## ANALYSIS OF ENVIRONMENTALLY FRIENDLY MARKING INK FOR MILITARY EQUIPMENT AND ELECTRONICS

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by Liang C. Li

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#### ABSTRACT

# Analysis of Environmentally Friendly Marking Ink for Military Equipment and Electronics

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Recent advancements in corrosion-resistance coating technology has reduce the use of environmentally harmful compounds such as Hexavalent Chromium by replacing Hexavalent Chromium in primers and topcoat. However, marking inks were neglected in the process. Products such as Enthone 50 series are still widely used, which contain compounds such as lead, hexavalent chromium, bisphenol A (BPA), Cadmium Sulfide, and more. Excluding catalyst compositions in Enthone, the chemicals in the ink alone contained three reproductive toxicant and numerous carcinogens. Therefore, it was essential to search and validate the performance of potential marking ink that would meet the standards in military applications.

Eleven products were tested, and two products were recommended for use: Sherwin Williams MIL-PRF-22750 Type I and Union Ink Uniglaze. Both products contain at most one carcinogen ingredient and no reproductive toxicant. Both products passed MIL-STD 202G Method 215K solvent test, MIL-STD 202G Method 107G thermal shock test, MIL-STD-810G, Method 507.5 humidity test, cleaning system test, ASTM D3359, measure adhesion by tape test.

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#### **Chapter 1 - Introduction**

Traditionally, aerospace coatings have been formulated for performance and corrosion resistance. In the 1950's, compounds such as hexavalent chromium were largely commercialized as a way to enhance the corrosion resistance. [1] Along with hexavalent chromium, other compounds such as lead, cadmium, and phenol solvents were also used in these coating and marking applications. Since then, the basic technology has essentially remained unchanged, while the environmental and toxicological hazardous of hexavalent chromium, cadmium, and lead have become well documented and regulations have eliminated some of these compounds from commercial and residential use; the use of these compounds can still be found in military applications.

In the past decade, there has been a growing interest to replace these compounds. This is partly due to ELV, RoHS, WEEE and REACH legislations in the EU. These regulations imposed international restrictions: ELV, the directive on End-of Life Vehicle 2000/53/EC is the first EU waste directive to prevent the use of certain heavy metals such as cadmium, lead, mercury, and hexavalent chromium for vehicles sold after July 2003. This was officially adopted by the European Parliament and Council in September 2000. The RoHS, Restriction of Hazardous Substances Directive 2002/95/EC, directive took effect on July 1, 2006. The directive restricted the sale of electronic equipment with the use of six substances: lead, mercury, cadmium, hexavalent chromium, polybrominated biphenyls, and polybrominated diphenyl ether from being used in the manufacturing of various types of electronic and electrical equipment. The WEEE, Waste Electrical and

Electronic Equipment, directive set collection, recycling, and recovery targets for all types of electronics. The directive took effective August 13, 2005. REACH, Registration, Evaluation, Authorization and Restriction of Chemicals, is a comprehensive regulation for tracking, testing, and reporting on all chemicals used in the EU that started back in early 2009. While ELV, RoHS and WEEE are exempt from the defense sector, REACH is still effective toward defense sectors with an individual member states in the EU granting substance-specific exemptions for national security reasons. RoHS directive is also well known in the industry to be a temporary exemption according to Journal of Military Electronics and Computing, "Even if military equipment does remain exempt, since commercial component manufacturers and board makers supply both the military and commercial electronics, programs will definitely be affected."

In the United States, regulations on these compounds are largely exempted in the defense industry. However, with increasing international restrictions, stricter regulations will only be a matter of time. In a memorandum from the Under Secretary of Defense to Secretaries of Military Departments on April 8<sup>th</sup>, 2009, regarding minimizing the use of hexavalent chromium stated,

Due to the serious human health and environmental risk related to its use, national and international restrictions and controls are increasing. These restrictions will continue to increase the regulatory burden and life cycle cost for DoD and decrease material availability. OSD, DoD Components, and industry have made substantial investment in finding suitable replacements for hexavalent chromium for many of the current DoD applications.[2]

This memo further directed the DoD military departments to take action by approving the investment in appropriate research, as well as the development on substitutes and the use of alternative where they can perform adequately for the intended application and operating environment. (Please refer to Appendix A for complete memorandum.)

In 2011, a report was presented at the environment, energy security and sustainability (E2S2) symposium, emphasizing the scope of the hexavalent chromium effort of the DoD, Department of Defense. [3] The report stated the current and past hexavalent chromium efforts:

- Low-Cr Conversion Coating
- NC Primer for C-130J OML
- Non-chrome primer C130J IML
- Mg-Rich Treatment
- Non-chrome, Low VOC Fuel Tank Coating (Mil Spec AMS-C-27725)
- Barrier coat for F-16

The current generation of commercial aerospace polyurethane topcoats have an expected service life of approximately 3-5 years. [4] In order to reduce the environmental impact of stripping and repainting aircraft, it is ideal to replace all of the primer, topcoat, and marking ink with formula free of chromium and other harmful agents. Marking ink is indelible ink for marking purposes. In our case, it could be warning sign for tactical

equipment, sub-assembly part labeling, and identification marking ect. While these chromium treatments mentioned above are suitable for primer and coating, it does not suit the purpose of marking ink as described in detail in the literature review session. Marking ink is usually the last step of a work order; with the working conditions and the size of parts, it does not allow users to use theses treatments mentioned above.

In this study, a wide variety of marking inks were investigated for the purpose of replacing marking ink such as the Enthone 50 series ink, which is the most common marking ink used at Raytheon, a major American defense contractor and industrial corporation with core manufacturing concentration in weapons, military, and commercial electronics. Enthone 50 series ink, a two component, epoxy-based screen printing ink, is not compliant with any EU directive mentioned above. Enthone 50 series ink contains harmful substances such as lead, hexavalent chromium, bisphenol A (BPA), Cadmium Sulfide, solvent naphtha, 2-bytoxyethanol, tetraethylenepentamine, Ethylene Glycol Butyl Ether, 2-Ethyl-4methlimidazole and much more. [5-16]

In order to find an alternative ink that "can perform adequately for the intended application and operating environment," stated by the Under Secretary of Defense, a market research was conducted to discover the most suitable and environmentally friendly marking ink available on the market. These samples were exposed to two phases of tests: Phase I, solvent testing, Phase II, more solvent test, cleaning system test, and accelerated life testing including thermal shock test and humidity test. This study will address the effects of various environmental testing of marking ink to find an alternative that can perform adequately with the intended purpose of marking military equipment while reducing the usage of environmentally harmful chemical compounds and solvents.

#### **Chapter 2 - Literature Review**

#### 2.1 Environmental and Health Concerns

#### 2.1.1 Lead

Lead is a naturally occurring element that is toxic to humans of all ages when ingested or inhaled. Lead can be bioaccumlated. While small exposure may not seem harmful, repeated exposure can build over time. In 1977, the Office of Information and Public Affairs issued a final ban on lead-containing household paint, toys, and furniture. [17] Due to the bioaccumulation effect, lead can cause permanent damage to human and marine life. While it is a dangerous compound, most coatings nowadays do not contain lead and it is no longer a main concern.

#### 2.1.2 Cadmium and Hexavalent Chromium

Hexavalent chromium and cadmium compounds are extensively used in the coating to protect industrial ferrous and nonferrous from corroding. Typically cadmium plating is provided as an undercoat to chromate-base primers on steel to achieve a longer service life. [18] Chromates are also used as pigments such as strontium chromate, and zinc chromate. [19]

Both Cadmium and Hexavalent Chromium are carcinogenic and could be fatal if inhaled. [12,14] OSHA, Occupation Safety and Health Administration, estimated that 558,000 workers are potentially exposed to hexavalent chromium annually. They are typically exposed in the following area:

- Welding and "hot work" on stainless steel and other metals containing chromium
- Use of pigments, spray paints and coatings
- Operating chromate plating baths [20]

On October 1, 2004 The *Federal Register* reviewed the Proposed Rule: Occupational Exposure to Hexavalent Chromium; 29 CFR Parts 1910, 1915, 1917, 1918, and 1926. [21] The author illustrated the danger of hexavalent chromium as follow,

Taking a 45-year working life from age 20 to age 65, as OSHA has always done in significant risk determinations for previous standards, the Agency finds an excess lung cancer risk of approximately 100 to 350 per 1000 workers exposed at the previous PEL of 52 [mu]g/m3 Cr(VI). This risk is clearly significant, falling well above the level of risk the Supreme Court indicated a reasonable person might consider acceptable. Even assuming only a 20-year working life, the excess risk of about 50 to 200 per 1000workers is still clearly significant. The new PEL of 5 [mu]g/m3 Cr(VI) is expected to reduce these risks substantially, to below 50 excess lung cancers per 1000 workers. However, even at the new PEL, the risk posed to workers with a lifetime of regular exposure is still clearly significant. [22]

#### Table 1- Selected OSHA Risk Estimates (Excess Cancers per 1000 Workers) [22]

Standard	Risk at prior PEL	Risk at new PEL	Federal Register date
Ethylene Oxide	63 - 109 per 1000	1.2 - 2.3 per 1000	June 22, 1984
Asbestos	64 per 1000	6.7 per 1000	June 20, 1986
Benzene	95 per 1000	10 per 1000	September 11, 1987
Formaldehyde	0.43 - 18.9 per 1000*	.0056 - 2.64 per 1000*	December 4, 1987
Methylenedianiline	6 - 30 per 1000**	0.8 per 1000	August 10, 1992
Cadmium	58 - 157 per 1000	3 - 15 per 1000	September 14, 1992
1,3-Butadiene	11.2 - 59.4 per 1000	1.3 - 8.1 per 1000	November 4, 1996
Methylene Chloride	126 per 1000	3.6 per 1000	January 10, 1997
Chromium VI	101 - 351 per 1000	10 - 45 per 1000	2006
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\* range is based on maximum likelihood estimate (0.43, .0056) and upper 95% confidence limit (18.9, 2.64)
 \*\* no prior standard; reported risk is based on estimated exposures at the time of the rulemaking

The new 5  $\mu$ g/m<sup>3</sup> chromium regulation, however, does not apply to aerospace industry. OSHA permissible exposure limit (U.S.) of Hexavalent Chromium in the aerospace industry was only reduced to 25  $\mu$ g/m<sup>3</sup> of airborne chromium, calculated as an 8-hour time-weighted average instead of 5  $\mu$ g/m<sup>3</sup> as shown in Table 1. If the-average of the prior and current risk are taken, assuming linear relationship, the new aerospace industry cancer risk at the new PEL will yield 50-197 per 1000 workers. The calculation is shown below. Followed by cadmium and benzene, which are also common among aerospace painting operations. [22]

Average cancer rate per 1000 people per  $\frac{\mu g}{m3}$  at lower extreme =  $\frac{rate \ of \ death \ at \ 52 \ \frac{\mu g}{m3} + rate \ of \ death \ at \ 5 \ \frac{\mu g}{m3}}{2} = \frac{\frac{101}{52} + \frac{10}{5}}{2}$ = 1.97

*New Aerospace lower bound Risk* =  $(1.97)(25 \frac{\mu g}{m_3})$ = 50 per 1000 workers

Average cancer rate per 1000 people per 
$$\frac{\mu g}{m3}$$
 at upper extreme  
=  $\frac{\text{rate of death at 52 } \frac{\mu g}{m3} + \text{rate of death at 5 } \frac{\mu g}{m3}}{2} = \frac{\frac{351}{52} + \frac{45}{5}}{2}$   
= 7.875

*New Aerospace upper bound Risk* =  $(7.875)(25\frac{\mu g}{m_3}) = 197$  per 1000 workers

#### 2.1.3 Current Ink

The most common marking ink used at Raytheon is Enthone 50 series. Enthone 50 series is a two-part, single stage polyurethane stencil ink. "Two-part" indicates the required mixing of catalyst and ink prior to application and "single stage", indicates that it only requires one layer of application. It is best for marking ink to be single stage, as it'll reduce curing time and complexity of marking a product. Marking ink is typically applied over a topcoat as displayed in Figure 1, or a corrosion resistance coating.

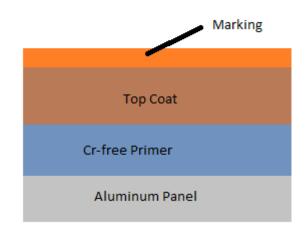


Figure 1- Layer of coating in aerospace applications.

Ideally, the Cr-free topcoat and Cr-free primer would be adequate for the aluminum panels to be resistant to corrosion as marking ink isn't applied to all parts of the equipment. However, this doesn't make the marking ink, such as Enthone 50 series, safe in anyway, as shown in Table 2. There are numerous carcinogenic components, and three of the components may even cause genetic mutations.

