from the color change of spot 7;  $\text{H}_2\text{S}$ , spot 9; acids, spot 23; and oxidants, spot 19.

(Figure SM2). Further work with metal nanoparticle sensor inks is underway to address this limitation.

### 3.2. Dosimetric sensitivity and TWA exposure

With cumulative sensors, the definition of detection sensitivity is a function of both analyte concentration and exposure time: dosimetric sensitivities are expressed in



**Figure 6.** Data used to determine DS and time-weighted averages (TWA) for hydrogen sulfide (H<sub>2</sub>S) exposure in a passive sampling environment over 10 min. (A) Difference maps of the sensor array color changes showing array response vs. H<sub>2</sub>S concentration; for visualization, the RGB color values were expanded from 3 bit to 8 bit color (i.e. RGB values 3–10 expanded to 0–255). The red box indicates the most responsive sensor spot. (B) Response profiles vs. time, depicting the linear, dosimetric response of the most responsive spot. (C) Calibration curve plotting optimal sensor response (i.e. ED of the color change of the most responsive spot) per exposure time vs. analyte concentration. In field tests, the measured ED of that sensor spot divided by total time of exposure determines the TWA analyte concentration.

units of concentration-time (e.g. ppb\*days), which expresses the lowest TWA exposure of some specific analyte that the sensor array is capable of measuring over some length of time.

This DS has been determined experimentally for hydrogen sulfide, formaldehyde, nitrogen dioxide, and ozone. Using a passive sampling environment with imaging on a flatbed scanner (Figure 3A), arrays were exposed to the target analyte at 0, 62.5, 125, 250, 500, and 1000 ppb in 50% RH filtered air for up to 2 h. As an example, the color difference maps and their analysis representing the response of the array to hydrogen sulfide are shown in Figure 6.

From the data collected in these experiments, one may define an apparent LOD ( $LOD_{app}$ ) for exposure over a specific fixed time to a specific analyte concentration by Equation (2):

$$LOD_{app} = (3*N*[A])/ED_t \quad (2)$$

where  $[A]$  is analyte concentration in ppm,  $N$  is the noise determined from multiple images of the same array, and  $ED_t$  is the Euclidean distance of the color changes ( $\Delta RGB$ ) of the single most responsive spot at a given time (e.g. after 2 min exposure). The extrapolated  $LOD$  was determined by plotting  $LOD_{app}$  vs. analyte concentration (ppm). A second order polynomial fit was used to extrapolate the  $LOD$  as the analyte concentration,  $[A]$ , approaches the  $LOD_{app}$ .

The  $DS$  (in units of ppb\*days), was then determined using the Equation (3) where  $t_{exp}$  is the exposure time in minutes for which the  $LOD$  was determined:

$$\begin{aligned} \text{Dosimetric Sensitivity (ppb*days)} &= DS \\ &= LOD (\text{ppb}) * \frac{t_{exp}(\text{min})}{1440 \text{ min}/1 \text{ day}} \end{aligned} \quad (3)$$

Processing of the data from these experiments can be taken a step further to produce a calibration curve to give a TWA exposure (in ppb) over some total exposure time. If one plots the optimal sensor response (i.e. the Euclidean distance of the color change of the most responsive spot) per exposure time vs. analyte concentration, then a linear fit of this data (Figure 6C) can be used to determine the TWA analyte concentration from the color changes recorded in real-world field observations.

Table 2 gives the dosimetric sensitivities achieved with our array in passive sampling environments. For all analytes, sensor array sensitivities are vastly better than standard Draeger tubes for each specific analyte. The sensor arrays are far less expensive to produce compared to Draeger tubes and a single sensor array works for multiple analytes. Due to the open exposure of our printed sensor arrays to the atmosphere, passive sampling sensitivities



**Table 2.** Dosimetric sensitivities (DS) achieved during passive sampling with the colorimetric sensor array compared to Draeger tubes, as taken from online product literature (Draeger Co. 2016).

