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United States
Environmental Protection
Agency

Development of Standard Test Methods and Evaluation of the Shelf Life and Weatherability of Fixative Coatings for Control of Radiological Contamination

Office of Research and Development
National Homeland Security Research Center

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November 2013

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

RESEARCH TRIANGLE PARK, NC 27711

DISCLAIMER

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ARCADIS-US

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Acronyms and Abbreviations

ASTM	American Society for Testing and Materials
CBRN	chemical, biological, radiological, nuclear
cm	centimeter(s)
CMAT	Consequence Management Advisory Team
COTS	commercial-off-the-shelf
cps	centipoises(s)
DOE	U.S. Department of Energy
DQI	Data Quality Indicator
EPA	U.S. Environmental Protection Agency
gal	gallon(s)
g	gram(s)
h	hour(s)
HSRP	Homeland Security Research Program
kg	kilogram(s)
KU	Krebs Unit(s)
lb	pound(s)
MIL-PRF	Military Performance Standard
Mg	milligram(s)
N/A	not available
NHSRC	National Homeland Security Research Center
OEM	Office of Emergency Management
ORD	Office of Research and Development
psi	pounds per square inch
QA	quality assurance
QAPP	quality assurance project plan
QC	quality control
QMP	Quality Management Plan
RH	relative humidity
rpm	revolutions per minute
RSD	relative standard deviation
UV	ultra violet

Executive Summary

The U.S. Environmental Protection Agency's (EPA's) Homeland Security Research Program (HSRP) is helping to protect human health and the environment from adverse impacts resulting from Chemical, Biological, Radiological and Nuclear (CBRN) contamination whether it results from an intentional act (for instance, terrorism), a criminal act, or an unintentional act (such as a natural disaster or industrial accident). One way HSRP helps to protect human health and the environment is by developing performance requirements and test methods for evaluating the performance of technologies relevant to homeland security. Through its involvement with the American Society for Testing and Materials (ASTM) the HSRP led the effort which developed ASTM E-2731-09 Standard Specification for Materials to Mitigate the Spread of Radioactive Contamination after a Radiological Dispersion Event. ASTM E-2731-09 articulates eighteen different material performance requirements (e.g., tear strength, abrasion resistance, weatherability, shelf life, etc.) for coatings which could be used to sequester contamination (particles) and fix them in place, thereby limiting the spread of contamination (and associated health/exposure impacts) and making future decontamination and recovery efforts more effective. Test methods for verifying the performance of any particular coating against the requirements in ASTM E-2731-09 exist for some parameters such as tensile strength or abrasion resistance. However, test methods for some of these requirements do not currently exist, including methods for assessment of weatherability and shelf life. ASTM E-2731-09 defines weatherability as the capability of the coating to remain stable (maintain film integrity) under a variety of outdoor environmental conditions for a minimum of one year. The environmental conditions specified in ASTM E-2731-09 include exposure to ultraviolet (UV) light, water immersion, high and low temperatures, and common bacteria. Similarly, the performance requirement for shelf life given in ASTM E-2731-09 is defined as the ability of the coating to be applied successfully and to meet the performance requirements as otherwise specified in the standard, after a specified period of climate-controlled storage (five years per ASTM E-2731-09).

The EPA HSRP, led by the EPA's National Homeland Security Research Center (NHSRC), has produced a set of test methods to verify performance of coatings intended to meet the ASTM E-2731-09 performance standards for shelf life and weatherability. These test methods were drafted, verified by laboratory exercise using a commercially available coating product widely used in the radiological industry, ALARA 1146 (Carboline Co., St. Louis, MO, USA), and then used to evaluate the shelf life and weatherability performance of three additional currently commercially available coatings selected as potential candidates for use as sequestration coatings to immobilize radioactive contamination. Selection of the three coatings to be tested was based primarily on material availability, reasonable deployment requirements, and past EPA experience. The three products selected included the two systems (a) CC-STRIP followed by CC Fix (Instacote, Erie, MI, USA); (b) CC Strip followed by SBR-10F (Instacote, Erie, MI, USA); and (c) Intergard 10220 (AkzoNobel, Strongsville, OH, USA).

1.0 Introduction

Research indicates that if a radioactive dispersal device (dirty bomb) is detonated, immediate action may be necessary to treat urban surfaces. For some radioactive particles migration and chemical binding are rapid, and after some period of time, significant decontamination may not be feasible. Thus, immediate treatment of surfaces (within hours) with a sequestrating compound that can be applied easily in large quantities could alleviate much difficulty in subsequent decontamination. Additionally, loose radioactive particles may be further dispersed into the environment (blown by wind or washed by rain), making decontamination even more challenging. The use of sequestrating compounds could immobilize these particles preventing their dispersion into the environment and increasing the effectiveness of subsequent decontamination efforts. One type of sequestration compound considered for deployment in the aftermath of a dirty bomb event would consist of a coating that would likely be a polymeric, paint-like substance. Such a coating would be capable of encapsulating and immobilizing radioactive particles deposited on outdoor construction surfaces such as steel, concrete, or asphalt.