<u>Chemical</u>	<u>Toxicological</u> Information	Carcinogenic	Environmental Information
Lead	Acute toxicity, Suspected human reproductive toxicant	IARC, International Agency for Research on Cancer, <u>Group 2B:</u> <u>Possibly</u> <u>carcinogenic to</u> <u>humans</u>	Toxicity to fish, daphnia, algae, and other aquatic invertebrates. [15]
<u>Hexavalent Chromium</u>	Toxic if swallowed or in contact with skin. Fatal if inhaled. Suspected human reproductive toxicant. May cause reproductive disorder.	IARC Group 1 – Carcinogenic to humans(Chromium trioxide)	Toxicity to fish, daphnia, and other aquatic invertebrates. Long lasting effects[14]
<u>Bisphenol A</u>	Acute toxicity. Overexposure may cause reproductive disorder(s) based on tests with laboratory animals.	No component of this product present at levels greater than or equal to 0.1% is identified as probable, possible or confirmed human carcinogen by IARC	Toxicity to fish, daphnia, algae, and other aquatic invertebrates. [13]
<u>Cadmium Sulfide</u> <u>2-bytoxyethanol</u>	Toxic if swallowed. Fatal if inhaled. May alter genetic material. Toxic if swallowed.	IARC Group 1 –         Carcinogenic to         humans(Benzene)         No component of         this are dust areas	Toxicity to fish, daphnia and other aquatic invertebrates[12] Toxicity to fish, daphnia and
<u>Present in Catalyst 9</u>	Fatal if inhaled. May cause reproductive disorders. May cause congenital malformation in the fetus.	this product present at levels greater than or equal to 0.1% is identified as probable, possible or confirmed human carcinogen by IARC	<u>daphnia and</u> <u>other aquatic</u> <u>invertebrates</u> [11]
<u>Naphtha</u>	<u>No data</u>	IARC Group 1 – Carcinogenic to humans(Benzene)	<u>No data[10]</u>
<u>Tetraethylenepentamine</u> <u>Present in Catalyst 9</u>	Acute toxicity	No component of this product present	<u>Toxicity to fish,</u> daphnia, algae,

Table 2 - Harmful Chemical ingredients of Enthone 50 Series. [5-16]

		at levels greater than or equal to 0.1% is identified as probable, possible or confirmed human carcinogen by IARC	and other aquatic invertebrates. Long lasting effects. [9]
<u>2-Ethyl-</u> <u>4methlimidazole</u> <u>Present in Catalyst 5</u>	<u>Acute toxicity</u>	No component of this product present at levels greater than or equal to 0.1% is identified as probable, possible or confirmed human carcinogen by IARC	Toxicity to fish[8]
<u>Ethylene Glycol Butyl</u> <u>Ether</u> <u>Present in Catalyst 5</u>	Acute toxicity Overexposure may cause reproductive disorder(s) based on tests with laboratory animals.	IARC Group 3: Not classifiable as to its carcinogenicity to humans	Toxicity to fish, daphnia and other aquatic invertebrates[16]

#### 2.2 New Solutions

New developments in the coating industry have led to more environmentally friendly coating agents. [3] In the following section, each category of advancement will be examined and evaluated for criteria of suitable marking ink purposes.

#### 2.2.1 Low-Cr Conversion Coating

Alternatives to hexavalent conversation coating have existed since the 1970s, when they were based on trivalent chromium compounds, and had been limited primarily to lower performing coatings. [1] In 2013, a study was performed to test the corrosion resistance and the electrical contact resistance (ECR) of a new generation of 'true' chromium free

conversion coatings as shown in Table 3. ECR was tested, as it is an integral part of the resistance of the overall circuit of a device. If ECR is significantly smaller than the total resistance of the circuit, it can impair the performance of a wide range of electric devices. A salt spray test (SST) was performed according to ASTM B117. The test was performed on AA2014-T3 and AA6082-T6 aluminum alloy with six different treatments of low-Cr or Cr-free conversion coating. The results were that, "among the 'true' chromium free treatments considered in this work, none can be considered a substitute for standard chromate process because requirements of both the SST and ECR test have not been satisfied". [23] While requirements for SST were satisfied, the coating couldn't be used due to ECR. This result indicates the new generation of conversion coating is only suitable for certain types of coatings that will not have contact with electronics.

 Table 3 - Different treatments for alloys AA6082-T6 and AA2014-T3 with 6 available

 treatment. [23]

Different treatments for alloys AA2014-T3 and AA6082-T6				
Treatments	AA2014-T3	AA6082-T6		
Treatment 1: Cr III	Х	X		
Treatment 2: Talc	Х	Х		
Treatment 3: Zr	Х	Х		
Treatment 4: Talc + Ce	-	Х		
Treatment 5: Talc +KMO <sub>4</sub>	Х	-		
Treatment 6: NaOH + HNO <sub>3</sub> + Talc + Ce	Х	-		

Aside from the results, the coating process is highly unlikely to be used in a marking application due to the treatment process, which requires samples to be submerged in above room temperature, and pH as high as 11.5. These processes could damage the parts for marking. [23]

#### 2.2.2 Non-chrome Primer for C-130J OML & Non-chrome primer – C130J IML

Non-chrome, primer for C-130J OML include both water-borne and solvent-borne nonchrome primer for C-130J Outer Mold Line and Non-chrome, primer for C-130J IML chrome primer for C-130J Inner Mold Line was developed by Lockheed Martin Aeronautics Company, and funded by Lockheed and US Air Force, ASC.

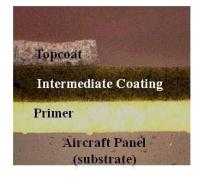
Primer for C-130J OML's performance actually exceeded chromate primers in a 3000 hour salt spray test. During the qualification test, the candidates finished as well as the baseline finish. There was no discoloration, chalking, thickness changes, adhesion loss or corrosion observed. [24] This product seemed to be an ideal candidate. However, because it is a primer it required a topcoat, making this product a duel stage product.

#### 2.2.3 Mg-Rich Treatment

Mg-rich treatment is a primer capable of sacrificial protection. Due to Aluminum's low position in the galvanic series, it's limited to anodic metals. [25] This work is capable of protection of high strength aircraft Al alloy such as 2024 T-3 and 7075 T-6 without the use of Chromium. However, these primer coatings need to be top-coated in order to function properly and have a long field life. [26]

#### 2.2.4 Non-chrome, Low VOC Fuel Tank Coating (Mil Spec AMS-C-27725)

Mil Spec AMS-C-27725 is a polyurethane coating developed in 1969. It includes two classes, Class A and Class B. Class A is for general use in areas where air pollution regulation does not exist. Class B is for limited use. In today's environment, these products are obsolete. [27]



#### 2.2.5 Barrier coat for F-16

*Figure 2 - Barrier coat encapsulating chrome primer. [3]* 

As seen from Figure 2, Barrier coat's purpose is to encapsulate chrome primer.

Therefore, it would not be suitable for marking purposes.

#### 2.3 Adhesion of Marking Ink

#### 2.3.1 Reliability of Marking Ink

Reliability of marking ink depends on several factors, and any combination of these factors could attribute to failure. [28]

- 1. Poor or inadequate surface preparation and application of the paint to the substrate
- 2. Atmospheric effects
- 3. Structural defects in a paint film
- 4. Stresses between the bond and the substrate
- 5. Corrosion

Atmospheric effects, structural defects, stresses between bond and the substrate, are largely dependent on the structure and the operating environment, while corrosion depends on the chemical composition of the coating layers.

#### 2.3.2 Surface Preparation and Adhesion

The surface preparation of the panels is critical in the adhesion of the marking ink. The consequences of poor surface preparation will yield results such as peeling, flaking, and delamination. The surface treatment needs to remove oils, dirt, grime, waxes, and loose particulates. In addition, surface treatment methods could also improve the adhesion

properties by increasing surface roughness and surface energy, and reducing other factors that could trap unwanted particles between the substrate and the coating.

The adhesion of a coating is improved when a clean panel contains pores, holes, or crevices. The roughness of the surface creates a mechanical anchor on the panel surface. Thus, making the removal of the coating more difficult as shown in Figure 3.

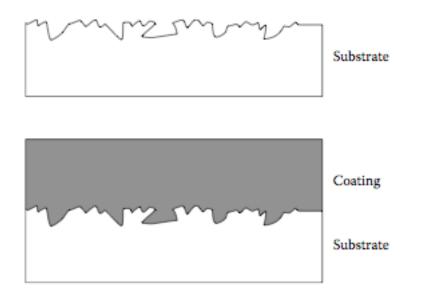


Figure 3 - Before and after image of paint coat anchoring to the substrate. [28]

Increasing the surface roughness could increase the surface area, as well as the bonding area by five times. [28] However, the effects of surface roughness are only possible if the coating can penetrate completely into all the surface irregularities. If compete penetration is not achieved, then there is less coating-to-interface contact. The typical method to increase surface area on a panel is to use either wet or dry sanding with grit of 40-400. [28] The grit size depends on the surface finish desired. Following the surface treatment, the panel must be removed of residual dust or grit and then treated with either detergent cleaning or solvent cleaning to remove the surface contamination by sanding. By sanding, it is also possible to get rid of the corrosion of the substrate. However, this does not apply to our case, due to the polymer coating that is already in place prior to the use of marking ink.

There are several other surface treatments that could improve the adhesion of paints and coatings:

- 1. Mechanical Treatments.
- 2. Chemical Treatments
  - i. Sulfuric Acid-Dichromate Etch
  - ii. Sodium Etch
  - iii. Sodium Hydroxide
  - iv. Sanitizing
  - v. Phenol
  - vi. Sodium Hypochlorite
- 3. Plasma Treatment
- 4. Flame Treatment
- 5. Corona Discharge
- 6. Primer [28]

Most of these treatments are unsuitable for marking ink, simply because most product are at the end of its production stage when it is being marked, and it is unrealistic to perform these treatments that could damage the parts.

In addition to surface treatments, surface contamination is the major concern in the adhesion of the coatings. The easiest and most common procedure is to remove surface contamination by organic solvents such as acetone, methyl ethyl ketone, toluol, 1,1,1,- trichloroethane, naphtha, and on occasion Freon. [28] The property of the organic solvents will remove process oils, dirt, grime, waxes, and other particulates left behind by sanding. These solvents are applied by simply wiping with a clean cotton pad in a swirling motion on the panel to remove the undesired contaminates.

#### **Chapter 3 - Materials and Methods**

#### 3.1 Product Selection

In order to evaluate alternatives to replace products such as Enthone, 17 companies were contacted to provide information about their eco-friendly product lines. The companies selected represented a diverse group ranging from small to large companies and included Domino and Leibinger from Europe.

- Akzonobel
- DuPont
- Nazdar
- Markem Imaje
- American Marking
- Sun Chemical
- DEFT
- JanTech
- Jetec

- Marco Ink
- Sherwin Williams
- Domino
- Leibinger
- Nanotech
- Independent Ink
- Union Ink
- Go Green World Products

In total, 11 product lines were selected for testing.

- AkzoNobel
  - 1. Aerofine a one compound low VOC, isocyanate free, waterborne topcoat.
  - 2. Spray2Fix (Intergard 10301SC) High solids epoxy primer.
- DuPont
  - 3. Industrial Strength Ultra low VOC polyurethane enamel high gloss topcoat.
  - 4. Imron 1.2 HG High gloss waterborne polyurethane copolymer topcoat.

- DEFT
  - 5. MIL-PRF-85285 two compound polyurethane topcoat intended for use on exterior application on aircraft and aerospace equipment.
- American Marking
  - 6. JS series RoHS compliant solvent-based ink. (Acetone based)
  - 7. WJ series water-based pigmented environmentally-friend spray ink.
- Sherwin Williams
  - KEM AQUA-BP Enamel One component low HAPS and low VOC water reducible enamel.
  - 9. MIL-PRF 22750G Type I 2.7 VOC compliant high solids two component epoxy topcoat. Intended for use as a top coat of interior ground equipment.
- Union Ink
  - Uniglaze Two compound epoxy ink intended for application such as printed circuit board markings and electronic equipment panels.
- Go Green World Products
  - 11. Green Polyurethane Isocyanine polyurethane paint.

These product lines were selected due to their relative eco-friendly compositions. Of 11 products, there were 4 water-based products, 1 acetone-based product, 4 polyurethane products, and 2 Epoxy products. Green Polyurethane and Aerofine were also iscyanate-free, a compound attributed to the cause of asthma, which is rarely found in the industry. Of the 11 products, 2 were already being used in military applications and were selected due to their relative eco-friendliness. The Uniglaze product line was being used by part of Raytheon, and therefore served as a control.

#### 3.2 Methods

#### 3.2.1 Evaluation of Metal Panels

Metal panels were evaluated for cleanliness prior to testing as shown in Figure 4. A qualitative water break test, ASTM F22, a procedure approved by the Department of Defense, was performed to determine if the surface contained any contaminates. This test method detects the presence of hydrophobic (non-wetting) films on surfaces and the presence of hydrophobic organic materials in process ambient. [29] A steady stream of water was applied across the panel at a 45° angle. Panels were judged clean if the film of water either did not break up or take a minute to do so. This is because water will bead up when it comes in contact with hydrophobic surface contaminates. Acetone was used to clean contaminated panels. This method is sufficient to provide a clean surface, but is not as thorough as using a batch or vapor degreaser cleaning system. However, batch or vapor degreaser cleaning systems are unpractical due to heat and moisture exposure to electrical systems and the sheer size of assembled tactical equipment.



*Figure 4 – Water-break test, Left - contaminated panel, center- clean panel with acetone, right - clean panel.* 

### 3.2.2 Painting Methodology and Curing Schedule

Clean panels were painted using the following steps:

Paint Instructions:

- 1. Clean panels with Acetone for at least 3 minutes.
- 2. Apply water onto the panel to confirm for surface cleanliness.
- 3. Arrange desired amount of stencils on the panel.
- 4. Use blue tape to secure the left and right sides of the stencils.
- 5. Use blue tape along with painter's masking paper to cover unused stencils.
- 6. Airbrush the stencils two inches away from the panel and apply at a steady pace for 2 passes (20-30 seconds). Dwelling time of the airbrush on the stencils vary with different paints due to varying viscosities. Dwell times were determined prior to painting the samples to produce a consistent thickness.
- 7. Pour away the unused paint and remove excess paint.
- 8. Pour airbrush cleaner and run airbrush for 3 minutes.

- 9. Disassemble airbrush and clean with water.
- 10. Remove all masking paper once all paint has been applied.
- 11. Force cure the panels at 70 °C for 60 minutes.

