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Quantification of Color-Changing Papers (M8) Using Industrial Devices and Methods

Darren K. Emge
Kathy Crouse
Eric Languirand
Michael W. Ellzy

DIRECTORATE OF PROGRAM INTEGRATION

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The use of "eye-readable" colorimetric technology to detect chemical warfare agents dates back to the development of M8 paper by Canadian scientists in 1963. Currently, a number of studies are attempting to augment M8 paper or develop other new colorimetric indicators. In this study, we demonstrate a German commercial device, the Micro-Epsilon (Ortenburg, Germany) CFS colorSensor CFO200 sensor, and industrial methods using the International Commission on Illumination (Vienna, Austria) standardized perceptual color space known as CIELAB to document the ability to quantify sample variations in M8 paper. More importantly, we also document the level of color change in color-changing paper such as M8 when it is exposed to H-, G-, and V-class agents. This ability to quantify color change levels and their relationship to the observable differences will be essential to developing and determining the efficacy of eye-readable colorimetric technologies.

15. SUBJECT TERMS

Color-changing paper	CIELAB color space (L*a*b*)	Eye-readable	M8 paper
Color quantification	CFS colorSensor CFO200	Observable difference	Color sensor

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PREFACE

The work described in this report was started in February 2021 and completed in March 2021.

The use of either trade or manufacturers' names in this report does not constitute an official endorsement of any commercial products. This report may not be cited for purposes of advertisement.

This report has been approved for public release.

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EXECUTIVE SUMMARY

The use of colorimetric technology to detect chemical warfare agents dates back to the development of M8 paper by Canadian scientists in 1963. M8 paper is easy to use; one must simply dab contaminated material with the paper, wait 30 s, and compare the color response to the chart on the inside cover of the booklet. The dimensions of each booklet are roughly 10 × 6.5 cm. A booklet contains 25 individual sheets, each perforated down the centerline of the long axis. Although M8 paper is simple to use, inexpensive, and a proven technology, it does show false responses to solvents and is only designed to respond to a limited range of materials, such as H-, G-, and V-class agents. Currently, there are a number of efforts and programs to augment current M8 capabilities or develop novel technological approaches, which include “eye-readable” colorimetric indicators.

In this study, we examine a German commercial device, the Micro-Epsilon (Ortenburg, Germany) CFS colorSensor CFO200 sensor, and other industrial methods used for quality control in industrial applications such as product consistency, printing, and color standardization. The industry uses the International Commission on Illumination (Vienna, Austria) standardized perceptual color space known as CIELAB. To work in this space, the CFO200 sensor uses a calibrated light source and specialized detector to record the luminance (L*), red-green (a*), and blue-yellow (b*) color responses of a small sample. The latter two responses cover color ranges that complement human vision, that is, the extremes of the ranges cannot be seen simultaneously but allow for quantification of perceptual colors and color changes.

Using CIELAB, we document the ability to quantify baseline intra- and intersample variations in M8 paper and more importantly, the level of color change in color-changing papers, such as M8, when they are exposed to H-, G-, and V-class agents. This ability to quantify the level of color change and its relationship to observable differences is essential for developing and determining the efficacy of “eye-readable” colorimetric technologies. In addition, the abundance of work in understanding and standardizing methods and devices for industrial applications can be readily leveraged to support development efforts and programs of record in the near term. Additional work will be required to establish a definitive link to metrics developed for industrial and general consumer applications to both the specialized environments (e.g., operating in personal protective equipment) and other unique requirements associated with defense operations.

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QUANTIFICATION OF COLOR-CHANGING PAPERS (M8) USING INDUSTRIAL DEVICES AND METHODS

1. INTRODUCTION

The use of colorimetric detection technologies dates back to 1963 with the development of M8 paper in Canada. In 1964, M8 paper was fielded to U.S. Forces as part of the M15A2, a semiautomatic version of the classic service rifle.[1] M8 paper is marketed as a booklet of 25 sheets. Each sheet measures 10 × 6.5 cm, has a center-line perforation along the long axis and on the left side near the spine, and is impregnated with three sensitive dye indicators. A sheet or half sheet split on the long axis is torn out for use. When the M8 paper comes into contact with a liquid, the dyes react and provide a unique, visibly perceptible response to either G-Type nerve- (yellow), H-type blister- (red), or V-type nerve (green) chemical warfare agents (CWAs).[2] M8 paper has been standardized across North Atlantic Treaty Organization forces for the detection of liquid chemical contamination.[1,3]

M8 paper is used to discern color changes due to solvation, reaction, and dispersion of the impregnated dyes via human color perception. Therefore, quantification of human-perceived color changes is necessary and was initiated by Munsell in 1933.[4] Thereafter, several perceptual color schemes and standards were developed and refined with the initial metric defined by Moon in 1943.[5] However, the current most widely used scheme in industrial, scientific, and engineering applications is CIELAB.[6] CIELAB is composed of a triple or 3-dimensional (3D) space noted as L*a*b* and is designed to quantitate human color perception in an independent space. In contrast to color systems such as red–green–blue (RGB) and hue–saturation–value, which vary depending on the presentation media (e.g., computer monitors and televisions), CIELAB is independent of media and standardizes measured colors in terms of human visual perception. The L* value is defined as perceptual lightness. It ranges from 0 (black) to 100 (the maximum intensity without damage to the eye).[6] The a* value indicates red-green color, and b* values indicate blue-yellow color; both of these range from –127 (green or blue, respectively) to 128 (red or yellow, respectively). The CIELAB space is based on an opponent color model in which opposites cannot be seen simultaneously and is therefore ideal for the quantification of human color perception.

After establishing color space, it is necessary to distinguish whether the colors are similar, identical, or different. Perceptual differences are divided into two general classes: (1) just notable differences (JNDs) and (2) just intolerable differences (JIDs). JIDs are associated with imagery, and research in this area includes not only color differences but also spatial and perceptual differences such as noise (i.e., blur and granularity),[7] which are not the focus of this effort. In this study, we focused on JNDs. In particular, we used the ΔE_{Lab} metric developed in 1976, also known as dE76 and defined as

$$\Delta E_{Lab} = \sqrt{(\Delta L*)^2 + (\Delta a*)^2 + (\Delta b*)^2}$$

One advantage in using the ΔE_{Lab} metric is that the reported shifts are independent of color change and strictly indicate a perceptual change (i.e., the ΔE_{Lab} value between red and green is equal to the one between blue and yellow).[8] Therefore, a change in ΔE_{Lab} indicates a change in human perception. The correlation for a standard observation[*9] is shown in Table 1.

Table 1. ΔE_{Lab} Metrics and Their Corresponding Differences

ΔE_{Lab}	**Noticeable Differences**
0–1	Observer does not notice a difference
1–2	Only experienced observer can notice a difference
2–3.5	Unexperienced observer can notice difference
3.5–5	Clear difference is notable
5 or greater	Two colors clearly noticeable

A Micro-Epsilon (Ortenburg, Germany) CFS colorSensor CFO200 sensor was used to measure the colors. The CFO200 sensor (Figure 1) is highly accurate and was designed for high speed (up to 30 kHZ) and precise color recognition. It is used in industrial measurement tasks, such as packaging control or color sorting. The CFO200 sensor can discern color distances of $\Delta E_{Lab} \leq 0.6$ with a repeatability of ≤0.3.