Analyte	Sensor array (ppb*days)	Draeger tube (ppb*days)	Improvement factor
Hydrogen sulfide	0.05	430	8600×
Formaldehyde	1.12	70	60×
Nitrogen dioxide	0.03	430	14000×
Ozone	0.04	20	500×

Note: Improvements in DS are given.

are improved substantially compared to conventional diffusion tubes. Quantitative comparisons to tests involving tarnishing of metal surfaces, i.e. the Oddy test developed originally at the British Museum in 1973 (Oddy 1973) and further expanded since (Bamberger 2012; Bamberger, Howe, and Wheeler 1999; Wang et al. 2011; Wang et al. 2012), are difficult due to the long evaluation times (days to weeks) required by the Oddy test and the subjective and qualitative nature of the evaluation (Pretzel and Shibayama 2003; Wang et al. 2011).



**Figure 7.** *Drawn from Life: The Art of Disney Animation*, the first exhibition of artwork from the Walt Disney Animation studios shown at the National Museum of China in Beijing from June 30 through August 18, 2015. Artwork ©Walt Disney Co.

### 3.3. Field-testing of sensor arrays

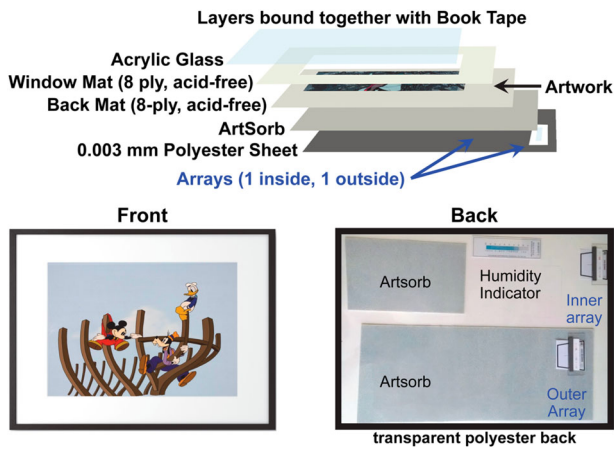
The first exhibition of artwork from the Walt Disney Animation studios took place at the National Museum of China in Beijing from June 30 through August 18, 2015. Featuring over three hundred art objects, many of which have not been shown before to the public, *Drawn from Life: The Art of Disney Animation* demonstrated the 90-year legacy of the Walt Disney Animation Studios (Figure 7).

To test the sensor arrays for application to monitoring the exposure of artwork during shipping and exhibition, a collaboration among the University of Illinois (UIUC), the Walt Disney Animation Research Library (ARL), and the Getty Conservation Institute (GCI) were established. Sensor arrays were placed at select locations (i.e. inside and outside of sealed and framed artworks, in sampling boxes placed in galleries, and in shipping crates storing artwork during travel). The sensor arrays were imaged at key times during the exhibition process: when initially mounted to the artwork, upon arrival at the Beijing exhibition site, and upon departure from the exhibition. All imaging of the sensor arrays used the digital camera of an iPhone 4S, as described earlier in Section 2.2.2.

#### 3.3.1. Sensor arrays for passe-partout frames

The method of passe-partout mounting, generally used for works on paper, produces a microclimate for the artwork inside a mat package. This packaging is used to protect artwork from ambient pollutants potentially present in the atmosphere surrounding works of art. Two sensor arrays were mounted to the back of eight separate pieces of artwork: one inside the transparent polyester back wrapping and one outside of the backing (Figure 8). The list of the passe-partouts that were monitored with sensor arrays, a description of the composition of the art, crate number used for shipping to the Beijing exhibition, and floor plan of the gallery space are given in Table SM2 and Figure SM3.

During control experiments conducted at UIUC, we discovered that the interior of the passe-partout was off-gassing a sulfide contaminant. To determine which passe-partout material was responsible for the sulfide emission, the cellphone imaging platform was used to image arrays exposed to each of the passe-partout materials individually in a passive sampling environment (Figure 3A). Array exposure to the materials was done by first imaging the array, then placing the array in a small polyethylene bag with the material of interest, heat-sealing the bag under nitrogen and then imaging again 8 days later. In addition to the passe-partout materials, we also tested the material used to make the card used as a gray calibration reference used in the imaging platform itself.