Curing schedule of different materials varied due to varying composition (Appendix D). Insufficient curing times resulted in improper samples. A longer curing time does not adversely affect the paint. Epoxy paint has the longest average cure time (Appendix D); therefore, 70 °C for 60 minutes was selected as the cure time.

It was observed that Green Polyurethane could not be applied using the airbrush due to its high viscosity. As a result, Green Polyurethane was applied via brushing. Due to the small size of the stencil, the numbers were deformed and the shape of the numbers were not as defined as other samples.

#### **3.3** Design of Experiment

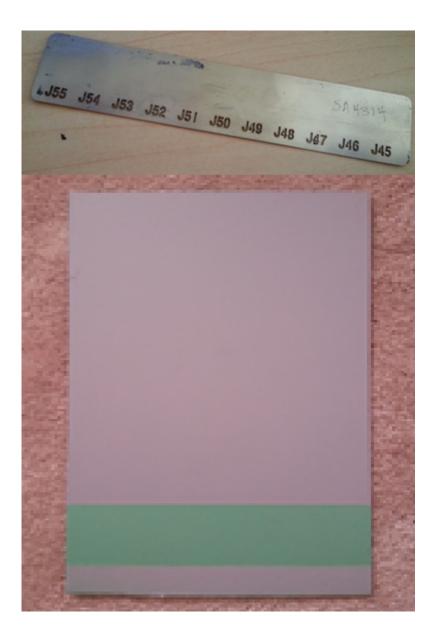
The selected marking inks were evaluated using the following performance standards:

- 1. Solvent resistivity.
- 2. Moisture resistivity
- 3. Discoloration due to high temperature.
- 4. Temperature related failures.
- 5. Cleaning system related failures.
- 6. Military requirements of marking ink. [30]

Additionally, it would be beneficial for the implementation of these marking inks if the marking ink could validate the various MIL-STD parameters recommended by the Department of Defense. Therefore, most of these tests were derived from the recommendations of many military standards.

Samples marking inks were sprayed on both sides, the back was M5541 T1 Class3 Chem-Filmed aluminum and the front was painted aluminum using M23377 T2 Class N primer with M22750 or M85285 topcoats as shown in Figure 5. The samples were applied using an airbrush and an 11-point font stencil provided by Raytheon. Each stencil included 11 samples, and each sample consisted of one letter and a number that ranged from 1 to 55. Each time the paint samples were applied, it would be covered with masking paper and scotch blue#2093EL as shown in Figure 6.

In the following test, 6 panels were painted front and back, yielding 66 samples on M5541 T1 Class3 Chem-Filmed aluminum and 66 samples on painted aluminum using M23377 T2 Class N primer with M22750 or M85285 topcoats.



*Figure 5 – Top, 11-point font stencil provided by Raytheon, Bottom, M23377 T2 Class N primer with M22750 or M85285 topcoats* 



*Figure 6 – Top, masking paper and scotch blue#2093EL. Bottom, stencil covered by masking paper and scotch blue#2093EL on Al panel.* 

Testing consisted of two phases. Phase I determined if the material could pass a typical polar-nonpolar solvent resistivity test described in MIL-STD202G method 215K. Phase II, consisted of further solvent testing, a humidity test, a thermal shock test, and a cleaning system test. These tests were designed to test the performance criteria of the ink.

#### 3.3.1 Solvent Resistivity:

During phase I of testing, MIL-STD 202G, Method 215K was used to reduce the possible candidates. MIL-STD 202G, Method 215K verifies that marking and color will not become illegible or discolored when subjected to solvent and processes normally used to clean electronics. It also verifies that the component protective coatings and encapsulation materials are not degraded to the point where electrical or mechanical integrity is disturbed when subjected to solvents and processes normally used to clean solder flux, finger prints, and other contaminants from printed-wiring and terminal-board assemblies.

The original MIL-STD 202G included four solvent tests. Solvent II was removed from the current MIL-STD, leaving three solvent tests in the MIL-STD. Solvent tests I and III had the same procedures, while Solvent IV had a slightly different procedure.

Solvent I included 1 part isopropyl alcohol and 3 parts mineral spirits by volume. Solvent III was an over-the-counter commercial terpene defluxer. Isopropyl alcohol, mineral spirit, and terpene defluxer were bought off –the-shelf. Solvent IV was mixed in the lab with 1 part propylene glycol monomethyl ether (PGME), 1 part monoethanolamine (MEA) by volume, and 42 parts water by volume.

Solvent I was maintained at a temperature of  $25^{\circ}C \pm 5^{\circ}C$ .

- 1. The specimens will be completely immersed for 2.5-3.5 minutes in the specified solution.
- 2. The bristle portion of the brush was dipped in the solution until wetted and the specimen shall be brushed with normal hand pressure (approximately 2 to 3 ounce force applied normal to the surface) for ten strokes on the portion of the specimen where marking has been applied.
- 3. The brush stroke shall be directed in a forward direction across the surface of the specimen being tested. Immediately after brushing, the procedure shall be repeated two more times, for a total of three immersions.
- 4. After completion of the third immersion and brushing, the specimens shall be airblown dry.

Solvent III was maintained at a temperature of  $25^{\circ}C \pm 5^{\circ}C$ .

- 1. The specimens were completely immersed for 2.5-3.5 minutes in the specified solution.
- 2. The bristle portion of the brush was dipped in the solution until wetted and the specimen was brushed with normal hand pressure (approximately 2 to 3 ounce force applied normal to the surface) for ten strokes on the portion of the specimen where marking had been applied.
- The brush stroke was directed in a forward direction across the surface of the specimen being tested. Immediately after brushing, the procedures were repeated two more times, for a total of three immersions.
- 4. After the completion of the third immersion and brushing, the specimens were rinsed in approximately 25°C water and all surfaces were air-blown dry.

Solvent IV was maintained at a temperature of 63°C to 70°C as shown in Figure 7.

1. The specimens were be completely immersed for 2.5-3.5 minutes in the specified solution.

- The bristle portion of the brush was dipped in the solution until wetted and the specimen was brushed with normal hand pressure (approximately 2 to 3 ounces of force was applied normally to the surface) for ten strokes on the portion of the specimen where marking had been applied.
- 3. The brush stroke was directed in a forward direction across the surface of the specimen being tested. Immediately after brushing, the procedure was repeated two more times, for a total of three immersions.
- 4. After completion of the third immersion and brushing, the specimens were rinsed in approximately 25°C water and all surfaces were air-blown dry.

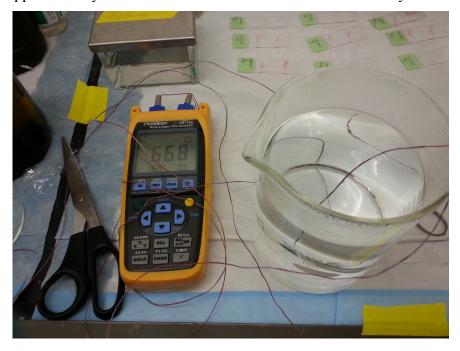


Figure 7 - Solution IV maintained at temperature of 63°C to 70°C

After the test, the panels were examined from a distance of at least six inches with normal lighting without the aid of magnification. This was done to ensure that there no panels were entirely or partially missing, faded, smeared, blurred, or shifted to the extent that they could not be readily identified.

In phase II, additional solvent testing included jet fuel, hydraulic fluid, and ethylene glycol coolant/de-icing fluids. These solvents are common solvents found in the

aerospace industry and are commonly presented in the environment of tactical equipment. In phase II, the procedures used were the same as Solvent I due to the common operating temperature and that the exposure these additional solvents were not washed off with water.

## **3.3.2** Thermal Shock & Color Discoloration Test

The purpose of this test is to determine the resistance of a part to temperatures at high and low extremes. These conditions may be encountered in equipment operated continuously in low temperature areas and high temperatures areas. Permanent changes in operating characteristics and physical damage produced in thermal shock, principally results from variation in dimension and other physical properties.

In thermal shock, we used MIL-STD 202G, Method 107G to simulate the closest operating conditions of tactical equipment. Two extremes were considered: the coldest part of the atmosphere, the area between the stratosphere and troposphere, tropopause, which could reach as cold at -57 °C, and the upper bound, which was chosen as 125°C due to operation conditions due to the heat produced by the engine and the environment. In testing, a recovery time of 5 minutes, a dwell time of 30 minutes in each of the chambers were established according to the weight of our panels, while the testing profile was set to profile B due to the two temperature extremes as shown in Table 4 & 5.

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Ten cycles were run and results were based upon the change in color of each specimen to

ensure there were no discolorations due to high temperature.

Weight of Specimen	Minimum time in chamber (hours)
1 ounce	1/4
Above 1 ounce to .3 pounds(28g to 136g), inclusive	1/2
.3 pounds to 3 pounds (136g to 1.36kg), inclusive	1
3 pounds to 30 pounds (1.36 kg to 13.6kg), inclusive	2
30 pounds to 300 pounds (13.6 kg to 136kg), inclusive	4
Above 300 pounds (above 136kg)	8

*Table 4 – MIL-STD-202G, Method 107G Exposure time in air at temperature extremes.* 

Table 5 - MIL-STD-202G, Method	107G Thermal shock test conditions in air
environment.	

Step	Test	Number	Test	Number	Test	Number of
	Condition	of cycles	Condition	of cycles	Condition	cycles
	А	5	В	5	C	5
	A-1	25	B-1	25	C-1	25
	A-2	50	B-2	50	C-2	50
	A-3	100	B-3	100	C-3	100
	Temp (°C)		Temp (°C)		Temp (°C)	
1	-50 +0,-3	see table 4	-65 +0,-5	see table 4	-65 +0,-5	see table 4
2	25 +10,-5	5 minute maximum	25 +10,-5	5 minute maximum	25 +10,-5	5 minute maximum
3	85 +3,-0	see table 4	125 +3,-0	see table 4	200 +5,-0	see table 4
4	25 +10,-5	5 minute maximum	25 +10,-5	5 minute maximum	25 +10,-5	5 minute maximum
Step	Test	Number	Test	Number	Test	Number of
	Condition	of cycles	Condition	of cycles	Condition	cycles
	D	5	E	5	F	5
	D-1	25	E-1	25	F-1	25
	D-2	50	E-2	50	F-2	50
	D-3	100	E-3	100	F-3	100
	Temp (°C)		Temp (°C)		Temp (°C)	
1	-50 +0,-3	see table 4	-65 +0,-5	see table 4	-65 +0,-5	see table 4
2	25 +10,-5	5 minute maximum	25 +10,-5	5 minute maximum	25 +10,-5	5 minute maximum
3	350+5,-0	see table 4	500 +5,-0	see table 4	150 +3,-0	see table 4
4	25 +10,-5	5 minute maximum	25 +10,-5	5 minute maximum	25 +10,-5	5 minute maximum

#### 3.3.3 Humidity Test

Humidity test was perform to ensure the resistance of material to the effects of a warm and humid atmosphere. Due to the vastly different operating environments of tactical equipment, humidity resistance must be consider. Warm, humid conditions can occur year-round in tropical areas, seasonally, in mid-latitude areas in combination to changes in temperature and relative humidity. These Humidity test in DoD typically follows environmental engineering considerations and laboratory tests standards, MIL-STD-810G, Method 507.5, Humidity testing. In general there are two types of humidity testing used in the Department of Defense: induced and aggravated.

"Induced" testing refers to storage and transit, and the typical test includes three unique cycles that may occur during storage or transit. Exposure to these temperatures showed drastically "lower" results when compared with the aggravated test.

Aggravated testing exposed samples to more extreme temperatures and humidity to simulate the worst-case scenario environment. Purpose of aggravated test procedure is to produce a representative effect that occurs when material is exposed to elevated temperature-humidity conditions. These cycles typically don't occur in nature or normal operating parameter. Thus, a failure in the test doesn't necessarily indicate failure in the real environment.

In our test, ten cycles were run in the temperature-humidity profile in Figure 8, an

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aggravated test. Each cycle indicates a full 24 hours. The relative humidity was kept at 95 % and the temperature varied from 30°C to 60°C.

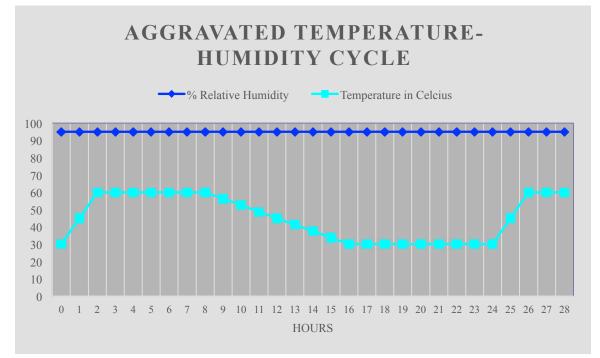


Figure 8- Temperature/Humidity Profile of MIL-STD-810G, Method 507.5G

Procedure for Humidity Test:

Step 1. With the test item installed in the test chamber in its required configuration, adjust the temperature to  $23 \pm 2^{\circ}C$  ( $73 \pm 4^{\circ}F$ ) and  $50 \pm 5$  percent RH, and maintain for no less than 24 hours.

Step 2. Adjust the chamber temperature to 30°C (86°F) and the RH to 95 percent.

Step 3. Expose the test item(s) to at least ten 24-hour cycles ranging from 30-60°C (86-

140°F). Unless otherwise specified in the test plan, conduct a test item operational check

(for the minimum time required to verify performance) near the end of the fifth and tenth cycles.