[*]The International Commission on Illumination (CIE; Vienna, Austria) has defined a colorimetric standard observer as an average person with normal color perception. The CIE has defined two standard observers with different observation angles as 2° and 10°, where 2° corresponds to the observation of an object with a small size (using an optical instrument), whereas an observer with the angle of 10° corresponds to the observation of an object in normal conditions.

Figure 1. (Left) CFO200 sensor with fiber-optic and blue connector and (right) the graphical user interface, which operates within any browser through a direct Ethernet connection.

In this work, we quantified various color-changing papers, including current inventory paper such as M8, out of date M8 paper, and samples of both Canadian 3-way paper (CAN Tricolour) and UK Mark IV (Mk4) paper (Anachemia Canada, Inc; Quebec, Canada). An initial base color was established by measuring the L*a*b* response of six locations on a single sheet of paper from seven different manufacturer lots and sources. After the base color was established, each paper sample was contaminated with six 2 µL applications of a G-series agent (sarin), an H-series agent (distilled mustard), and a V-series agent (*O*-ethyl *S*-(2-diisopropylaminoethyl) methyl phosphonothiolate). Each contaminated location was scanned for approximately 150 samples with the CFO200 sensor.

2. METHODS

We examined a total of seven different types or lots of color-changing paper. To establish a base color for each sample, a single sheet from each booklet was removed and placed under the CFO200 sensor at a distance of ~12 mm, which resulted in an interrogation spot size of ~14 mm (Figure 2). An image of each booklet is shown in Appendix A, and details are presented in Table 2.

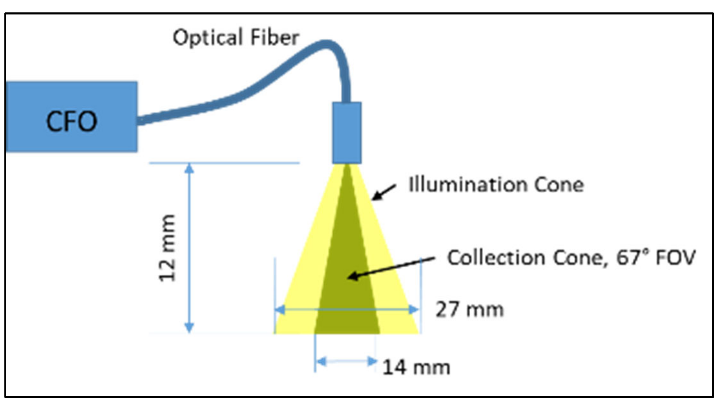


Figure 2. Schematic of data collection with the CFO200 sensor. All dimensions are approximate due to variation in paper flatness and, in the case of the illumination cone, qualitative perception of the lighted region.

Table 2. Color-Changing Paper

Manufacturer/Source	Lot No.	Mfg. Date	Exp. Date
Truetech (Riverhead, NY)	CCR14B318-001	2/14	2/24
Truetech	CCR14B318-001 (unopened)	2/14	2/24
Truetech	CCR14E318-002	5/14	5/24
Luxfer Magtech (Riverhead, NY)	CCR15K318-003	10/15	10/25
Truetech	96-2	12/03	*
UK Mk4	6665-99-225-3102**	*	*
CAN Tricolour	ANC03B031-59	2/06	*

*Not listed on packaging.
**This National Stock Number number is the only identifying markings on this packaging.

The Truetech and Luxfer papers were from U.S. stock (see Table 2 for details). The booklet with lot number 96-2 was old and was sourced from one of the U.S. Army Combat Capabilities Development Command Chemical Biological Center (Aberdeen Proving Ground, MD) laboratories. Each booklet consisted of 25 sheets, which measured 10 × 6.5 cm. Both the Canadian and UK booklets had 12 sheets of paper with an adhesive backing and physically appeared to be made of heavier grade paper than the U.S. booklets. An additional unopened, still fully sealed package of U.S. paper (Truetech lot no. CCR14B318-001) was located and used as a control to account for exposure to the laboratory environments. That package was opened on the first date of base color testing. The lot number 96-2, UK Mk4, and CAN Tricolour papers had no listed expiration date but were over 10 years old, which is the average life span for the modern lot CCR######-###, hereafter referred to as CCR papers.

For each measurement with the CFO200 sensor, the samples were placed in the field of view and several seconds of data were collected. For consistency, only the first 150 samples were used for each data file. Data were collected from six locations randomly selected for base color establishment. These six locations were chosen to take into account any variations in either the paper color baseline or the response, which was observed and is detailed below. Thereafter, data were collected from each application location where an aliquot of liquid agent was applied.

To quantify the color changes due to exposure, three standard CWAs were used to contaminate six locations on each sample page. Six 2 µL droplets of a CWA were placed on the same samples used for the base color measurements. At the completion of the last deposition, the CWA was allowed to diffuse into the sample for 1 min to ensure a developed color change. It was noted during the trials that the M8 paper responded within 30 s.[10,11] The samples were then moved under the CFO200 sensor, and data were collected on the color change (Figure 3).

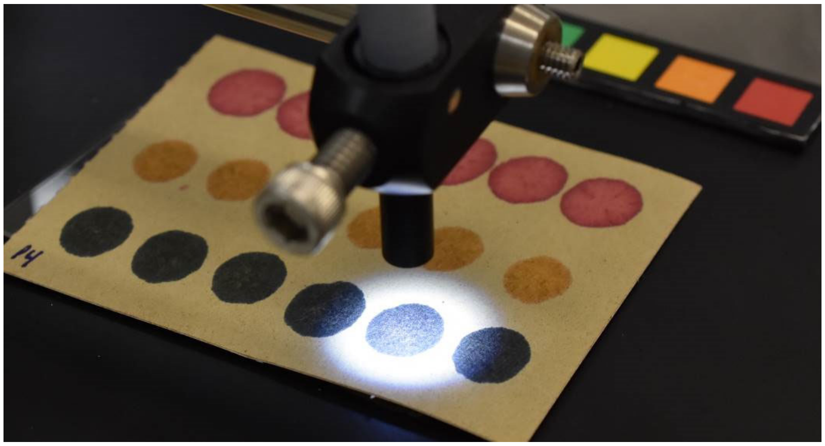


Figure 3. Collection of CIELAB data with CFO200 sensor.