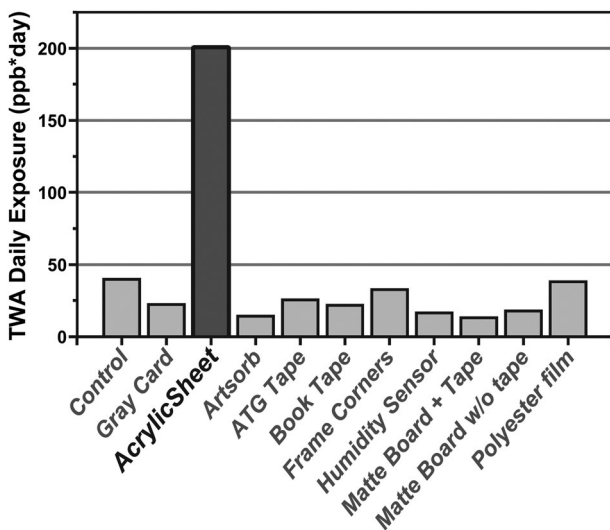


**Figure 8.** Passe-partout framing used by the Walt Disney Animation Research Library. Artsorb sheets are included to serve as a humidistat. Two colorimetric sensor arrays have been mounted to the back of the artwork: one inside the transparent polyester back sheet and one outside of the backing. The cel depicting the Boat Builders scene (bottom left) is an example of one piece in the exhibition. Artwork ©Walt Disney Co.

The TWA concentration of sulfides off-gassing from each material was determined, as shown in **Figure 9**. The source of the sulfide off-gassing was unambiguously determined as the acrylic sheet used as the front glass of the passe-partout (Acrylite OP3-plex), with an effective TWA emission of roughly  $200 \pm 80$  ppb\*days.

### 3.3.2. Sensor arrays in shipping crates

The shipping crates used to transport artwork for the exhibit were made from AC grade Plywood and heat-treated, kiln dried #2-grade pine with Tyvek liner. Sensor



**Figure 9.** TWA concentration of sulfide emission calculated from array response to the individual materials in the passe-partout packaging. The acrylic sheet is the only material that shows significant off-gassing of sulfides ( $200 \pm 80$  ppb\*days).

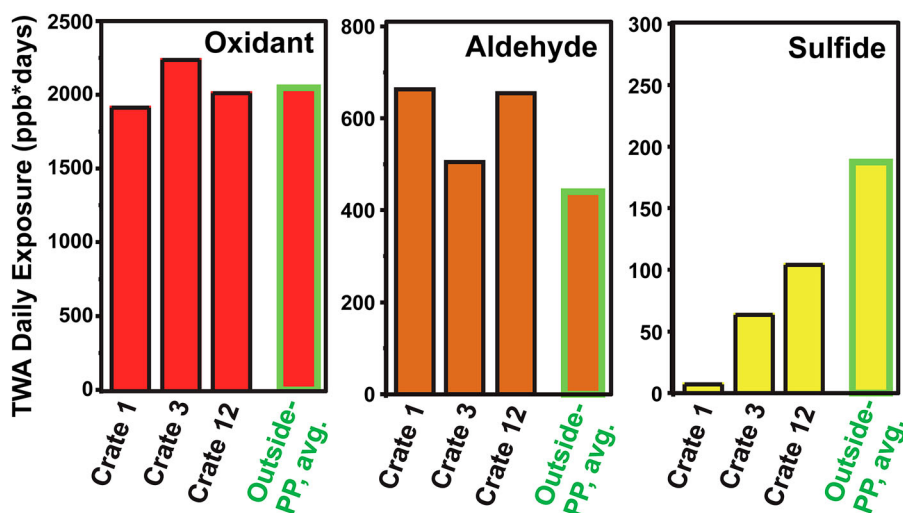
arrays were mounted on the interior (i.e. inside of the Tyvek liner) of the three shipping crates that were used to transport the art pieces that had sensor arrays mounted on the back of their passe-partout frames (both inside and outside).

To differentiate any changes in the sensor arrays due to aging from changes that occur due to pollutant exposure, a comparison was made to non-traveling control arrays. Images were taken at the same length of time after printing of both the traveling sensor arrays mounted on crates or artwork and of a separate set of three arrays kept in a controlled 50% RH filtered air environment at the University of Illinois: i.e. analysis was made on the difference of differences,  $\Delta RGB_{Traveling}$  minus  $\Delta RGB_{Control}$ . The traveling and control sensor arrays all came from the same printing batch. To monitor any aging effects on the arrays, a set of control arrays were imaged about once a week on-site at UIUC using an imaging platform identical to the one used to image the traveling arrays, as described in Section 2.2.2; the air inside the storage bag containing the control arrays was flushed with fresh filtered air weekly.