Step 4. At the completion of 10 or more successful cycles, adjust the temperature and humidity to  $23 \pm 2^{\circ}C$  ( $73 \pm 4^{\circ}F$ ) and  $50 \pm 5$  percent RH, and maintain until the test item has reached temperature stabilization (generally not more than 24-hours).

Step 5. Perform a thorough visual examination of the test item, and document any conditions resulting from test exposure.

Step 6. Conduct a complete operational checkout of the test item and document the results. See paragraph 5 for analysis of results.

Step 7. Compare these data with the pretest data

#### **3.3.4** Cleaning System Test

Aside from accelerated environmental testing, the test subject must also be able to withstand typical cleaning systems used in military facilities. In our testing, panels were sent to military facilities provided by Raytheon to be tested. Testing include:

- Branson B-series Degreaser with ultrasonic using Asahiklin AK-225T solvent.
- Aqueous Technologies Trident batch cleaning system using Kyzen A4615

Asahiklink AK-225T solvent is a vapor degreaser that's designed to replace chlorofluorcarbon, perfluorcarbon, and other hydrochloroflurocarbon. It's ideal for general and precision cleaning.

Kyzen A4615 is a MEA-free aqueous blend specially designed for electronics processes for removal of some lead-free flux, tacky flux, misprinted paste, no clean flux, RMA flux, OA paste, oils, finger prints, light oxides. Samples were run 3 times in each system to simulate worst-case conditions before parts were reworked and run through more than once.

# 3.3.5 Military Marking Ink Requirement

MIL-STD-130N, Department of Defense standard practice identification marking of U.S. military property determines the clarification, insight, guidance, and marking criteria regarding implementation of machine-readable information (MRI) for identification marking of U.S. military property and automatic data capture. This MIL standard provides the criteria by which product designers develop specific identification marking requirements for items: the marking content, size, location, application processes, and any product definition data that the marking be in accordance with this standard. For our purposes, the primary interest of MIL-STD-130N is if the samples are capable of producing minimum character heights of 6 pts or .08-inch height fonts.

#### 3.3.6 ASTM D3359 Tape Test

In addition to accelerated life testing, the samples were also tested by tape test described in ASTM Designation: D3359 – 97, a common test intended to test for paint adhesion. Due to the size of our samples, Test Method A was used, "An X-cut is made in the film to the substrate, pressure-sensitive tape is applied over the cut and then removed, and adhesion is assessed qualitatively on the 0A-5A scale in the analysis of the paint." The apparatus used was razor blade and one-inch wide pressure-sensitive tape with an adhesion strength agreed upon by Raytheon. The adhesion in accordance with the following scale:

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- 5A No peeling or removal
- 4A Trace peeling or removal along incisions or at their intersection
- 3A jagged removal along incisions up to 1/16 in. on either side
- 2A jagged removal along most of incision up to 1/8in. on either side
- 1A removal from most of the area of the X under the tape
- 0A removal beyond the area of the X.[31]

#### Chapter 4 – Results and Analysis

#### 4.1 Phase 1 Test Result

In phase 1, painted panels were exposed to solvents in MIL-STD 202G method 215k and the procedures can be summarized in Table 6.

**Testing Procedure** Solvent I: Solvent II: Solvent III: Alcohol Terpene PGME, defluxer and MEA, Mineral Water Spirit **1.** Maintain solution temperature (°C) 20-30 63-70 20-30 2. Immerse sample for 3 minutes Х Х Х 3. Brush with normal hand pressure (ten Х Х Х strokes) 4. Repeat twice Х Х Х Х 5. Rinse in 25(°C) water Х Х Х Х 6. Air dry

Table 6 - Summary of testing procedure of MIL-STD 202G Method 215K

11 product lines were tested, all of which were painted with red and black, except for DuPont 1.2 HG and Green Polyurethane. DuPont 1.2 HG and Green Polyurethane was tested in white due to the delay and out of stock.

The panels were examined from a distance of at least 6 inches with normal lighting and without the aid of magnification for partially of fully missing plates, faded, smeared, blurred, or shifted to the extent that they cannot be readily identified. Of the 11 products tested, 5 passed all 3 solvent tests. The results were very definite, due to the way the paints failed in Table 7. The results were also analyzed similarly in Figure 10 for discoloration using DigitalColor Meter. However, the results were inconclusive due to lighting sensitivity and lack of difference in the colors.

As shown in Table 7, The 4 water-based products, American Marking WJ series,

Akzonobel Aerofine, DuPont 1.2 HG, and Sherwin Williams AQUA-BP, failed Solvent I, alcohol and mineral spirit solution. When exposed to solvent 1, the water-based products were able to be brushed off easily. The acetone-based, American Marking JS series also failed the test. After 3 exposures, missing markings and faded letters are clearly visible. Akzonobel Spray2Fix also failed due to faded letters. (Please refer to Appendix E for raw results)

Company			Befor	e Test					Afte	r Test		
American Marking: JS	J55	J54	J53	J52	J51	J50	2555	.154	J53	352	151	150
American Marking: WJ	J66	J65	J64	J63	J62	J61	1000	25	166	193	102	100
Akzonobel Spray2Fix	J44	J43	J42	J41	J40	<b>J</b> 39	144	J43	142	J41	J40	<b>J</b> 39
Akzonobel Aerofine	J44	J43	J42	J41	J40	J39	•					
DEFT MIL-PRF-85285	J6	J5	J4	J3	J2	JIA	J6	J5	J4	J3	J2	JIA
Dupont 1.2 HG	J40	J39	<b>J</b> 38	<b>J</b> 37	<b>J</b> 36	J35	7.45%		· ·			-
Dupont Industrial Strength	<b>J</b> 39	J38	<b>J</b> 37	<b>J</b> 36	J35	J3#	<b>J</b> 39	J378	J37	<b>J</b> 36	J35	J3.1
Green Polyurethane		(5)	443	AJ3	12	AIL	Alte	13	1	AB	12	(JIM
Sherwin Williams KEM AQUA-BP Enamel	J55	J54	J53	J52	J5 I	J50	J55	J54	353	J52	J51	J50
Sherwin Williams MIL- PRF 22750G	J33	J32	J31	J30	J29	J28	J33	J32	J31	J30	J29	J28
Union Ink – Uniglaze	J17	J16	J15	J14	J13	J12	J17	J16	J15	J14	J13	J12

Table 7 – Snapshot of samples in Phase 1 Testing

Company		Before Test							After	r Test		
American Marking: JS	J50	J49	J48	J47	J46	J45	J50	J49	148	J47	J46	J45
American Marking: WJ	J66	J65	J64	J63	J62	J61	J66	J65	J64	J63	J62	J61
Akzonobel Aerofine	J6	J5	Jet	J3	J2	JIA						
DEFT MIL-PRF-85285	J40	<b>J</b> 39	<b>J</b> 38	J37	<b>J36</b>	<b>J</b> 35	J40	<b>J</b> 39	<b>J38</b>	<b>J</b> 37	<b>J</b> 36	<b>J35</b>
Dupont Industrial Strength	J66	J65	J64	J63	J62	J61	J66	J65	J64	J63	J62	J61
Sherwin Williams KEM AQUA-BP Enamel	J22	J21	J20	J19	J18	J17	122-	121	.120	110	.110	117
Sherwin Williams MIL-PRF 22750G	J17	J16	J15	J14	J13	J12	<b>J</b> 17	J16	J15	J14	J13	J12
Union Ink – <u>Uniglaze</u>	111	JIL	) 1	) ]8	J7	JG	111	JIC	) J	) ]8	J7	16

Of the 11 product lines tested, 5 product line remain as shown in Table 8.

- DuPont Industrial Strength Ultra low VOC polyurethane enamel high gloss topcoat.
- DEFT MIL-PRF-85285 two compound polyurethane topcoat intended for use on exterior application on aircraft and aerospace equipment.
- Sherwin Williams MIL-PRF 22750G Type I– 2.7 VOC compliant high solids two component epoxy topcoat. Intended for use as a top coat of interior ground equipment.
- Union Ink Uniglaze Two compound epoxy ink intended for application such as printed circuit board markings and electronic equipment panels.
- Go Green World Products Green Polyurethane Isocyanine polyurethane paint.

Table 8 - Results of Phase 1 Testing

Company Name & Product Line	Solvent test I: Alcohol & Mineral Spirit	Solvent test III: Terpene Defluxer	Solvent test IV: MEA&PGME
American Marking: JS series - Black/ Red	Failed	N/A	N/A
American Marking: WJ series - Black / Red	Failed	N/A	N/A
Akzonobel Spray2Fix - Black	Failed	N/A	N/A
Akzonobel Aerofine - Black	Failed	N/A	N/A
DEFT MIL-PRF-85285-Black/ Red	Pass	Pass	Pass
DuPont 1.2 HG - White	Failed	N/A	N/A
<b>DuPont Industrial Strength - Black/Red</b>	Pass	Pass	Pass
Green Polyurethane - White	Pass	Pass	Pass
Sherwin Williams – KEM AQUA-BP Enamel - Black/ Red	Failed	N/A	N/A
Sherwin Williams – MIL-PRF 22750G - Black /Red	Pass	Pass	Pass
Union Ink – Uniglaze - Black/Red	Pass	Pass	Pass

These results also confirmed what we already know: three of the five products that passed the test, Uniglaze, MIL-PRF-85285, and MIL-PRF22750G, have been used on tactical equipment.

# 4.2 Phase 2 Test Results

Phase II consisted of further solvent testing, a humidity test, a thermal shock test, and a cleaning system test. These test were conducted on black and red paints. Yellow and white paints were added for the thermal shock portion to ensure the absence of discoloration. (Please refer to Appendix F for raw results)

## 4.2.1 Additional Solvent Resistivity

In addition to the solvent test in Phase I, three other solvents: jet fuel, hydraulic oil, and glycol coolant were added in Phase II to simulate some of the solvents that tactical equipment could be exposed to. These samples were exposed to the solvent in the same manner as Solvent III of MIL-STD- 202G method 215K. The panels were examined from a distance of at least 6 inches with normal lighting and without the aid of magnification for entire or partially missing panels, as well to observe if any were faded, smeared, blurred, or shifted to the extent that they cannot be readily identified. The results are displayed in the Table 9 and 10. No noticeable degradation was observed.

Company		Before Test								Aft	ter T	`est		
DEFT MIL-PRF-	J66	J65	J64	<b>J63</b>	<b>J62</b>	<b>J61</b>	<b>J60</b>	J66	<b>J65</b> J54	J64	J63	<b>J62</b>	<b>J61</b>	<b>J60</b>
85285	J55	J54	J53	J52	J51	J50	J49	J55		J53	J52	J51	J50	J49
Dupont Industrial	J11	J10	J9	J8	J7	J6	J5	J11	J10	) J9	J4 I	3 J7	J6	J5
Strength	J44	J43	J42	J4 I	J40	J39	J38	J44	J43	J42		J40	J39	J38
Green Polyurethane	366 531	<b>J65</b> J30	364 J29	J63 J28	Jボル2 1年7	13	J25	366	<b>J65</b> J30	<b>36</b> 4 129	J63 J28	JT-12 34-7	1	125
Sherwin Williams	J33	J32	J31	J30	J29	J28	J27	J33	J32	J31	J3()	J29	J28	J27
MIL-PRF 22750G	J55	J54	J53	J52	J51	J50	J49	J55	J54	J53	J52	J51	J50	J49
Union Ink – Uniglaze	J22 J11	J24 J10	750 750	119 21	J18 J7	J17 J6	J16 J5	J22 J11	J24 J10	) J20	91L	Section and	J17 J6	J16 J5

Table 9 – Snapshot of samples in Phase II, Additional Solvent Test

Table 10 – Results of Phase II, Additional Solvent Test

Company Name & Product Line	Jet fuel	Hydraulic Oil	Glycol + Coolant
DEFT MIL-PRF-85285 – Black	Pass	Pass	Pass
<b>DuPont Industrial Strength – Black</b>	Pass	Pass	Pass
Green Polyurethane – White	Pass	Pass	Pass
Sherwin Williams MIL-PRF 22750G – Black	Pass	Pass	Pass
Union Ink – Uniglaze – Black	Pass	Pass	Pass

#### 4.2.2 Thermal Shock & Discoloration Test

Thermal shock tested 5 samples, DEFT MIL-PRF-85285, DuPont Industrial Strength, Green Polyurethane, Sherwin Williams MIL-PRF22750G Type I, and Union Ink Uniglaze. In thermal shock testing, the results were evaluated by discoloration, visual check, and a peel test.

Table 11 and Figure 9 show that the samples passed all testing except for Green Polyurethane. Green Polyurethane failed due discoloration, as seen in Figure 10, and was eliminated from further testing. Visual inspection was performed on a stereoscope under 10x magnification to observe for cracks and flaking, but none were observed. The tape test was also performed, but all samples passed the test without flaking.

Company Name & Product Line	Peel Test	Visual Check	Discol (white	loration
DEFT MIL-PRF-85285	Pass	Pass	Pass	132/132
DuPont Industrial Strength	Pass	Pass	Pass	132/132
Green Polyurethane	Pass	Pass	Fail	0/132
Sherwin Williams MIL-PRF 22750G	Pass	Pass	Pass	132/132
Union Ink – Uniglaze	Pass	Pass	Pass	132/132

Table 11- Results of Phase II, Thermal Shock Test

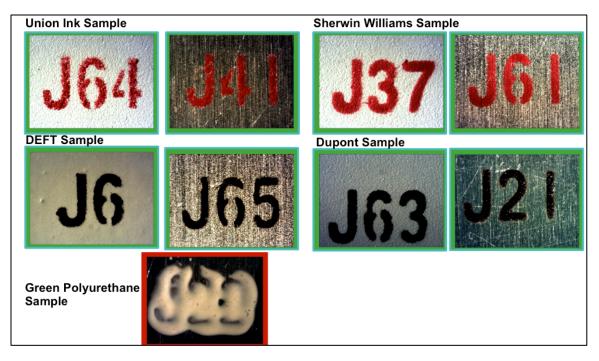


Figure 9 - Snapshot of 10x magnification of Thermal Shock results

	R: 204
	G: 183
2 1	B: 144
2 . 3	
110	R: 191
12 13	G: 187
and the second second	B: 161

Figure 10- Discoloration of Green Polyurethane in white

# 4.2.3 Humidity Test

Humidity testing was performed at Raytheon Facility in Goleta, CA using the humidity profile observed in Figure 5. The test ran for 10 days and results were observed under 10x magnification on a stereoscope, as seen in Table 12.