By using 2 μL droplets of agent, we ensured that the color response of the M8 paper filled the 14 mm field of view of the CFO200 sensor. The observed average droplet size was ~15 mm for the G-series agent and ~18 mm for the H- and V-series agent. The illumination source shown in Figure 3 was larger than the integration field (Figure 2) and ensured uniform illumination over the interrogation area, which was incorporated completely within the droplet. After completion of the third set of droplets were placed on the paper, a standard camera image was taken of each sheet using a Nikon (Melville, NY) D7200 camera (SN 2610421) with a Nikon DX AFS micro Nikkor 40 mm 1:28G lens (SN US6064722), in programmed automatic mode P_m. The contaminated paper samples are shown in Figure 4.

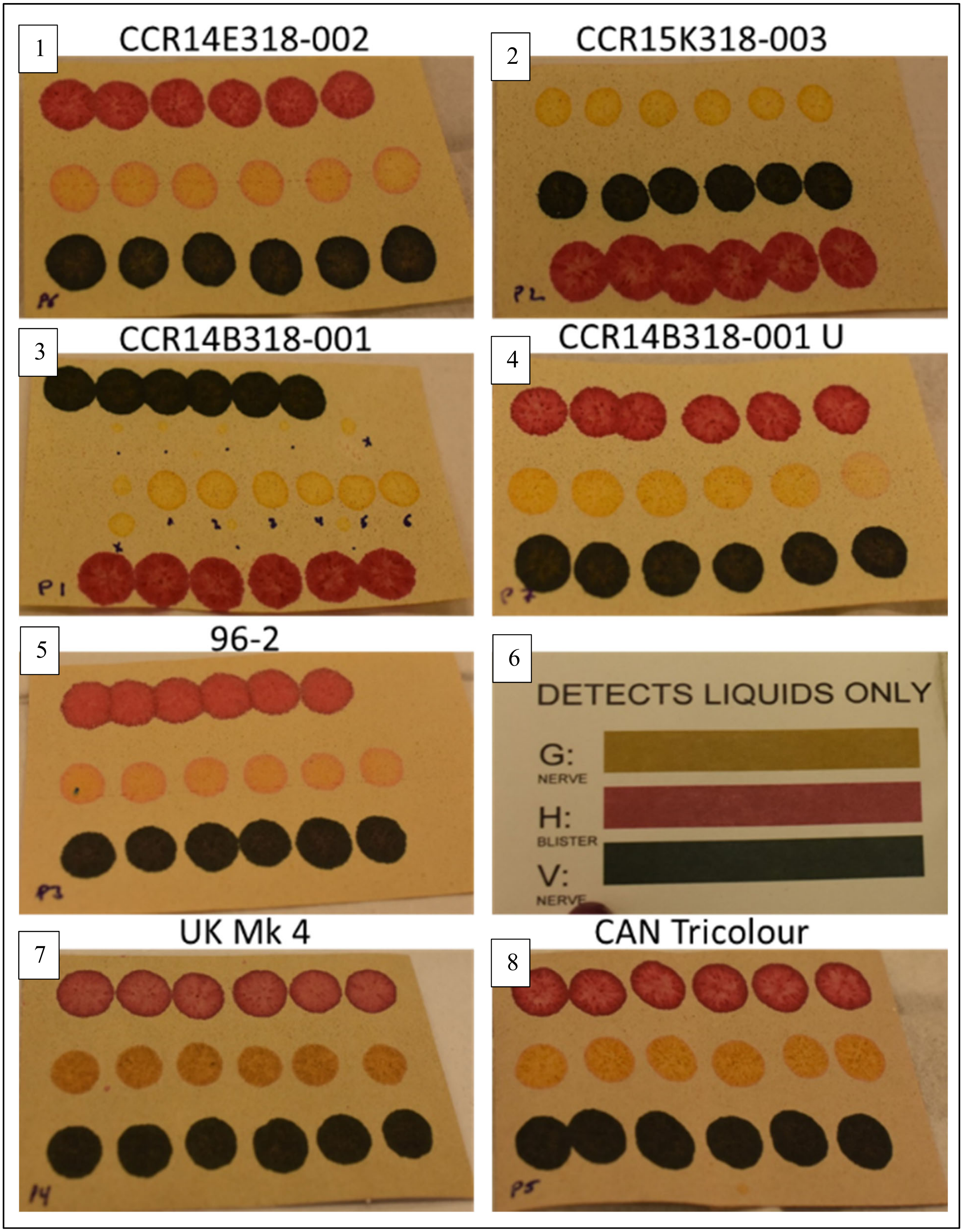


Figure 4. Final images of contaminated paper samples. A sample key (inside booklet cover) is shown in image number 6.

3. RESULTS AND DISCUSSION

The CFO200 sensor outputs all data in a comma-separated variable file. Each data file contained several seconds of data and for consistency, only the first 150 data points were used. All data were read into MATLAB 2020a software (Mathworks; Natick, MA) and processed using custom scripts. Initially, the variance within each data file was evaluated, and the average variance of the measured values was <0.026. Figure 5 shows the average for each sample location. The consistent baseline values, within each paper type and across all sample measurements, are evident in the nearly indiscernible error bars and the tightness of the y-axis values.

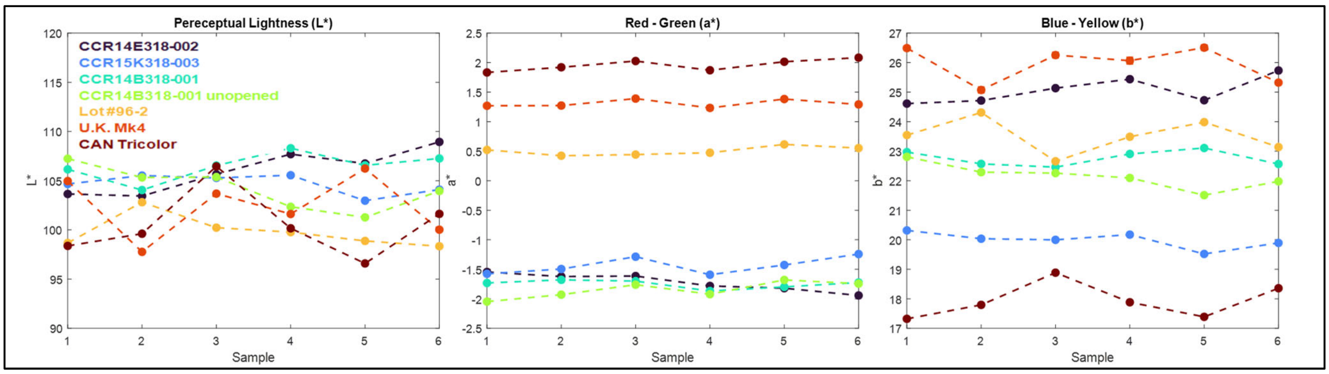


Figure 5. Average measurements from each sample location with error bars. The recorded values were uniform for each sample.

First, the base color was established for each paper sample. Figure 6 shows the average for the 3D space (L*a*b*) from each of the six locations tested on all seven samples. The data are very tightly clustered due to the limited range of the three axes.