A semi-quantitative analysis of pollutant exposure was generated as described in Section 3.2. The response of the oxidant, aldehyde, and sulfide sensitive spots of the sensor arrays was converted to a dosage expressed as a TWA Daily exposure (ppb\*days) for each class of pollutant, as shown in **Figure 10**. The comparison was made between the sensor arrays mounted inside the crates and the arrays mounted on the *outside* of the passe-partout frames (labeled as outside-PP) of artwork carried inside the crates.

Oxidant exposures measured by the arrays mounted to the crates and by the arrays outside of the passe-partout frames within the crates are quite similar between the crate arrays and the outside-PP arrays (**Figure 10** left). Given that the oxidant sources during shipping are external (e.g.  $NO_x$  and ozone in the atmosphere), these results are as expected. **Figure 10** does, however, emphasize the importance of pollutant exposure during shipping, even during the relatively short period of flying time.

Aldehyde exposure was generally higher with the sensor arrays mounted inside the crates than with the arrays mounted outside the passe-partout of artwork within the crates, by roughly 40% (**Figure 10** middle). This is likely due to the well-known emission of formaldehyde from wood (Schäfer and Roffael 2000). Even with the use of Tyvek liners, the close proximity of the crate arrays to the wood crate material appears to be the likely source of higher aldehyde exposure. The containment of the passe-partout frames within wrappings within the crates appears to partially ameliorate aldehyde exposure.



**Figure 10.** Oxidant (left), aldehyde (middle), and sulfide (right) exposure of individual arrays mounted to pine plywood crates with Tyvek liners compared to average response of sensor arrays mounted to the *outside* of artwork passe-partout (PP) frames within the same crates. TWA exposures (ppb\*days) were measured on arrival in Beijing at days 19–21 after initial mounting of arrays in ARL.

In contrast, sulfide exposure of arrays mounted to the artwork is consistently and substantially higher than the sensor arrays mounted to the crates (Figure 10 right). This is probably due to the discovered source of sulfide out-gassing (the acrylic glass, cf. Section 3.3.1) from the pass-partout.

### 3.3.3. Results from Beijing exhibition

By having two arrays mounted on the back of artwork with one outside the passe-partout and one inside, we have an immediately useful comparison of the external pollutant exposure to that actually seen by the artwork inside the sealed frame. Qualitatively, this can be easily illustrated by the color difference maps generated from subtracting the RGB values of each spot in the image taken at time of mounting from the image taken at the end of the exhibit, in this case, 77 days later. Such a comparison of difference maps is given in Figure SM4.

In a qualitative comparison of the magnitude of color change from arrays mounted on the outside of the passe-partout vs. the inside of the passe-partout, it is clear that the passe-partout mounting is very effective in keeping atmospheric pollutants from interacting with the art pieces (Figure SM4 and SM5). We see multiple spot responses that are much greater for the sensor array outside of the passe-partout than for the sensor inside. We suggest that a significant part of this successful diminution of pollutants inside the passe-partout may be due, at least in part, to the sorption of pollutants by the Artsorb, which is a high surface area silica in sheet form meant to control humidity. One might expect the poly film that covers the back of the passe-partout to be a minor barrier to pollutants over a period of a few