Company Name & Product Line	Visual (	Check	Peel Test
DEFT MIL-PRF-85285– Black	Pass	126/132 95.45%	Pass
DuPont Industrial Strength - Black	Pass	131/132 99.24%	Pass
Green Polyurethane - Black	Fail	0/132 0%	-
Sherwin Williams MIL-PRF 22750G - Black	Pass	127/132 96.21%	Pass
Union Ink – Uniglaze - Black	Pass	132/132 100%	Pass

## Table 12 - Results of Phase II, Humidity Test

Figure 11 displays a snapshot of each sample. Unlike the previous test, the humidity test did not display a 100% pass rate on all of the samples. However, at least 95% of all samples passed the test, it was determined that the failure rate was most likely due to surface contamination that was not properly cleaned by acetone. The peel test also did not produce any flaking of the samples.



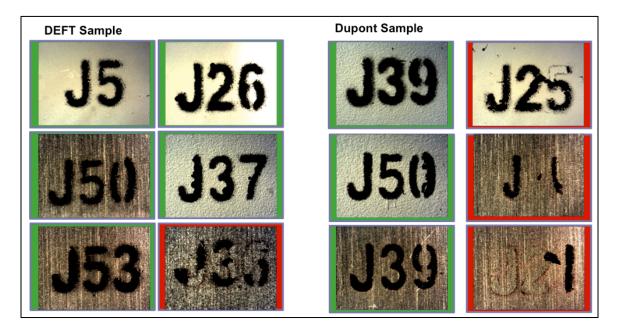
DEFT Sample

**Dupont Sample** 

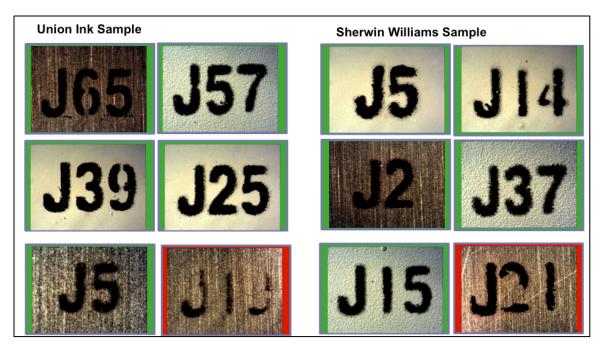
Figure 11 - Snapshot of 10x magnification of Humidity Shock results

#### 4.2.4 Cleaning System Test

Cleaning system testing was performed in Raytheon facility in Forest, MS. Samples were exposed to Branson B-series Degreaser with ultrasonic using Asahiklin AK-225T solvent and Aqueous Technologies Trident batch cleaning system using Kyzen A4615. Visual Check test indicated a 21.97% failure rate with DEFT MIL-PRF-85285, a 41.06% failure rate with DuPont Industrial Strength, a 5.3% failure rate with Sherwin Williams MIL-PRF 22750G Type I, and 12.88% failure rate with Union Ink. Union Ink and MIL-PRF 22750G was found to have the highest pass rate as shown in Table 13. The failure of the other three samples was due to missing parts in the letters. In Figures 12 and 13, a snapshot of the samples display some of the missing parts from the samples.



*Figure 12 - Snapshot of 10x magnification of Cleaning Systems Test of DEFT and DuPont samples.* 



*Figure 13 - Snapshot of 10x magnification of Cleaning Systems Test of Union Ink and Sherwin Williams samples.* 

Table 13 - Phase II,	Cleaning Systems Test
----------------------	-----------------------

Company Name & Product Line	Visual Check		Peel Test
DEFT MIL-PRF-85285– Black	Fail	103/132 78.03%	-
DuPont Industrial Strength– Black	Fail	91/132 58.94%	-
Green Polyurethane – Black	Fail	0/132 0%	-
Sherwin Williams MIL-PRF 22750G – Black	Pass	125/132 94.70%	Pass
Union Ink – Uniglaze – Black	Pass	115/132 87.12%	Pass

To determine the cutoff and to justify for the failure rates, further investigation was needed to observe that Union Ink currently works for marking at Raytheon. Therefore, Union Ink was determined to be a suitable control to compare the data with. By that standard the only paint that passed the test was Sherwin Williams MIL-PRF 22750G Type I.

#### **Chapter 5 – Conclusion and Future Work**

Eleven product lines were tested, including epoxy, polyurethane, and water-based solvent marking ink. Two product line, Sherwin Williams MIL-PRF-22750G Type I and Union Ink Uniglaze, passed:

- MIL-STD 202G Method 215K solvent test
- MIL-STD 202G Method 107G thermal shock test
- MIL-STD-810G, Method 507.5 humidity test
- Cleaning Systems Test
- ASTM D3359, measure adhesion by tape test

Sherwin Williams MIL-PRF-22750G Type I is a high solids epoxy topcoat designed for use as a topcoat for the interior of military ground equipment. Uniglaze Epoxy ink is designed for use on printed circuit board markings, electronic equipment panels, and automotive/aeronautical/nautical components. As with all marking and paint related products, these products also contain some harmful ingredients as shown in Table 14.

Comparing the composition of these two product in Table 15, the composition of Enthone 50 series ink contain far more carcinogenic compounds and reproductive toxins vs Sherwin Williams MIL-PRF 22750G Type I and Union Ink Uniglaze. (Please refer to Appendix B for Union Ink TDS and MSDS and Appendix C for Sherwin Williams TDS and MSDS)

Given the readily availability of alternatives, and their success through all levels of testing, it is recommended that all use and purchase of Enthone marking inks cease and that the Union Ink Uniglaze and Sherwin Williams MIL-PRF-22750 Type I be the replacements.

Sherwin Williams I Type I	MIL-PRF-22750G	Union Ink Uniglaze			
Ink	Catalyst V93V00228	Ink	Catalyst UGLZ-9120, UNGL-9130		
Acetone	2 – Propanol	Epoxy Resin	Naphtha		
Methyl n-amyl ketone	4 – Nonlphenol	Organo-titanates- quat	Polyamide Resin		
Diisobytyl Ketone	1,3- benzendimethanamine	2-butoxyethanol	Naphthalene		
Epoxy Polymer	Polyamine	1-methoxy-2- propanol acetate			
Non-organic Pigments i.e. Titanium Oxide, Carbon Black		Solvent naphtha			
		Butyl carbitol			
		acetate			

Table 14 - Harmful Chemicals in Sherwin WIlliams MIL-PRF-22750G Type I and Union Ink Uniglaze

*Table 15 - Carcinogenic and reproductive toxin comparison of Sherwin Williams MIL PRF-22750G Type I, Union Ink Uniglaze and Enthone 50 series product line.* 

	Sherwin Williams MIL PRF-22750G Type I	Union Ink Uniglaze	Enthone 50 Series
Ink to Catalyst ratio	4:1	4:1 to 6:1	Varies on catalyst
Carcinogenic	1 possible, depending on the color i.e. Titanium Oxide(white), Carbon Black(black) 0 in catalyst	0 in ink 1 in catalyst	4 in ink catalyst: vary on the catalyst used
reproductive toxin or suspected reproductive toxin	0	0	3 in ink catalyst: vary on the catalyst used

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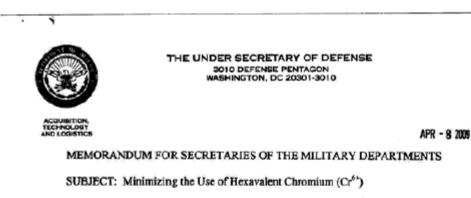
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#### Appendix A

Memo from Under Secretary of Defense, April 8<sup>th</sup>, 2009



Cr<sup>5+</sup> is a significant chemical in numerous Department of Defense (DoD) weapons systems and platforms due to its corrosion protection properties. However, due to the serious human health and environmental risks related to its use, national and international restrictions and controls are increasing. These restrictions will continue to increase the regulatory burdens and life cycle costs for DoD and decrease materiel availability. OSD, DoD Components, and industry have made substantial investments in finding suitable replacements for Cr<sup>6+</sup> for many of the current DoD applications. In particular, a number of defense-related industries are minimizing or eliminating the use of Cr<sup>6+</sup> where proven substitutes are available that provide acceptable performance for the application.

This is an extraordinary situation that requires DoD to go beyond established hazardous materials management processes. To more aggressively mitigate the unique risks to DoD operations now posed by Cr<sup>44</sup>, I direct the DoD Military Departments to take the following actions:

- · Invest in appropriate research and development on substitutes.
- Ensure testing and qualification procedures are funded and conducted to qualify technically and economically suitable substitute materials and processes.
- Approve the use of alternatives where they can perform adequately for the intended application and operating environment. Where Cr<sup>6+</sup> is produced as a by-product from use or manufacture of other acceptable chromium oxides, explore methods to minimize Cr<sup>6+</sup> production,
- Update all relevant technical documents and specifications to authorize use of the qualified alternatives and, therefore, minimize the use of materials containing Cr<sup>5+</sup>.
- Document the system-specific Cr<sup>6+</sup> risks and efforts to qualify less toxic alternatives in the Programmatic Environment, Safety, and Occupational Health Evaluation for the system. Analyses should include any cost/schedule risks and life cycle cost comparisons among alternatives. Life cycle comparisons should address material handling and disposal costs and system overhaul cycle times/costs due to any differences in corrosion protection.
- Share knowledge derived from research, development, testing and evaluations (RDT&E) and actual experiences with qualified alternatives.



#### Appendix B

#### TDS and MSDS of Union Ink: Uniglaze



#### Applications

- Printed circuit board markings
- · Electronic equipment panels
- · Glass before or after mirroring
- Metal Casings
- Automotive / Aeronautical / Nautical components
- Polyethylene bottles (for superior product resistance)
- Polypropylene (Polyallomer Plastics)

# SPECIFICATIONS: UGLZ-Series

Appearance: High Gloss, semi-gloss or flat, depending upon type of catalyst used. Black and white are available in both gloss and flat finishes. Uniglaze Inks have excellent flexibility, even for flexing and creasing on metal, polyethylene and other surfaces.

#### Opacity:Excellent.

Drying: Air Dry: 12 hours. Force Dry: 325°F. (162°C) for 5 to 10 minutes. 250°F. (121°C.) for 10 to 20 minutes. Whites should not be cured above 250°F. (121°C.). When air drying, chemical resistance and hardness will be good after 72 hours but full cure may not be achieved for 7 days.

Coverage: Approximately 800 to 1200 square feet per gallon.

Mesh: Monofilament meshes from 156 to 305 depending on substrate and art work.

Stencils: Direct or Capillary film type emulsions

Wash-up: Screen solvent SOLV-1540 or Safety Screen Solvent SOLV-1500.

#### Mixing instructions:

UNIGLAZE Colors must be mixed with Catalyst prior to use according to the following recommendations (by volume or weight). See specifications for recommended mixing ratios in the following paragraphs.

#### Features

- Outstanding chemical resistance
- Excellent flexibility
- Excellent opacity
- Choose high gloss, semi-gloss or flat
- Meets many US military specs.

#### Ink to Catalyst Ratios:

#### UGLZ-9120 (Standard) & UGLZ-9130 (Fast):

6 parts INK to 1 part Catalyst provides maximum gloss, hardness, electrical & chemical resistance. To gain maximum flexibility and adhesion the ratio can be moved towards 4 parts INK to 1 part Catalyst.

These ratios are not absolutely critical. However the end user is responsible for testing to ensure maximum performance on the materials being imprinted.

<u>UGLZ-9141(Flat/Fast</u>): When utilizing this catalyst care should be taken not to exceed the recommended ratio of 4 parts INK to 1 part Catalyst as the inks overall performance will be diminished.

When screen printing add Reducer SOLV-1498 as necessary, perhaps 10-20%. When using the above mixtures for application by rubber stamp, reducer may not be necessary.

Pot life: Ink/Catalyst Mixture may be used for up to eight hours. Ambient temperatures and climatic conditions may alter pot life. Always be sure to keep ink and catalyst in a proper container and cover when not in use.

Shelf life: The shelf life of the unmixed ink and catalyst is at least two years from Date of Shipment if sealed and stored at room temperature (approx.73°F / 23°C). Uniglaze Inks, like most epoxies, are not recommended for outdoor weathering. Always pre-test for adhesion, chemical resistance or other specific requirements.