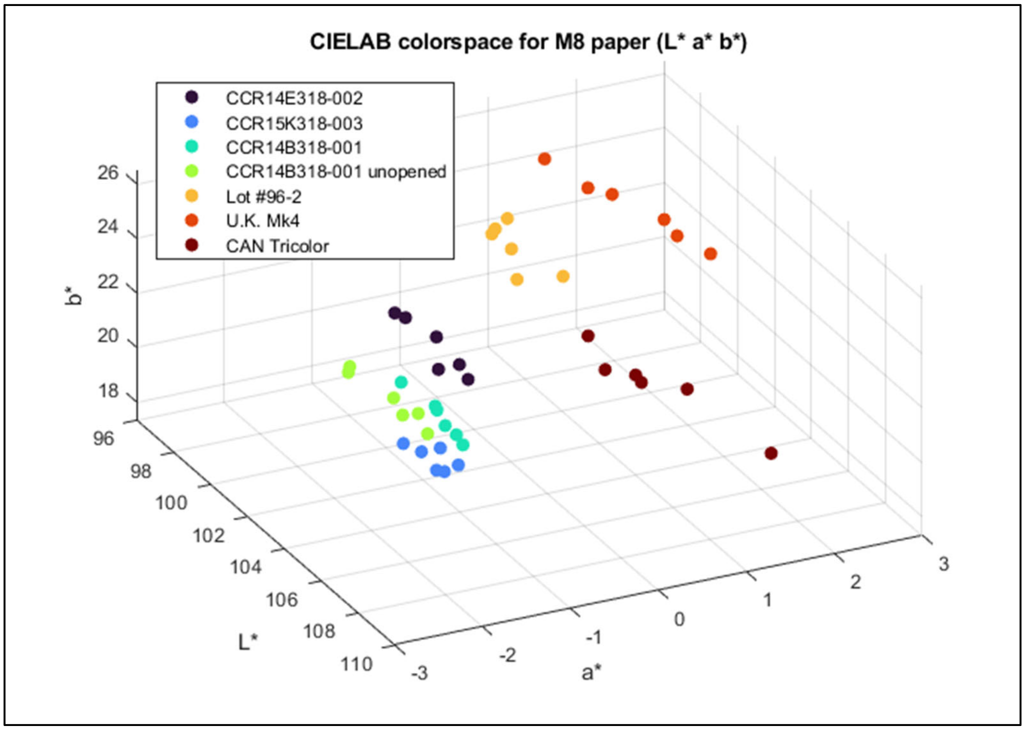


Figure 6. Measured baseline values for each paper sample in L*a*b* space.

The specific ΔE_{Lab} values are shown in Table 3. There are discernible color differences, especially between the first four (current U.S. CCR papers), and the latter three (the old stock and foreign paper types).

Table 3. ΔE_{Lab} Value Differences across Baseline Paper Measurements

	CCR15K318-003	CCR14B318-001	CCR14B318-001 (unopened)	96-2	UK Mk4	CAN Tricolour
CCR14E318-002	5.25	2.34	3.40	6.80	4.82	9.75
CCR15K318-003	–	3.32	2.25	6.34	6.95	5.79
CCR14B318-001	–	–	2.31	7.10	6.02	8.55
CCR14B318-001 (unopened)	–	–	–	5.23	5.28	6.82
96-2	–	–	–	–	3.65	5.81
UK Mk4	–	–	–	–	–	5.26

Note: 96-2, UK Mk4, and CAN Tricolour were over 10 years old at the time of testing.
–, redundant data omitted for clarity.

Figure 7 displays all of the samples of color-changing paper overlapping in a single image. The three older (>10 year) paper types are clearly less luminous (bright) than the more modern CCR papers. Both the UK Mk4 and CAN Tricolour papers are physically a heavier stock of paper with an adhesive backing and appear darker. This difference is quantified in the ΔE_{Lab} values shown in Table 3. All of the CCR samples were quantified as being different to some extent ($2 < \Delta E_{Lab} < \sim 5$) but the differences between them were not clearly noticeable ($\Delta E_{Lab} > 5$) as they were quantified to be when the CCR papers were compared with the other three types of paper.

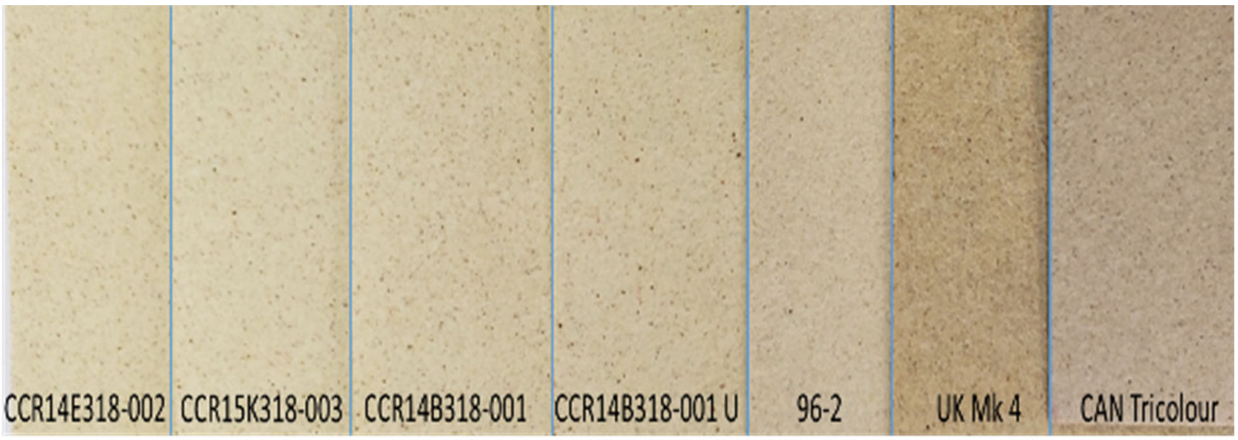


Figure 7. All seven color-changing papers overlapped for visual comparison.

The purpose of this work was to determine whether the color changes in M8 paper and its equivalents after exposure to CWAs could be quantified using industry standard measurement devices and methods. Figure 8 shows the average baseline CIELAB color values (stars) as well as the recorded changes when the samples were contaminated with each of the three agent classes: G (circle), H (square), and V (triangle). Each state of the paper, clean or exposed, is clearly grouped, as indicated by the elliptical shaded regions. Each ellipse is located at the centroid of all the observations, and the extent covers the full range of values. In all cases, the responses of the CCR papers tend to cluster more tightly than the general population. For the H and V agent classes, the responses for the out of date 96-2 paper are clustered close to the CCR responses. It is interesting to note that the 96-2 paper G-type response is separated from the CCR G-type response, which may be indicative of the degradation in the dye used for that particular class of agents. The UK Mk4 and CAN Tricolour are unique when compared with the U.S. CCR papers for all agent classes. They are, however, still similar when compared to U.S. CCR papers, as shown in their clustering.