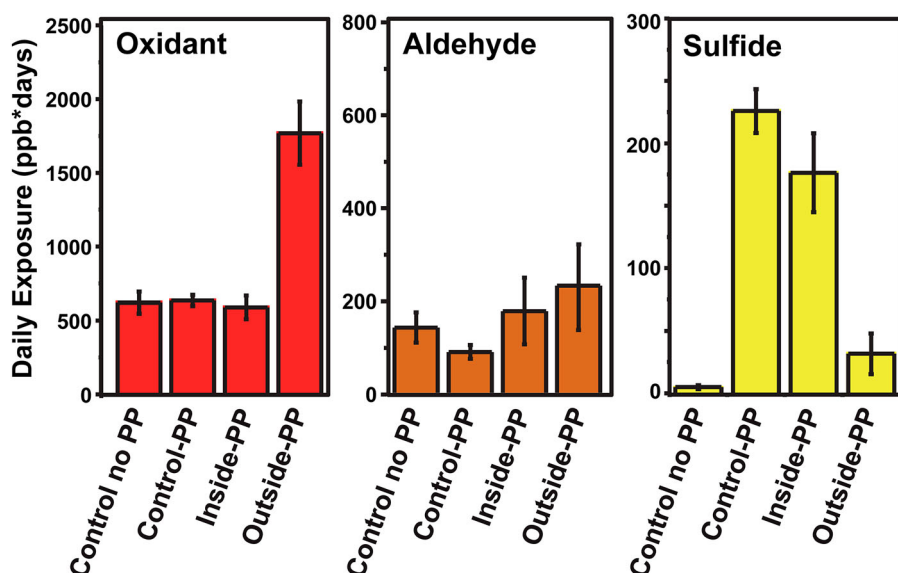
days, but the construction of the passe-partout is certainly not hermetically sealed over the period of a month. Silica gel (which makes up Artsorb) is certainly well-known as a general sorbent for VOCs. Further quantitative investigation of the sorption of VOCs and pollutants by Artsorb is warranted.

In contrast, the sulfide sensitive spot (#9, boxed in red in Figure SM4) showed nearly no exposure in the sensor arrays mounted *outside* of the passe-partout, whereas the same sensor spot on arrays *inside* the passe-partout responded strongly to sulfides produced inside the sealed frame. As previously discussed, this is due to the internal source of sulfides inside the passe-partout: the acrylic glass within the closed microenvironment inside the passe-partout frame.

Figure SM5 shows quantitative response profiles for spots sensitive to each class of pollutants from (1) the date the arrays were mounted at the ARL in Glendale, (2) to their arrival at the Beijing Exhibition (19–21 days), and (3) up to the conclusion of the exhibition (77 days after the original mounting of arrays). Average total array responses from all artwork are given for sensor arrays mounted outside of the passe-partout frames vs. those inside the passe-partout at all three time-points. For the arrays mounted in the shipping crates, the average response is given at the end of shipping to Beijing (days 19–21); as discussed earlier, during shipping, the aldehyde and oxidant responses were similar among arrays mounted to the outside and inside of the passe-partout packaging.

The total responses of the sensor arrays during the full 77 days of the shipping plus exhibition are shown in Figure SM5. Oxidant exposure outside of the passe-





**Figure 11.** TWA daily exposure (ppb\*days) to oxidants (left), aldehydes (middle), and sulfides (right) over the course of the Beijing exhibition for each of the environments listed on the x-axis. Dosage (ppb\*days) compared the array images upon arrival in Beijing (days 19–21) to images at the end of exhibition (day 77). Note that the Daily Exposures are plotted on the same scale as Figure 10. Labels: Inside-PP, arrays mounted to the inside passe-partout; Outside-PP, arrays mounted to the outside of the passe-partout; Control-PP, non-traveling arrays mounted to passe-partout packaging imaged at UIUC; Control no PP, arrays aged without the presence of passe-partout, exposed to 50% RH filtered air environment and imaged at UIUC.

partout is *dramatically higher* than that measured inside, by a factor of more than 7.0 at day 77. Aldehyde response inside the passe-partout during exhibit was nearly unchanged from its exposure during shipping, but the external sensor continued to respond to atmospheric aldehydes; total exposure to aldehydes diminished slightly (but within the error bars of our analysis) by the end of the exhibit. These results support the effectiveness of the sealed frame at preventing oxidants and aldehydes present in the gallery space from coming into contact with the artwork itself. Consistent with our previous discussion, the environment inside the passe-partout has a significantly higher sulfide level than the outside environment due to the internal sulfide source within the passe-partout.

Exposure during the exhibition itself is shown in Figure 11. For this analysis, the before and after images of the sensor arrays were those taken of the arrays upon arrival in Beijing (days 19–21) and at the end of the Exhibition in Beijing (day 77). Figure 11 gives the time-weighted daily exposure (ppb\*days) to sulfides, aldehydes, and oxidants over the course of the Beijing Exhibition ( $\approx 57$  days). The daily exposures plotted in Figure 10 are the same scale as Figure 11. Notably, the exposure outside of the passe-partout during shipping is high compared to the relatively controlled exhibition environment (especially for aldehyde exposure).