# Uniglaze Epoxy Ink (Page 2@2)

Colors: UGLZ-1000 White UGLZ-1020 Extra Opaque White UGLZ-1050 Flat White UGLZ-2001 Primrose Yellow UGLZ-2010 Lemon Yellow UGLZ-2020 Chrome Yellow UGLZ-2050 Orange UGLZ-3000 Vermillion Red UGLZ-3000 Vermillion Red UGLZ-3030 Maroon UGLZ-4010 Magenta UGLZ-5020 Mono Blue UGLZ-5030 Ultra Blue UGLZ-5040 Navy Blue

Colors Cont: UGLZ-6010 Tahiti Green UGLZ-7000 Dark Brown UGLZ-7030 Sienna Brown UGLZ-7030 Sienna Brown UGLZ-80000 Black UGLZ-80500 Flat Black UGLZ-80500 Flat Black (for Type II Cert.) UGLZ-80500 Flat Black (for Type II Cert.) UGLZ-9030 Clear For Gold UGLZ-9040 Sharp Printing Compound UGLZ-9040 Sharp Printing Compound UGLZ-9040 Half-tone Base UGLZ-9110 Overprint Gloss Clear UGLZ-9110 Overprint Gloss Clear UGLZ-9110 Silver

#### Catalysts:

UGLZ-9120 Gloss Catalyst (Reg) UGLZ-9130 Gloss Catalyst (Fast) Rec. when mirroring glass. UGLZ-9141 Flat Catalyst (Fast) Rec. when mirroring glass.

Additives & wash-ups: SOLV-1329 Tropical Retarder SOLV-1498 Standard Reducer SOLV-1500 Safety Wash SOLV-1540 Screen Washing Thinner

#### CERTIFICATES OF COMPLIANCE (C. OF C.'S)

If requested, AT THE TIME OF PLACING THE ORDER, we can test a batch and certify that particular Uniglaze ink batch will comply to certain federal, military and industrial specifications when properly applied and properly stored. Contact our customer service department for testing and documentation charges or lot test data charges.

#### Some of the specifications that Uniglaze meets and C. of C.'s can be issued for are:

MIL-I-43553 MIS-11272A	U.S. ARMY MISSILE COMMAND REDSTONE ARSENAL
MIS-19916C	U.S. ARMY MISSILE COMMAND REDSTONE ARSENAL
2424192 2424193	BU WEPS BU WEPS
61A5A65	U.S. NAVAL ORDINANCE
WS19208A 13042164	NAVAL SEAS SYSTEMS COMMANDS U.S. ARMY MISSILE COMMAND REDSTONE ARSENAL
MS18038 (AS)	MILITARY STANDARD
MM817Y CID A-A-56032	MARTIN MARIETTA INK MARKING, EPOXY BASE (TYPE I AND TYPE II)

<u>Medical & Surgical devices:</u> Although this ink system is being utilized widely and successfully in the medical industry, Union Ink has not tested this product nor has it received any FDA approval regarding Bio-Compatibility or any autoclave sterilization processes, as it relates to markings on actual surgical devices or any part thereof. It is the sole responsibility of the end user to have this ink tested for feasibility on such devices.

#### ALWAYS TEST BEFORE USING IN PRODUCTION!

While Union Ink Company believes that the data contained herein are factual and the opinions expressed are those of qualified experts regarding the results of the tests conducted, the data are not to be taken as a warranty or representation for which Union Ink Company assumes legal responsibility. They are offered solely for your consideration, investigation, and verification. Always pre-test inks on surfaces to be printed. r

<b>U</b> n				AFETY D	ATA SHE	453 Broa Ridgefield	: Company d Avenue d, NJ 07657	
1.	CHEMI	CAL PRO	DUCT	AND CON	IPANY II	DENTIFIC	ATION	
SUPPLIER LOC Union Ink Compa 453 Broad Avenu Ridgefield, NJ 07 201-945-5766 IN CASE OF EM	any ie 1657	CONTACT: 2	01-945-57		ZARDOUS I	Personal	IFORMATIO Health: mmability: Reactivity: Protection: ction 8 for Pl	2 2 0 *
PRODUCT NAM CHEMICAL FAN	NLY:	Uniglaze Epoxy Mix	kture				E REVISED: PERSEDES: VERSION:	
UGLZ-	1000 1020 1050 1627 2001	2010 2020 2050 3000 3005	3020 3030 4010 4153 5040	6010 7000 7030 8050R P110U	P189C	P253C P265U P268U P297C P298C		P5783
PREPARED BY:		Kimberly	C. Leitch	(704) 553-00	)46 ext. 155			
		2. H	AZARD	S IDENTI	FICATIO	N		
EMERGENCY OVERVIEW: DANGER! Combustible. Causes respiratory tract irritation. PRIMARY ROUTE OF ENTRY: Respiratory, eye and skin contact								
EYE CONTACT:		May cause	e severe di	scomfort or in	itation.			
SKIN CONTACT	SKIN CONTACT: May cause skin irritation and sensitization							
INHALATION: Respiratory irritation. Breathing high vapor concentrations may cause CNS depression resulting in dizziness, light-headedness, headache, nausea, and loss of coordination. Continued inhalation may result in unconsciousness and death. Prolonged and repeated exposure to high concentrations may cause hearing loss.								
INGESTION:		May be ha	armful. Asj	piration hazard	1.			

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3. IN	3. INGREDIENTS AND HAZARDS						
INGREDIENTS	CAS#	% WEIGHT	TLV	PEL			
Epoxy Resin organo-titanates-quat 2-Butoxyethanol 1-Methoxy-2-propanol Acetate Solvent naphtha (petroleum) Light Aromatic	25036-25-3 198840-66-3 111-76-2 108-65-6 64742-95-6	23-53% 4.5-15% 4.5-11.6% 3.5-8.5% 0-7%	5 mg/m <sup>3</sup> (dust) Not Established Not available Not Established 100 ppm	5 mg/m3 (dust) Not Established 25 ppm (skin) Not Established 100 ppm OSHA TWA			
butyl carbitol acetate	124-17-4	<2%	Not Available	150ppm OSHA PEL/STEL Not Available			
These products are defined as hazardo				9 CFR 1910.1200.			
	4. FIRST	AID MEAS	URES				
EYES: Flush with water for 15 SKIN: Remove contaminated (				al attention.			
	2						
INHALATION: Remove to fresh air for	relief. If overc	ome by vapors,	, get medical attention.				
INGESTION: Do not induce vomiting. If spontaneous vomiting occurs, keep head below hips to prevent aspiration. Get medical attention.							
5.	FIRE FIG	HTING ME	ASURES				
FLASH POINT (° F): >125 °F	(closed cup)						
OSHA FLAMMABILITY CLASSIFICATION: Combustible Liquid, Class II							
EXTINGUISHING MEDIA: CO <sub>2</sub> , dry chemical, water fog or fine spray, foam (ATC type preferred). Water stream may spread fire.							
SPECIAL FIRE FIGHTING PROCEDURES: Wear self contained breathing apparatus and full protective gear.							
EXPLOSION LIMITS IN AIR - LOWER	(%):	Unknown	UPPER (%):	Unknown			
AUTO IGNITION TEMP (° F): Unknown							
UNUSUAL FIRE AND EXPLOSION HAZARDS: None Known							
6. ACCIDENTAL RELEASE MEASURES							
	from entering	drains or water	Dike to contai ways. Remove possible ning product with absort	-			

10. STABIL	ITY AND	D REACTIVITY (CONTINUE	D)
INCOMPATIBILITY (MATERIALS TO	AVOID):	Strong oxidizing agents	
CONDITIONS TO AVOID: Storage	temperature	es above 95° F. Igniotion sources.	
		2	
11. T	OXICOL	OGICAL INFORMATION	
		not listed as carcinogens by NTP, IAR not listed as reproductive or development	
	2-butoxyet organo-tita organo-tita Solvent Na 2-butoxyet organo-tita Solvent Na big): organo-tita titanates-qu	anates-quat (base titanate component) aphtha (petroleum) Light Aromatic: hanol: inates-quat (DMAPMA component) aphtha (petroleum) Light Aromatic: 2-butoxyethanol: inates-quat (DMAPMA component)	I
12.	ECOLO	GICAL INFORMATION	
PERSISTENCE AND DEGRADABILITY:		thanol —BOD (% Oxygen consumption) 74%, Day 20 – 88%	): Day 5 – 26%,
		-2-Propanol Acetate— BOD (% Oxyger 3%, Day 10 – 25%; Day 20 – 98%	n consumption):
ECOTOXICITY: 2-Butox	yethanol:	Toxicity to Microorganisms: Bacteria Toxicity to Aquatic Invertebrates: Da >1,000 mg/l. Toxicity to Fish: Fathead Minnow: L 1,700 mg/l.	phnia LC50, 48 hours,
1-Methoxy-2-Propanol /	Acetate:	Ecotoxicity to Microorganisms: Bact >10,000 mg/l. Ecotoxicity to Aquatic Invertebrates: 629.4 mg/l. Ecotoxicity to Fish: Fathead Minnow 200.8 mg/l.	Daphnia LC50, 48 hours,

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## MSDS of Catalyst

-

<b>V</b> Unior	MATERIAL SAFETY	Union Ink Company 453 Broad Avenue Ridgefield, NJ 07657 DATA SHEET
1. CHEMI	CAL PRODUCT AND CO	OMPANY IDENTIFICATION
SUPPLIER LOCATION: Union Ink Company 453 Broad Avenue Ridgefield, NJ 07657 201-945-5766 IN CASE OF EMERGENCY		HAZARDOUS MATERIAL INFORMATION SYSTEM: Health: 3 Flammability: 2 Reactivity: 0 Personal Protection: * * See Section 8 for PPE
PRODUCT NAME: CHEMICAL FAMILY: PRODUCT CODE: PREPARED BY:	Uniglaze Gloss Catalyst Epoxy UGLZ-9120, UGLZ-9130 Kimberly C. Leitch (704) 553	DATE REVISED: 1/25/2012 SUPERSEDES: 10/10/2011 VERSION: 4
EMERGENCY OVERVIEW:	-	TIFICATION n and may cause skin irritation and sensitization.
PRIMARY ROUTE OF ENTE	RY: Eye, Skin, Inhalation	
EYE CONTACT:		to severe eye irritation. Direct contact with the or mists may cause stinging, tearing, redness and
SKIN CONTACT:	This material causes moderate cause sensitization in some ind	skin irritation. Repeated and prolonged contact may ividuals.
INHALATION:	the nose and throat. May cause	ated product may cause irritation or sensitization of e headaches, dizziness, anaesthesia, drowsiness, ntral nervous system effects, including death
INGESTION:	Minimal toxicity, and not a likely is possible.	v route of exposure. Aspiration of liquid into lungs

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3. IN	IGREDIE	NTS AND H	AZARDS	
INGREDIENTS	CAS #	% WEIGHT	TLV	PEL
Naphtha (petroleum) heavy aromatic Polyamide Resin 2,4,6-Tris(dimethylaminomethyl)Phenol Naphthalene	64742-94-5 68410-23-1 90-72-2 91-20-3		Not determined Not determined Not determined 10 ppm	Not determined Not determined Not determined 10 ppm
These products are defined as hazardo	us by the OSI	HA Hazard Com	nmunication Standard, 2	9 CFR 1910.1200.
	4. FIRST	AID MEAS	URES	
EYES: Flush with water for 15	minutes. If im	itation develops	s or persists, get medica	l attention.
SKIN: Remove contaminated of	lothing. Was	sh with soap and	d water.	
INHALATION: Remove to fresh air for	elief. If overc	come by vapors,	get medical attention.	
INGESTION: Do not induce vomiting. aspiration. Get medica	-	us vomiting occ	urs, keep head below hi	ips to prevent
5.	FIRE FIG	HTING ME	ASURES	
FLASH POINT (° F): 145 °F (	TCC ASTM D	56)		
OSHA FLAMMABILITY CLASSIFICAT	ION:	Combustible		
	r chemical, wa nay spread fir	-	spray, foam (ATC type p	referred). Water
SPECIAL FIRE FIGHTING PROCEDUR	RES:	Wear self conta gear.	ained breathing apparate	us and full protective
EXPLOSION LIMITS IN AIR - LOWER	(%):	0.8 @ 77°F	UPPER (%):	5.7 @ 77°F
AUTO IGNITION TEMP (° F):		849°F		
UNUSUAL FIRE AND EXPLOSION HA	ZARDS:	None Known		
6. ACC	IDENTAL	RELEASE	MEASURES	
	from entering	drains or water	D: Dike to contain ways. Remove possible ning product with absorb	-

Page 2 of 5

1/26/2012

10. \$		REACTIVITY (	CONTINUED)		
INCOMPATIBILITY (MATER	IALS TO AVOID):	Strong oxidizing age	nts and acids		
CONDITIONS TO AVOID:	Storage temperature	es above 95° F. Ignioti	on sources.		
	11. TOXICOL	OGICAL INFORI	MATION		
CARCINOGENICITY:	Cancer evaluated na for carcinogenicity ir in exposed humans human carcinogen ( The U.S. National Te	aphthalene and conclud n experimental animals . Accordingly, IARC cla Group 2B).	emational Agency for Research on led that there was sufficient evidence , but inadequate evidence for cancer assified naphthalene as a possible P) has evaluated naphthalene and found a human carcinogen.		
ACUTE ORAL LD50 (Rat):	Polyamide Resin:	>5 g/kg			
SKIN (Rabbit):	Polyamide Resin:	Moderate			
EYE (Rabbit):	Polyamide Resin:	Severe			
	12. ECOLO	GICAL INFORM	ATION		
The following test results are	for a similar product to	o the polyamide resin u	used in this product ::		
Fish Toxicity, OECD 203 (G	olden Orfe):	LC50 (96hr):	2.3 mg/l		
Crustacean Toxicity, OECD	202 (Daphnia):	EC50(48hr):	0.55 mg/l		
Algistatic Effect, OECD 201	:	ErC50(24-48hr): EbC50(72hr):	2.9 mg/l 2.3 mg/l		
Ready Biodegradation, OE	CD301B (Modified St	urm):	0% degraded after 28 days.		
Partition Coefficient, OECD	117/1:	Log10Pow	3.51		
	13. DISPOS	AL CONSIDERA	TIONS		
RCRA:			d this product become a waste waste (Hazardous Waste # D001).		
WASTE DISPOSAL:	Incinerate liquid and federal regulations.	I contaminated solids in	n accordance with local, state, and		
EMPTY DRUMS: Empty contains retain product residue (liquid and/or vapor) and can have the same hazardous waste characteristics as the material they previously contained. Empty drums should be completely drained, properly bunged and promptly returned to a drum reconditioner or properly disposed of.					