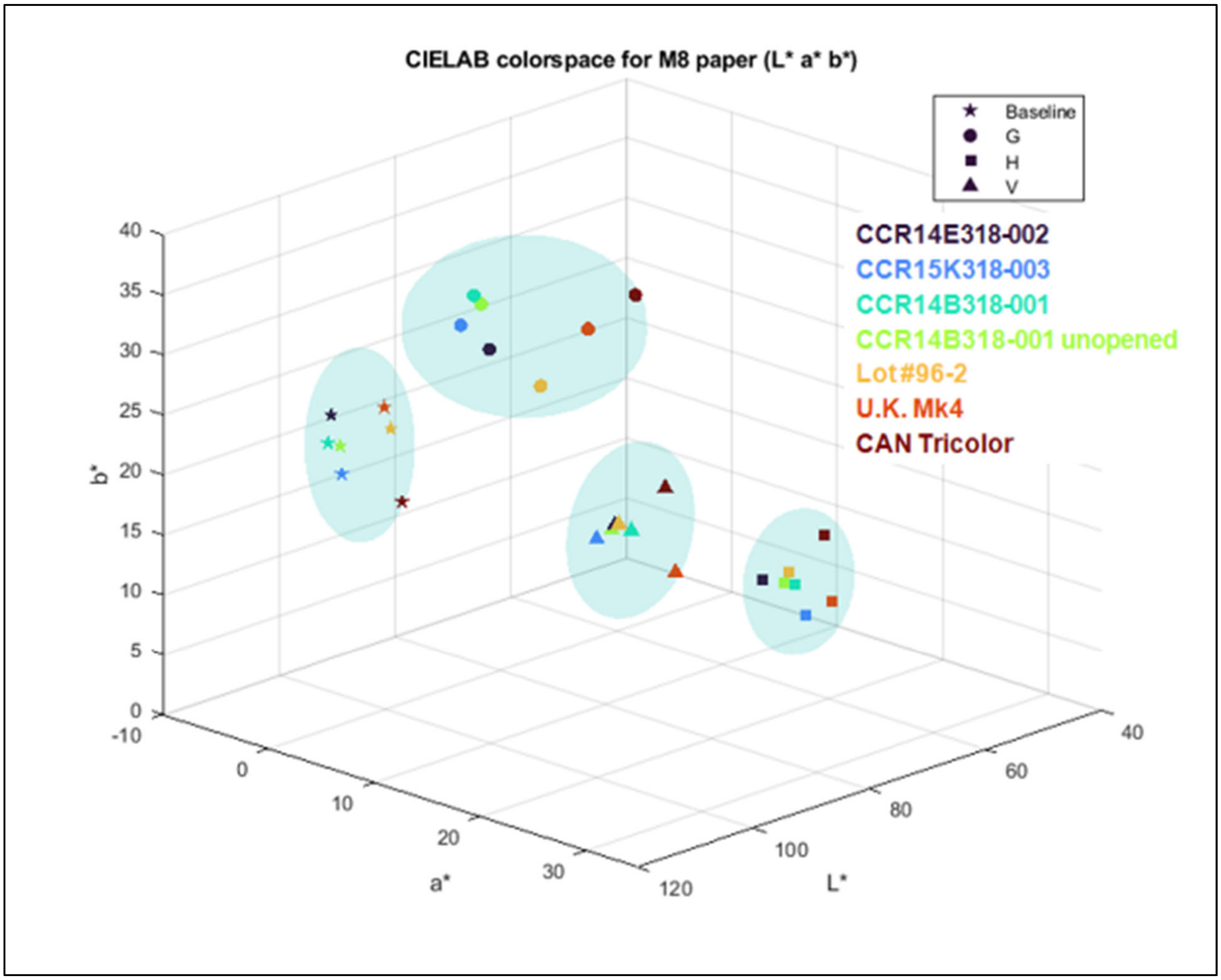


Figure 8. Average 3D space (L*a*b*) locations for all seven paper types: unexposed (star) and exposed to agent classes G (circle), H (square), and V (triangle). Each is clearly clustered in a unique region of this 3D space.

A direct comparison of the ΔE_{Lab} values is shown in Figure 9. A clearly noticeable color change can be seen for all seven paper samples based upon the industry standard ($\Delta E_{Lab} > 5$). The extent of the discernibility in all of the cases aligns with physical observations made during data collection (Figure 4). The color change for the G-series agent was observed as tan to dark yellow, which was not as dramatic as the change to red and green for the H- and V-series agents, respectively. This is exemplified through the ΔE_{LAB} value, which is lower in magnitude for the G-series agent across all types of paper when compared with the H- and V-series agents in Figure 9.