The difference between oxidant exposure of the arrays mounted to the outside of the passe-partout over the

course of the Beijing exhibition is substantial, greater than a threefold difference. This reaffirms that the passe-partout is effective in preventing oxidants from the environment outside the passe-partout from building up within the passe-partout. Daily exposure to aldehydes was higher outside the passe-partout than inside, but still within the error bars of the analysis.

Daily exposure to sulfides for arrays mounted *inside* the passe-partout during the Beijing Exhibition is substantially higher (by roughly sevenfold) than the exposure outside. The larger gallery environment substantially decreased the concentration of sulfides outside of the passe-partout microenvironment: sulfide concentrations within passe-partouts (either in control experiments at UIUC or in field tests in Beijing) were  $\sim 200$  ppb\*days vs. 25 ppb\*days outside of the passe-partout.

In taking the analysis of sulfide exposure a step further, systematic differences were observed depending on the nature of the material making up the artwork. Sensor arrays mounted on the inside of a passe-partout encasing a painted cellulose acetate cel (whether it was a reproduction or an original) showed significantly lower average daily dosage of sulfide than arrays inside a passe-partout encasing an animation drawing on paper (i.e. graphite, pen, or Conte crayon) (Table 3). Location within the galleries did not affect the exposure measured. One possible explanation is that the cellulose acetate substrate or the paint upon it absorbs sulfide volatiles more effectively than the paper drawings.



**Table 3.** TWA daily exposure to sulfides inside passe-partout for artwork containing paints on cellulose acetate (cels) vs. drawings on paper.

Artwork material	Artwork	Sulfides, TWA exposure (ppb*days)
Cel	Boat Builders	80
Cel	Snow White	100
Cel	Lady and the Tramp, 2015	80
Cel	Lady and the Tramp, 1955	80
Paper	Jungle Book, pen	150
Paper	Snow White, graphite	120
Paper	Lady and the Tramp, Conte	150

Note: Data taken at the end of the Beijing exhibition (77-day exposure).

#### 4. Conclusions and outlook

A colorimetric array of cumulative (dosimetric) sensors has been developed for the portable and inexpensive monitoring of air pollutant exposure of cultural heritage objects. A system for exposing these arrays in passive sampling environments has been used to determine dosimetric sensitivities to important classes of pollutants and to provide TWA concentrations of pollutants in the museum and exhibition environment. To quantitatively measure the color changes of these sensor arrays, a facile imaging method using an ordinary cellphone camera has been created and proven functional even during exhibition and shipping of artwork. With a colorimetric sensor array and cellphone imaging, we were able to calculate TWA daily exposure (in ppb\*days) to sulfides, oxidants, and aldehydes in a variety of environments. Further work is needed (and underway) for identification of acidic pollutants during prolonged exposure, in large part due to the cumulative effects of long-term (months) exposure to atmospheric carbon dioxide.

The sensor arrays were used to monitor the environmental exposure to pollutants of artwork from the Disney Studios during shipping to and on display in Beijing as part of the *Drawn from Life: The Art of Disney Animation* exhibition. We have demonstrated quantitatively the effectiveness of the passe-partout packaging in protecting artwork from sulfide, oxidant, and aldehyde pollutants as well as determine a source of emission of sulfides from the passe-partout materials themselves. The importance of exposures to pollutants during shipping is particularly worth noting.

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No potential conflict of interest was reported by the authors.

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*Kristen McCormick* is the Art Collections & Exhibitions Manager of the Walt Disney Animation Research Library, Glendale. Kristen has been at the Walt Disney Company for over a decade and a half where she has been responsible for the safe keeping, care, and transport of a broad range of artworks from African Art to Animation. In her current role, she oversees the conservation care of the Walt Disney Animation Collection which comprises of over 64 million pieces of artwork, from all facets of the production process including storyboard drawing, visual development, layouts, backgrounds, animation drawings, and animation cels. Kristen is also responsible for the traveling exhibitions and loans related to this Collection, with extensive tours around the world, including Taiwan, Korea, Australia, Japan, China, and Europe.

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