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Appendix C

### TDS and MSDS of Sherwin Williams

TDS



DESCRIPTION

# **Product Finishes**

CC-M17

# MIL-PRF-22750G, Type I & Type II High Solids Epoxy Topcoat

 Gloss White 17925
 F91W226
 Flat Gray 36375
 F93A603

 Semi-Gloss Tan 23446
 F92H101
 Catalyst
 V93V228

 Semi-Gloss Seafoam Green 24533
 F92G227
 V93V228

### CHARACTERISTICS

#### **SPECIFICATIONS**

MIL-PRF-22750G Type High Solids Epoxy Topc gal VOC compliant high sc ponent epoxy topcoat. Thi ing is intended for use as the interior of military groun This product is applied of epoxy primers. The following products are the U. S. Army Research L Proving Ground, Aberdeen Sherwin-Williams#	oat is a 2.8 lb/ blids two com- is epoxy coat- a topcoat for nd equipment. over specified e approved by .ab, Aberdeen	Component A: Component B: Admixed: Viscosity: Admixed: Recommended fill Mils Wet Film builds will va hiding. Higher build Mils Dry Spreading Rate (n	61 - 66% (Typical) 20-32 seconds #4 Ford m thickness: 2.8-3.1 uy by color to achieve full is may be necessary. 1.5-2.5 o application loss) @ 1.8-2.0 mils DFT , 77°F, 50% RH): 4 hours maximum 8 hours maximum	Steel: Surface must be clean and free of grease, dirt, oil, rust, fingerprints, and other contaminants to insure optimum adhesion and performance properties. Chemical pretreatment, TT-C-490, Type I, zinc phos- phate or DOD-P-15328D wash primer, E90G4, gives best adhesion and perform- ance results. Where blasting is appropri- ate, blast in accordance with SSPC-SP10 or SSPC-SP5. For optimum adhesion pre- treat blasted surface immediately. Prime with wash primer E90G4 within two hours after blasting. Aluminum: Clean with acidic cleaner or other appropriate cleaner depending on contamination. Pretreat with chromate conversion coating MIL-DTL-5541, wash
F91W226	Q2043	Force Dry:	/ days to obtain dry hard	primer DOD-P-15328 (E90G4), or anodize
F93A603	Q2042		30 minutes at 145°F	per MIL-A-8825-MIL-C-8514 (E90G16).
F92G227	Q2128		3227 will be lower than the um of 15 units if force dried.	Galvanized and other metals: Clean and remove oxidation contamination on surface
Note: Approval is only requ two colors. Sherwin-Will proved to supply any addit all gloss levels. See produ CC-M17A MIL-PRF-22750 System colors.	iams is ap- ional colors in ict data page	Thicker films or low cure time. Flash Point: Mixing Ratio: 4 parts 1 part Induction Time: Pot Life: ture - higher temper Package Life: Air Quality Data: Non-photochemical Volatile Organic Co catalyzed as above 2.8 lb/gal, 335 g/	mpounds (VOC) , maximum	remove oxidation contamination on surface, followed by treatment with DOD-P-15328D wash primer (E90G4), MIL-C-8814 (E90G16), or anodize per MIL-A-8825. Due to the variability in these surface, test- ing adhesion on each situation is recom- mended. <b>Primers must be applied under the top-</b> coat. For ferrous substrates, use MIL- DTL-53022 primers. For non-ferrous substrates, MIL-P-23377 (E90G203) (Type I, Class C2, 2.8 VOC); or MIL-P-53022 (see above). Check the data sheet of each primer for recoat time of topcoat. <b>Testing:</b> Due to the wide variety of sub- strates, surface preparation methods, application methods, and environments, the customer should test the complete system for adhesion, compatibility and performance prior to full scale applica- tion.

CC-M17

continued on back

APPLICATION Typical Setups           Reduction:         Reduction is not recommended.           Optimum sag resistance is obtained without reduction.         If required, use MAK (R6K30), MEK (R6K10), MIL- 1-81772 Type II Reducer (R91K210) or CARC Reducer (R91K25) - 75/25 (T-BAc/MAK).           Please consult with your Sherwin-Williams sales representative for proper settings for your spray equipment.           Cleanup:           Clean tools/equipment immediately after use with MIL-T-81772 Type II Thinner (R90K210), MEK (R6K10), or other epoxy reducer. A blend of MIBK and Xylene works well also.           Follow manufacturer's safety recommendations when using any solvent.	Product Lin MIL-PRF- A) must b (Compone by volume Do not us Do not va Compone prior to us Agitate er Compone fore spray A 30-minu sary. Potlife will temperatu Force dry this produ mended fi	22750 coatin e catalyzed v ent B), V93V: e. e other cataly ry catalyst m int A must be se. htire mixture, int B, and Re /. ute induction I be shorter v ures. ing will lower ict. This coal or interior app	igs (Compo with Catalys 228, at 4:1 r ysts. ixing ratio. well agitate Componen ducer well k period is ne vith warmer the gloss o ting is recon plication onl	t ratio t A, be- eces- f n- ly.	Thorou Materi safety produc A Mat able fi facility Please ments facility Please ments facility velating the cus uct Dat The ii stated rentty of tests b to vari method known William	NDUSTRIAL ughly revie al Safety D and cautio t. verial Safety rom your k e direct an to your k Product Da updated to r g to the prod stomer obtain ta Sheet for information, here pertai offered and elieved to be iations in c ds of appli or under ou	JTIONS SHOP APPLICATION w product label a ata Sheet (MSDS) ins prior to using to potential Sheet is avoid to Data Sheet is avoid occal Sherwin-Willia by questions or co- board Sherwin-Willia ta Sheets are perior reflect new information to the most recent Pr the product being us rating, and opini- in to the material of represent the results reliable. However, of ustomer handling a cation which are ir control, The Sherv cannot make any w d result.
	MIL PRF	22750G Tyj	pe I & Type	e II			
COLOR	SW CODE	Vol Solids	VISC (#4 Ford)		Film kness	Dry Film Thickness	SPREAD RATE
Gloss White 17925	F91W226	62-64	20-30	2.9	- 3.2	1.8 - 2.0	504-560
	F92G227	60-63	22-32	<u> </u>	- 3.2	1.8 - 2.0	492-547
	F92H101	55-57	20-30	3.2	- 3.6	1.8 - 2.0	448-497
	93A603 62-64 - 2.8-					1.8 - 2.0	508-564

CC-M17

MIL-PRF-22750G Type I & Type II High Solids Epoxy Topcoat

### MSDS of white Ink

#### MATERIAL SAFETY DATA SHEET

V93V00228 04 00

DATE OF PREPARATION Apr 18, 2013

SECTION 1 - PRODUCT AND COMPANY IDENTIFICATION

PRODUCT NUMBER

V93V00228 PRODUCT NAME V93V00228 MIL-PRF-22750G T1 HIGH SOLIDS EPOXY TOPCOAT CATALYST MANUFACTURER'S NAME THE SHERWIN-WILLIAMS CO. 101 Prospect Avenue N.W. Cleveland, OH 44115

Telephone Numbers and Websites

receptione manufers and messiles	
Regulatory Information	(216) 566-2902
Medical Emergency	(216) 566-2917
Transportation Emergency*	(800) 424-9300
"for Chemical Emergency ONLY (sp	ill, leak, fire, exposure, or
	accident)

#### SECTION 2 - COMPOSITION/INFORMATION ON INGREDIENTS

% by Weight	CAS Number	Ingredient	Units	Vapor Pressure
26	67-63-0	2-Propanol		
		ACGIH TLV	200 PPM	33 mm
		ACGIH TLV	400 PPM STEL	
		OSHA PEL	400 PPM	
45	84852-15-3	4-Nonylphenol		
		ACGIH TLV	Not Available	
		OSHA PEL	Not Available	
22	1477-55-0	1,3-Benzenedimethana	amine	
		ACGIH TLV	0.1 ppm (Skin) CEILING	
		OSHA PEL	0.1 ppm (Skin) CEILING	
7	Proprietary	Polyamine		
		ACGIH TLV	Not Available	
		OSHA PEL	Not Available	

#### SECTION 3 - HAZARDS IDENTIFICATION

ROUTES OF EXPOSURE

INHALATION of vapor or spray mist. EYE or SKIN contact with the product, vapor or spray mist.

EFFECTS OF OVEREXPOSURE

EYES: Causes burns. SKIN: Causes burns.

INHALATION: Causes burns of the upper respiratory system.

May cause nervous system depression. Extreme overexposure may result in unconsciousness and possibly death.

Prolonged overexposure to hazardous ingredients in Section 2 may cause adverse chronic effects to the following organs or systems: the reproductive system

SIGNS AND SYMPTOMS OF OVEREXPOSURE

Headache, dizziness, nausea, and loss of coordination are indications of excessive exposure to vapors or spray mists.

Redness and itching or burning sensation may indicate eye or excessive skin exposure.

MEDICAL CONDITIONS AGGRAVATED BY EXPOSURE

May cause allergic skin reaction in susceptible persons or skin sensitization. CANCER INFORMATION

For complete discussion of toxicology data refer to Section 11.

HMIS Codes

Health 3\* Flammability 3

Reactivity 0

### Catalyst MSDS

#### MATERIAL SAFETY DATA SHEET

V93V00228 04 00

DATE OF PREPARATION Apr 18, 2013

#### SECTION 1 - PRODUCT AND COMPANY IDENTIFICATION

PRODUCT NUMBER V93V00228 PRODUCT NAME V93V00228 MIL-PRF-22750G T1 HIGH SOLIDS EPOXY TOPCOAT CATALYST MANUFACTURER'S NAME THE SHERWIN-WILLIAMS CO. 101 Prospect Avenue N.W. Cleveland, OH 44115

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Transportation Emergency*	(800) 424-9300
for Chemical Emergency ONLY (sp	oill, leak, fire, exposure, or
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SECTION 2 - COMPOSITION/INFORMATION ON INGREDIENTS

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		ACGIH TLV	200 PPM	33 mm
		ACGIH TLV	400 PPM STEL	
		OSHA PEL	400 PPM	
45	84852-15-3	4-Nonylphenol		
		ACGIH TLV	Not Available	
		OSHA PEL	Not Available	
22	1477-55-0	1,3-Benzenedimethana	amine	
		ACGIH TLV	0.1 ppm (Skin) CEILING	
		OSHA PEL	0.1 ppm (Skin) CEILING	
7	Proprietary	Polyamine		
		ACGIH TLV	Not Available	
		OSHA PEL	Not Available	
SECTION 3 - HA	ZARDS IDENTI	FICATION		
ROUTES OF EXPOSURE				HMIS Codes
INHALATION of vapor o	r spray mist.			Health 3*

INHALATION of vapor or spray mist. EYE or SKIN contact with the product, vapor or spray mist. EFFECTS OF OVEREXPOSURE EYES: Causes burns. SKIN: Causes burns.

INHALATION: Causes burns of the upper respiratory system.

May cause nervous system depression. Extreme overexposure may result in unconsciousness and possibly death. Prolonged overexposure to hazardous ingredients in Section 2 may cause adverse chronic effects to the following organs or systems:

· the reproductive system SIGNS AND SYMPTOMS OF OVEREXPOSURE

Headache, dizziness, nausea, and loss of coordination are indications of excessive exposure to vapors or spray mists.

Redness and itching or burning sensation may indicate eye or excessive skin exposure. MEDICAL CONDITIONS AGGRAVATED BY EXPOSURE

May cause allergic skin reaction in susceptible persons or skin sensitization. CANCER INFORMATION

For complete discussion of toxicology data refer to Section 11.