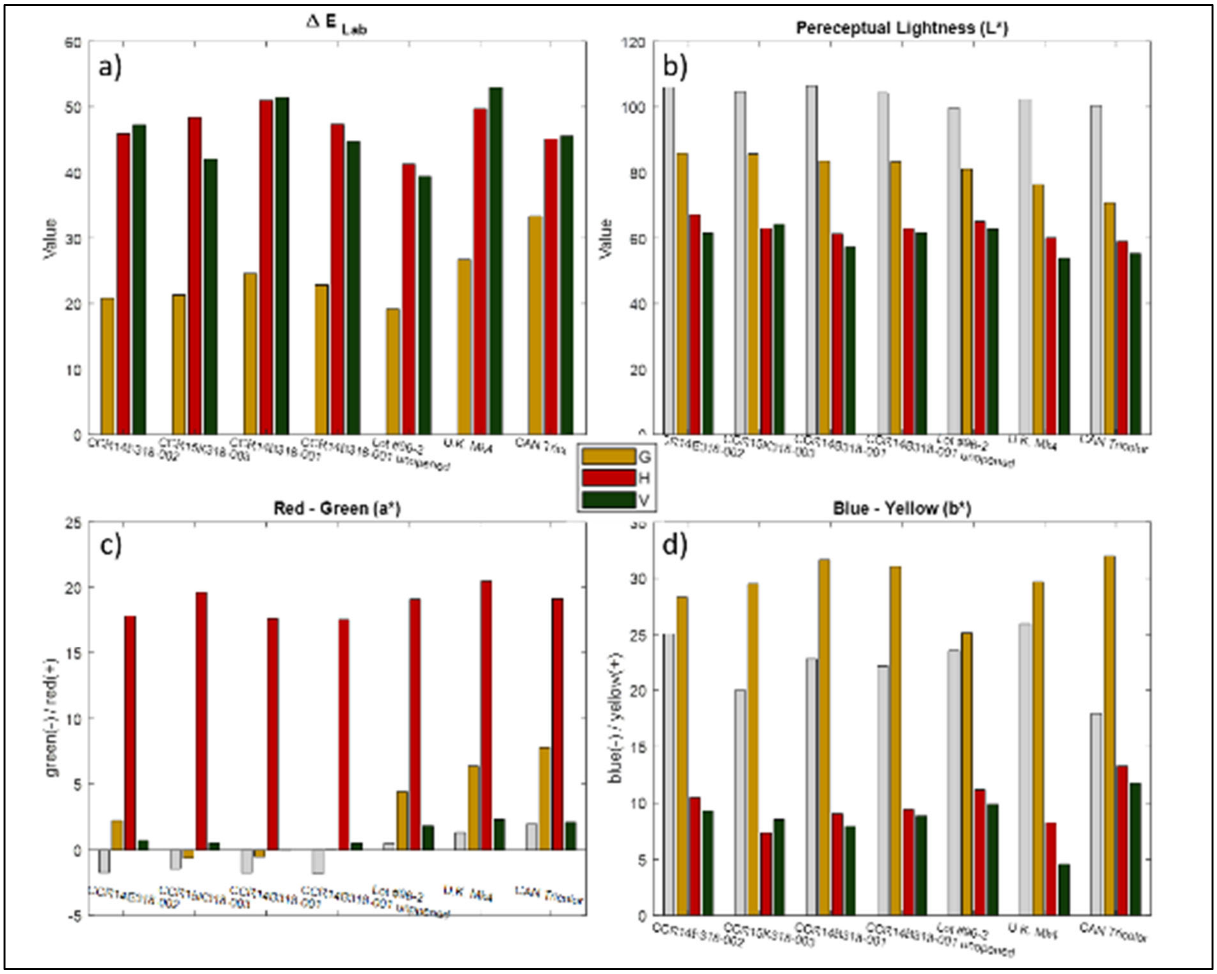
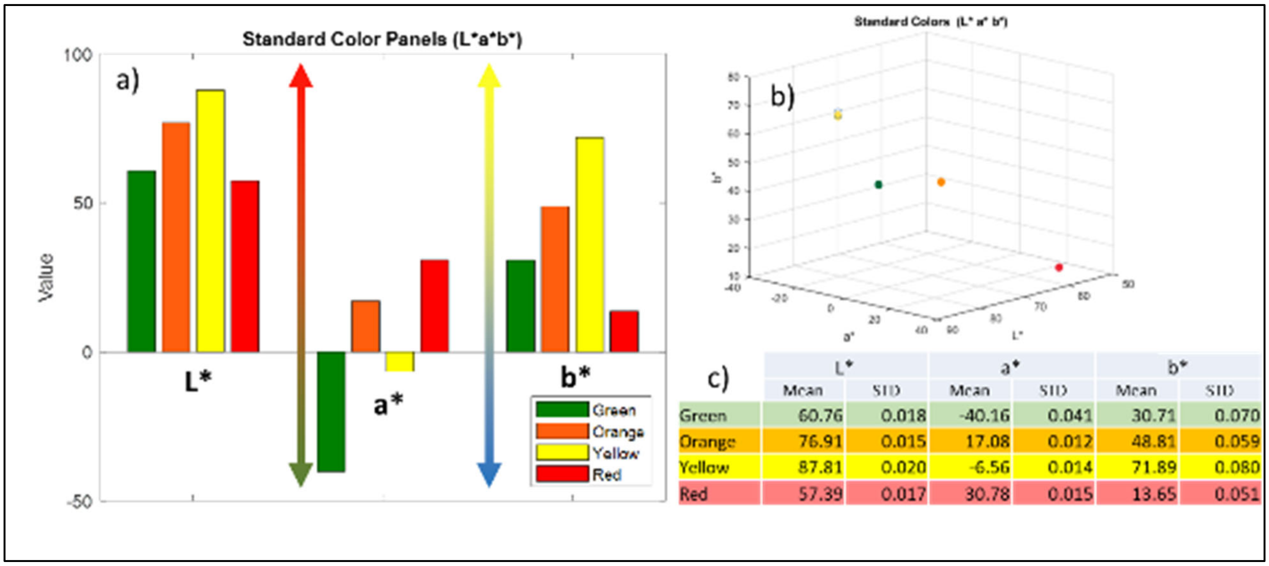


Figure 9. (a) Direct comparison of ΔE_{Lab} values and each of the measured CIELAB values, (b) L* space, (c) a* space, and (d) b* space. In Figure 9b–d, the baseline, uncontaminated values are shown as gray bars for reference.

Comparisons of each channel in the L*a*b* space are shown in Figure 9b, c, and d, respectively, for the baseline measurements in gray and exposed responses (key in center of the figure). The perceived lightness (Figure 9b) corresponds to an observed darkening associated with the color changes, with the G-series agent transitioning slightly from light tan to dark yellow. The deeper color changes associated with the H- and V-series agent result in lower perceived lightness values, as expected. The red–green channel a* (Figure 9c) aligns with the observed red when the papers were exposed to an H-series agent. In contrast, the green response, which would correspond to exposure to a V-series agent, is not readily apparent in this channel. The blue–yellow (Figure 9c) color offers some insight into the lack of green color change for the V-series agent. As expected, the G-agent caused a significant yellow response. Color changes for both H- and V-series agents also have similar yellow responses.

Examination of the L*, a*, and b* values associated with the green, orange, yellow, and red panels of a standard color plate (X-Rite [Grand Rapids, MI] ColorChecker Passport Photo, model MSCCPP) helps to clarify the above findings. In Figure 10a, the measured values for the standard panels are shown in a bar plot for easy comparison with data shown in Figure 9. In particular, the response to the green panel is positive in the b* direction, which, as shown in the transition arrow, is green in color. The V-series agent response is very dark in color (Figure 4), which could be the source of the strong yellow response in Figure 9d. Figure 10b shows very good separation of the measured values, with larger differences in the values attributed to the brilliance and purity of the standard color plates in contrast to the more subdued and muted colors observed in the paper responses. For the sake of completeness, the mean and standard deviation values are provided in Figure 10c.



	L*		a*		b*	
	Mean	STD	Mean	STD	Mean	STD
Green	60.76	0.018	-40.16	0.041	30.71	0.070
Orange	76.91	0.015	17.08	0.012	48.81	0.059
Yellow	87.81	0.020	-6.56	0.014	71.89	0.080
Red	57.39	0.017	30.78	0.015	13.65	0.051

Figure 10. Measured values of standard color plate in the L*a*b* 3D space:
(a) direct comparison of measured values with transition arrow for the a* and b* spaces shown for perceptual reference, (b) 3D space representation for reference and to show separation, and (c) mean and standard deviation of measured values. Note: standard color plate can be seen at the rear in Figure 3.

One interesting data point observed was for CCR14B318-001 (unopened). The response for the sixth droplet of the G-series agent was visibly lighter than that of the other five droplets (Figure 11). The distance between the first five droplets is shown as $\Delta E_{Lab} < 2.9$, which is just perceptible to the untrained observer. In contrast, the ΔE_{Lab} for the sixth droplet is 8.6, which is significantly different and removed from the centroid of the cluster composed of the first five droplets. This value quantifies the discernible difference between the first five drops and the last one, thus supporting the observation of the operators that this droplet was different from the others.

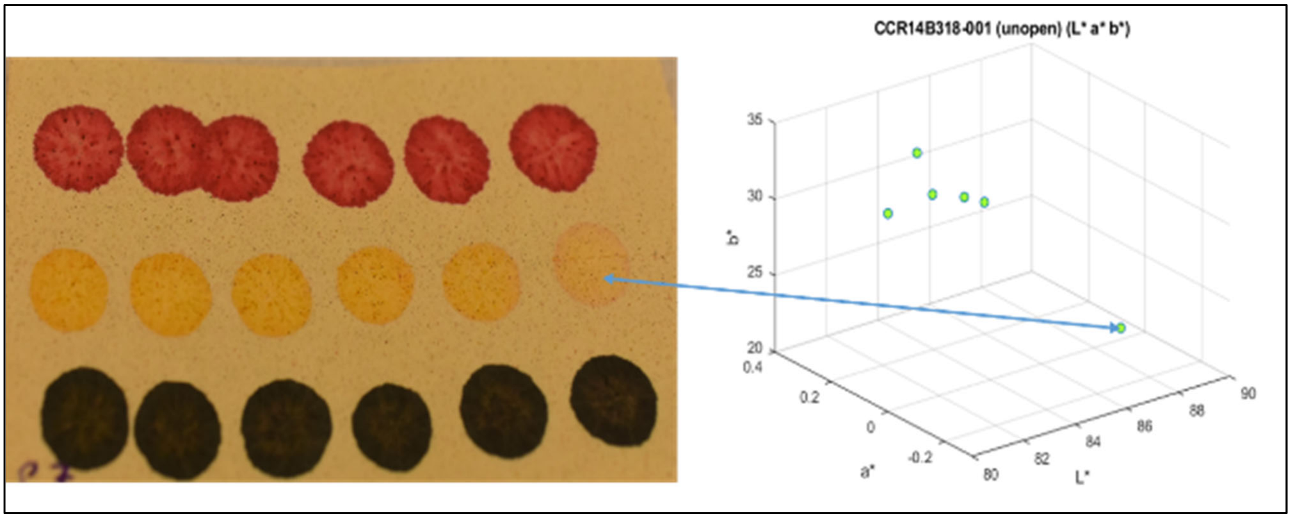


Figure 11. (Left) CCR14B318-001 (unopened) shows the sixth droplet of G being (right, center row) visibly lighter and (right) close-up of only the CCR14B318-001 (unopened) CIELAB values shows that the sixth point shifted in the a* and b* spaces.

4. FUTURE WORK

This study was limited in scope to establish the utility and capability of the CFO200 sensor for the quantification of colorimetric changes in the M8 and other color-changing papers. A variety of extensions, tests, and experiments are left for future work, the foremost being the addition of a perceptual element. A perceptual study using an expert design set of subjective metrics should be developed and implemented. The cooperative development of a set of visual perceptive-related questions with an outside expert in ergonomics, physiology, or psychology would provide a qualitative validation between the ΔE_{Lab} and laboratory personnel, the warfighter and specialist, and subjective evaluation, thereby addressing possible user bias. This should be expanded to include the examination of visual interpretation of known interferent response to establish any relationship between the subjective surveys and the quantified results using the CFS color sensor.

The CFO200 sensor has several possible applications with little or no modification. The most immediate would be in the quantification of color changes and perceptibility of those changes for colorimetric analysis currently in development. Integration of the unit onto unmanned ground vehicles, such as those used to monitor perimeter colorimetric sensors, would remove the noise and variations due to lighting, debris on lenses, and range to target and human perception of images on a monitors with varying degrees of color response.

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ACRONYMS AND ABBREVIATIONS

a*	red–green response
b*	blue–yellow response
CCR	U.S. M8 paper lot no. CCR######-###
CFO200	Micro-Epsilon CFS color-sensor CFO200
CIE	Commission Internationale de l'Éclairage
CSV	comma separated variable
CWA	chemical warfare agent
JID	just intolerable difference
JND	just noticeable difference
L*	luminance
Mk4	Mark IV
RGB	red–green–blue

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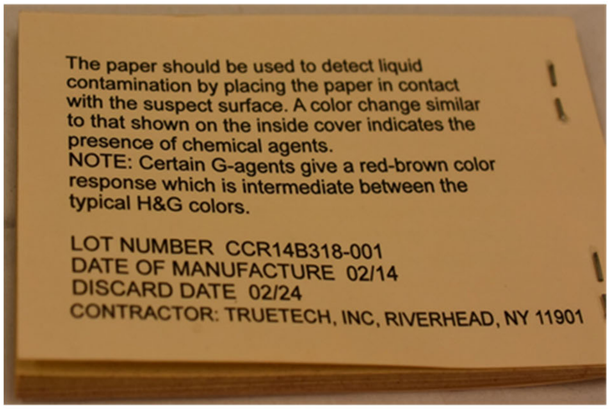
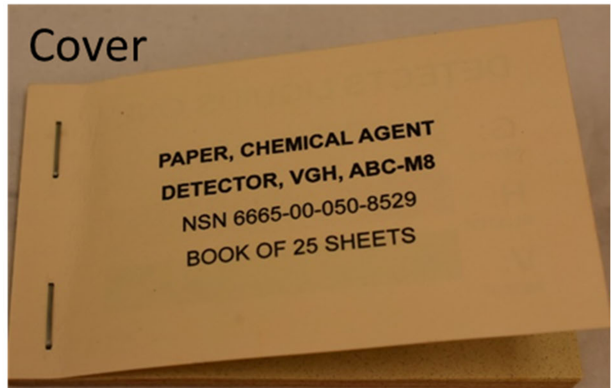
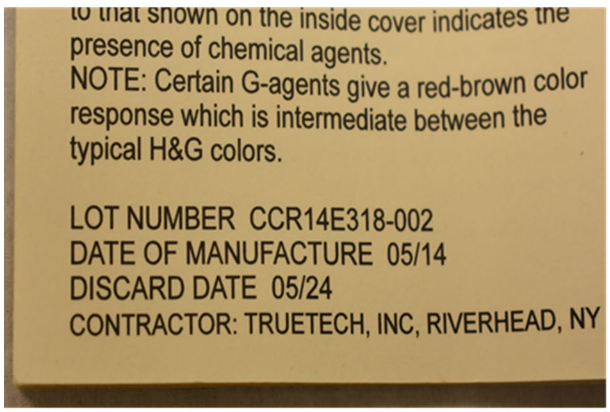
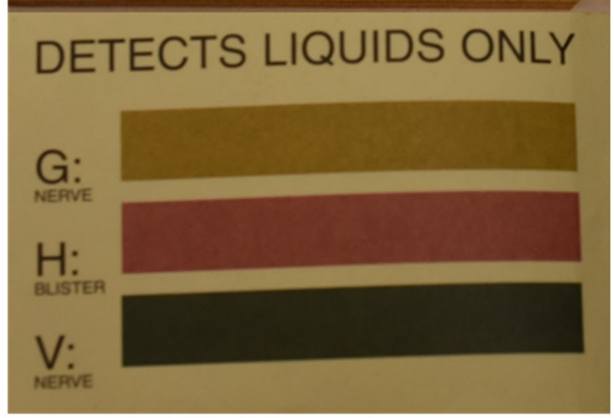
APPENDIX A
IMAGES OF COLOR PAPER BOOKLETS