Flammability 3

Reactivity 0

# Appendix D

# Curing Schedule of 11 Products

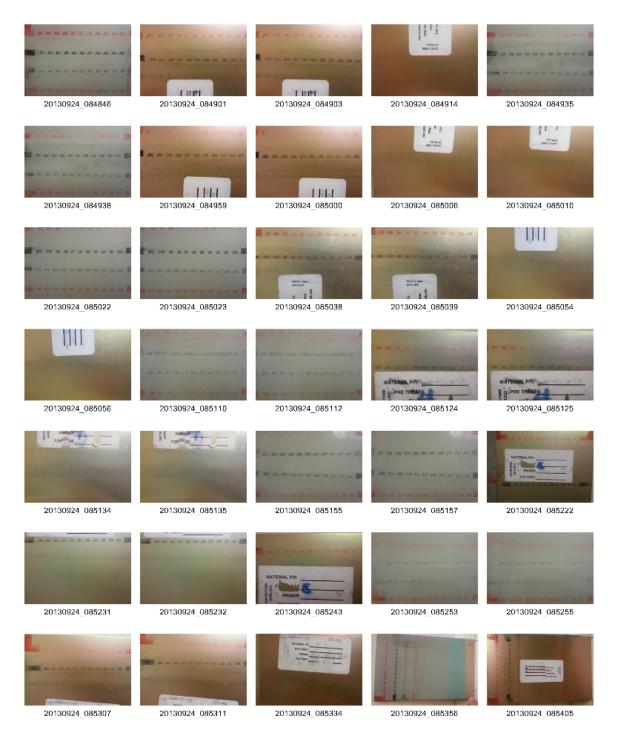
Akzonobel		21 Celsius	40 Celsius	70 Celsius	
Spray2Fix	Dry to dust	3 hours	60 minutes	30 minutes	
	Dry to				
	handle	16 hours	90 minutes	60 minutes	
_	Minium				
Aerofine 8250	Recoat time		30 minutes		
	Maximum re	coat time	48 hours		
Union Ink - Uniglaze		21 Celsius	121 Celsius	162 Celsius	
		401	10.00		
		12 hours	10-20 min.	5-10 minutes	
	Complete cure	7 Days			
	cure	7 Days			
Sherwin Williams		21 Celsius	62.8 C		
KA BP Enamel	To Touch	10-15 minutes			
	To Handle	40-50 minutes			
	Tack Free	15-20 minutes			
	To Sand	50-60 minutes			
22750G Epoxy	To Touch	4 Hours			
	Dry Hard	8 Hours maximum	30 minutes		
	Complete Cure	7 Days			

					(71.1C)16	
DEFT		21 Celsius	(48.9C)120F	(60C)140F	OF	(82.2C)180F
85285E Polyurethane	To Touch	6 Hours				
					20	
	Dry Hard	12 Hours	45 minutes	30 minutes	minutes	15 minutes
	Dry to Tape	12 Hours				
	Full Cure	14 Days				
Green Polyurethane		21 Celsius				
	Complete	1			1	
	Cure	4 Days				
Dupont		25 Celsius		120-140 F(48	-60C)	
HG 1.2	Tack Free	20-30 minutes				
1.0 1.2						
	Dry to recoat	30 minutes				
		30 minutes 2 Hours		15-20 Minute	es	
Industual Strength	Dry to recoat			15-20 Minute	es	
	Dry to recoat Hard Dry	2 Hours		15-20 Minute	25	
	Dry to recoat Hard Dry To Touch	2 Hours 3 Hours		15-20 Minute	25	
	Dry to recoat Hard Dry To Touch Tack Free	2 Hours 3 Hours 3 Hours		15-20 Minute	25	
	Dry to recoat Hard Dry To Touch Tack Free To Handle	2 Hours 3 Hours 3 Hours 4.5 hours		15-20 Minute	2S	
	Dry to recoat Hard Dry To Touch Tack Free To Handle Hard Dry	2 Hours 3 Hours 3 Hours 4.5 hours 18 hours		15-20 Minute	25 	
Industual Strength	Dry to recoat Hard Dry To Touch Tack Free To Handle Hard Dry	2 Hours 3 Hours 3 Hours 4.5 hours 18 hours 7 Days		15-20 Minute	25 	
Industual Strength	Dry to recoat Hard Dry To Touch Tack Free To Handle Hard Dry Full Cure	2 Hours 3 Hours 3 Hours 4.5 hours 18 hours 7 Days 25 Celsius		15-20 Minute	25 	
Industual Strength	Dry to recoat Hard Dry To Touch Tack Free To Handle Hard Dry Full Cure	2 Hours 3 Hours 3 Hours 4.5 hours 18 hours 7 Days 25 Celsius 50 minutes		15-20 Minute	25 	

### Appendix E

### Phase 1 Test Results

### American Marking JS and WJ series Before and After Test



20130924_08543B	20130924_08544B	20130924_085504	20130924_085515	20130924_085528
20130924_085537	20130924_085555	20130924_085610	20130924_085629	20130924_08564D
20130924_085718	20130924_085720	20130924_085726	20130924_085741	a) 23 a) a) 29 a) 29 a) 29 a) 49 a) 49 a) 10 a) 40 a) 30 a) a) 40 a) 40 a) 10 a) 40 a) 10 a) 10 a) 10 a) 40 a) 10 a) 10 a) 10 a) 10 a) 10 a) 10 a) 10 a) 10 a) 10 a) 10 a) 10 a) 10 a) 10 a) 10 a) 10 a) 10 a) 10 a) 10 a) 10 a) 10
аз ат ы не ад и ат еги не ал и на т от еги а от ал и и 20130924_085752	23 48 21 29 43 21 21 29 49 29 20 20 21 24 24 27 29 49 20 20 20130924_085754	20130924_085827	20130924_085831	285 287 287 287 288 287 288 287 289 289 289 289 289 289 289 289 289 289
48. 68 63 48 48 49 49 48 48 40 40 40 40 52 21 26 46 46 47 16 46 46 49 40 20130924 085848		23 * * * * * * * * * * * * * * * * * * *	ала ала ала ала ала ала ала ала ала ала ала ала	20130924 085924
20130924_085926		ин ин жаласана ала и ил и ил ил ил ил ил ил 20130924_085943	az an ar	28 - 31 - 32 - 34 - 34 - 34 - 34 - 34 - 34 - 34
20130924_090012	20130924_090044	20130924_090051	20130924_090105	

# DEFT, Akzonobel Spray2Fix, Sherwin William Photo 9 to next page

20130924_090120	ан раза и стана раза на раза на 20130924_090129	20130924_090131	20130924_090150	20130924_090157
	JII 30 & 3 D & 4 A A A A A A A A A A A A A A A A A A	41 49 4 5 7 8 5 3 8 4 4 46 22 80 80 98 98 47 46 45 44 35 46 14 99 49 49 48 47 46 45 44 35 46 20130924_090231	рание вология 20130924_090300	ания Энина 20130924_090301
20130924_090306	20130924_090324	20130924_090329	117 41 45 29 46 47 46 45 41 25 47 35 47 12 21 25 46 46 47 46 48 46 46 46 47 MATERIAL PN	27 28 29 39 38 37 38 37 38 38 21 39 39 39 39 39 39 39 39 39 39 39 39 39
20130924_090351	ени и на на на на на на на на на на на на на 20130924_090353	20130924_084837		

	65 87 85 95 95 87 87 95 97 97 97 97 97 97 107 92 18 19 19 19 19 19 19 19 19 19 19 19 19 10 10 10 10 10 10 10 10 10 10 10 10 10 1	20140226_124922	п ж а желя п и и и и и и и и а в и и и и и и и 20140226_124923	20140226_124927
20140226_124928	20140226_124942	20140226_124943	20140226_124950	20140226_124951
20140226_124959	20140226_125000	и и и ла да на на на на на матениа и ри 20140226_125018	RA dH dB dD dA dA dH dH dB dD dA RATERAL PN	20140226_125023
а ан ал ан ан ан ан ан 20140226_125024		20140226_125033		
		20140226_125113		

# Union Ink and Green Polyurethane



### DuPont 1.2 HG, DuPont IS, Aerofine before

# DuPont 1.2 HG, DuPont IS, Aerofine After

20140113_113629	201 An an 19 19 An an an an an an an an an an 20140113_113634	20140113_113648	51 7000007 H D D D D D D D D D D D D D D D D D D D	20140113_113703
201 201 201 201 201 201 201 201 201 201	20140113_113716	20140113_113723	20140113_113256	20140113_113334
20140113_113337	20140113_113351	2 300,123,30 1 30 30 30 30 30 30 30 30 30 30 30 30 30 31 30 30 30 30 30 30 30 30 30 30 30 31 31 30 30 30 30 30 30 30 30 30 30 30 30 30 30 30 30 30 30 30 30 30 30 30 30 3	20140113_113419	20140113_113444
20140113_113505	мателиц ри В рие тиеат 20140113_113520	100 40 40 10 10 10 10 10 10 10 10 10 10 10 10 10	20140113_113544	ан 31 ай и и и и и и и и и и и и И и и и и и и и
20140113_113604	атал жала ал жала на на на мала на на кала на на на на 20140113_113610			

# Appendix F

## Phase 2 Test Results

### Before

20131114_090834	20131114_090847	20131114_090912	20131114_09091B	20131114_090926
20131114_090932	20131114_090936	20131114_090946	20131118_120435	20131118_120508
20131118_120519	20131118_120524	20131118_120530	20131118_120536	20131118_120544
20131118_120552	20131118_120559	20131118_120608	201311118_120614	20131118_120622
20131118_120633	20131118_120645	20131118_120652	20131118_120659	20131118_120704
20131118_120718	20131118_120723	20131118_120729	20131118_120737	20131118_120745
	10100000000000000000000000000000000000			

20131118\_120755

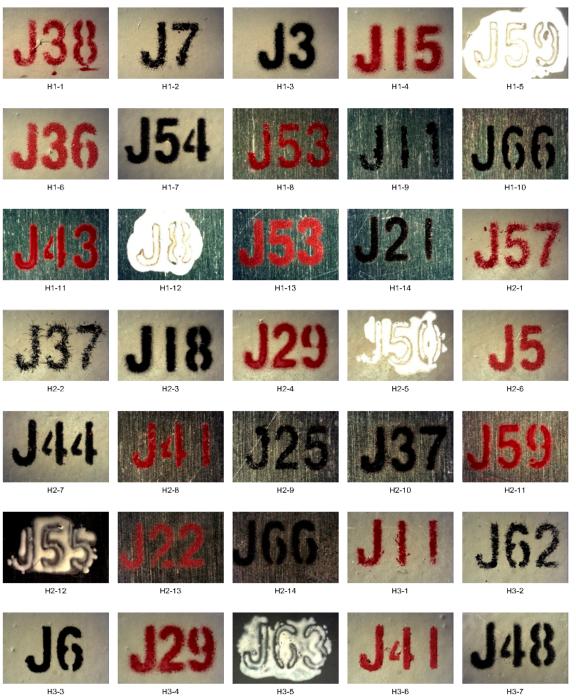
<sup>20131118</sup>\_120800

20131118_120807	20131118_131530	20131105_115017	20131105_115249	20131105_125627
20131105_125631	20131105_125633	20131105_125635	20131114_090414	20131114_090423
20131114_090428	20131114_090432	20131114_090442	20131114_090449	20131114_090454
20131114_090500	20131114_090505	20131114_090523	20131114_090525	20131114_090535
20131114_090545	20131114_090549	20131114_090559	20131114_090604	20131114_090615
20131114_090617	20131114_090626	20131114_090645	20131114_090646	20131114_090737
20131114_090758	20131114_090811	20131114_090819	20131114_090825	20131114_090827

# 10X Stereoscope After Images

*Table 16 – Key to Images, Each Prefix indicates type of paint and suffix indicates panel number.* 

H x - y	Description	L x - y	Description
Н	Cleaning System Test	L	Humidity Test
X	Panel Number	X	Panel Number
У		y:	
y-1	Union Ink	y-1	DuPont IS
y-2	Union Ink	y-2	SW
y-3	DEFT	y-3	Union Ink
y-4	DEFT	y-4	DEFT
y-5	Green Poly	y-5	Green Poly
y-6	SW	y-6	DuPont IS
y-7	SW	y-7	SW
y-8	Union Ink	y-8	Union Ink
y-9	Union Ink	y-9	DEFT
y-10	DEFT	y-10	Green Poly
y-11	DEFT		
y-12	Green Poly		
y-13	SW		
y-14	SW		



H3-3

H3-4

H3-5

H3-7



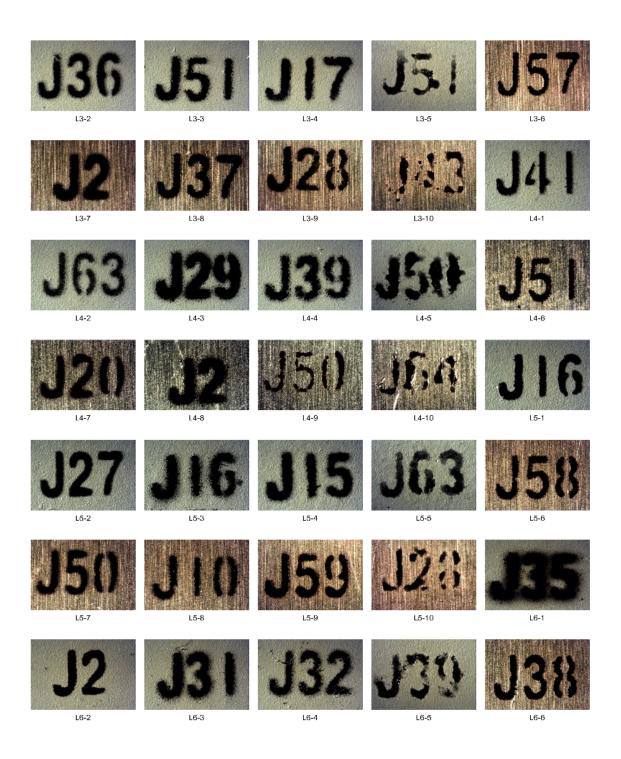
H5-10

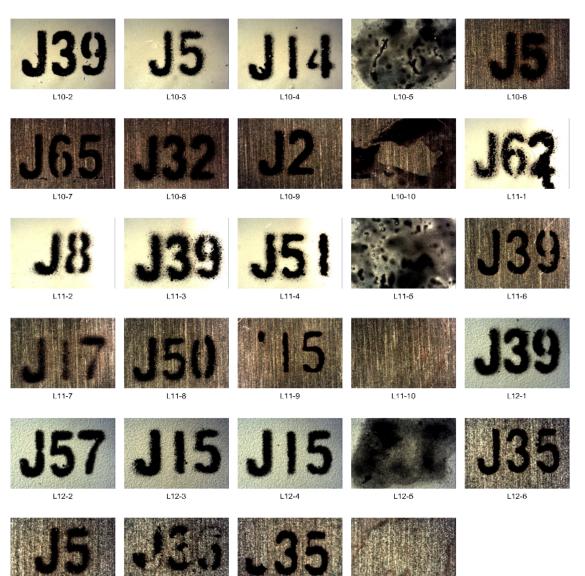
H5-11

H5-12

H5-14







L12-7

L12-8

L12-9

L12-10