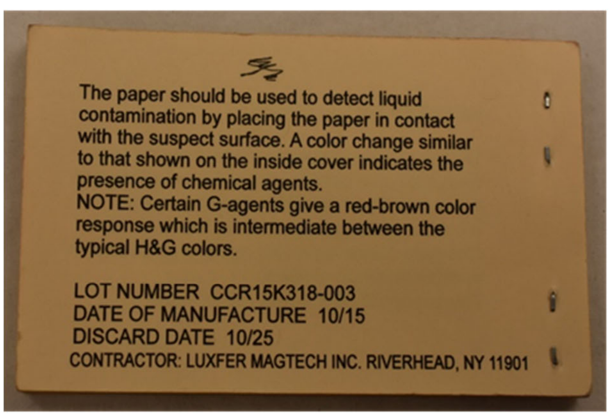
Current U.S. M8 Paper



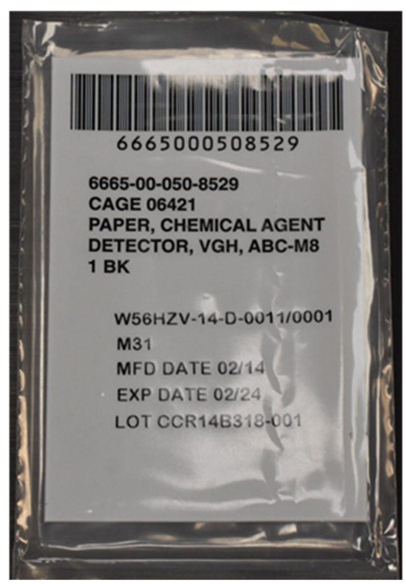




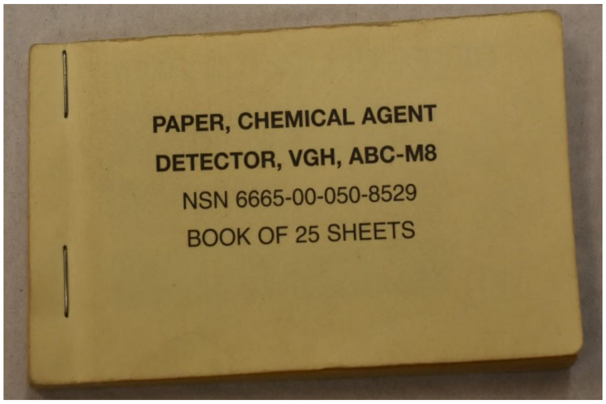


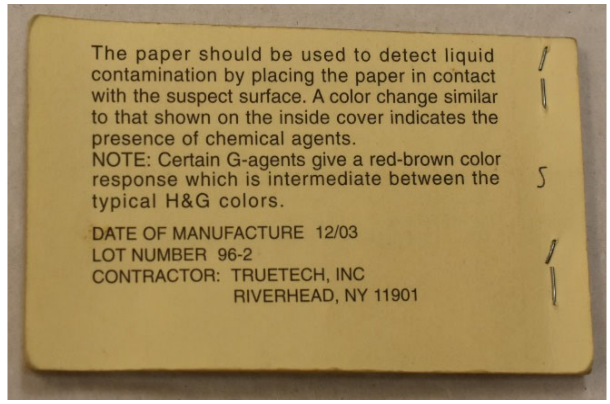


Plastic Packaging (CCR14B318-001)

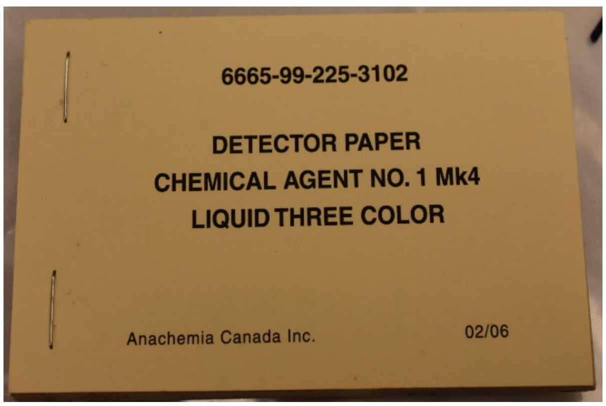


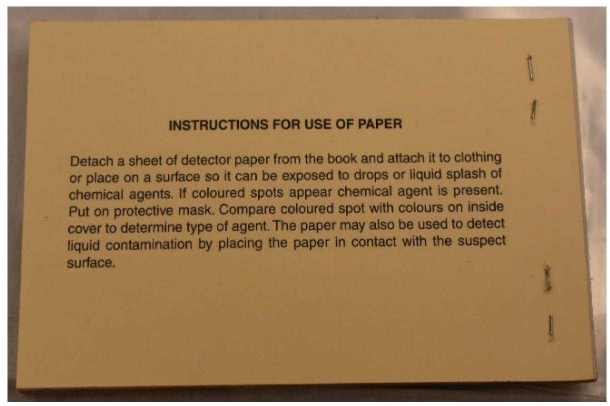
Out of Date U.S. M8 Paper

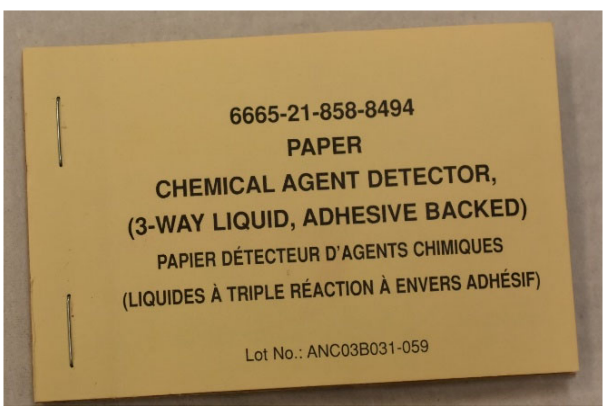


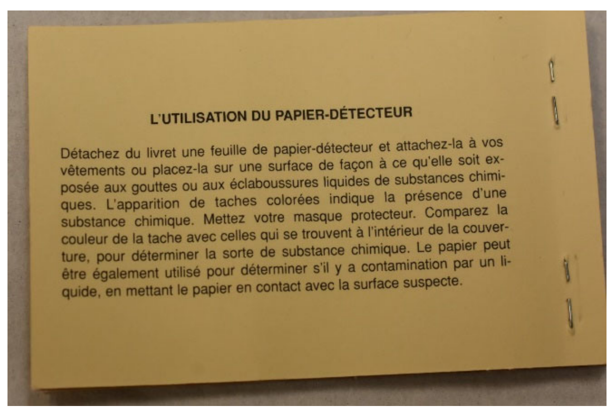


Foreign Paper









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