Performance specifications for such a sequestration coating were previously developed and promulgated as ASTM E-2731-09 Standard Specification for Materials to Mitigate the Spread of Radioactive Contamination after a Radiological Dispersion Event. ASTM E-2731-09 articulates eighteen different performance requirements (e.g., tear strength, abrasion resistance, weatherability, shelf life, etc.). Test methods for verifying the performance of any particular coating against these requirements exist for some parameters such as tensile strength or abrasion resistance. However, test methods for some of these requirements do not currently exist, for example, methods for assessment of weatherability and shelf life. ASTM E-2731-09 defines weatherability as the capability of the coating to remain stable (maintain film integrity) under a variety of outdoor environmental conditions for a minimum of one year. The expectation is that the coating will maintain its mechanical properties during that exposure period. The environmental conditions specified in ASTM E-2731-09 include exposure to ultraviolet (UV) light, water immersion, high and low temperatures, and common bacteria. Similarly, the performance requirement for shelf life given in ASTM E-2731-09 is defined as the ability of the coating to be applied successfully and to meet the performance requirements as otherwise specified in the standard, after a specified period of climate-controlled storage (five years per ASTM E-2731-09).

The Environmental Protection Agency's (EPA) Homeland Security Research Program (HSRP), led by the EPA's National Homeland Security Research Center (NHSRC), has produced a set of test methods to verify performance of coatings intended to meet the ASTM E-2731-09 performance standards for shelf life and weatherability. These test methods were drafted, verified by laboratory exercise, and then used to evaluate the shelf life and weatherability performance of three commercially available coatings selected as potential candidates for use as sequestration coatings to immobilize radioactive contamination.

2.0 Literature and industry search

An extensive search of available literature and industrial sources was undertaken to identify potential coating test methods applicable to weatherability and shelf life, and to identify candidate sequestration coatings that could be used for verification of the test methods being developed. The material standards community and the coatings industry were canvassed to determine how industry evaluates coating weatherability and shelf life. Multiple vendors were contacted based on the literature search. Not unexpectedly, the majority of the manufacturers contacted were focused primarily on marketing and sales issues and potential sales volume. Technical information, especially with regards to weatherability and shelf life, was sparse to non-existent. Most manufacturers did not have a method for evaluating shelf life other than leaving the coating on a shelf (i.e., in storage) for some period of time and testing either the viability of the coating or the viability of the chemical components of their products. In addition, only very limited information was available on the ability of any of the commercial coatings to withstand various weather-related elements. Limited testing or performance data were obtained during this search process. Table 2-1 lists sequestration coatings that have been tested in the past where results have been reported in publicly available literature.

Several potential candidate coatings were identified as a result of the literature and industrial sources search and are listed in Table 2-2. Table 2-2 is intended to illustrate only the availability of the potential sequestration coatings found during the search and is not intended as a complete list of coatings that may be offered by the industry.

One coating, ALARA 1146 (Carboline Co., St. Louis, MO, USA), was initially selected for verification of the test methods derived during this project. Verification of an analytical procedure is the demonstration that a laboratory is capable of replicating, with an acceptable level of performance, a standard method. Following further consideration and discussions with various coatings manufacturers, three additional coatings were selected for further testing using the verified test methods. Selection was based primarily on material availability, deployment requirements, and past EPA experience. Two multiple-component coating systems were selected from Instacote, Inc. (Erie, MI, USA), and one from AkzoNobel (Strongsville, OH, USA). The Instacote coating systems require a two-step application protocol. Although a two-step application was not considered most desirable from a field deployment perspective, these coating systems were selected due to their reported durable nature and removability. The three products selected included the Instacote systems (a) CC-STRIP followed by CC Fix and (b) Instacote CC Strip followed by SBR-10F; and (c) AkzoNobel's Intergard 10220 (all three in green in Table 2-2).

Table 2-1. Previously tested sequestration coatings

Product Name Manufacturer	Literature Source
ALARA 1146 Carboline Co. (St. Louis, MO, USA)	Cleaning and Decontamination using Strippable and Protective Coatings at the Idaho National Engineering and Environmental Laboratory, INEEL/CON-98-00797 ALARA™ 1146 Strippable Coating, DOE OST/TMS ID 2314, April 2000
Bartlett TLC Bartlett Services, Inc. (Plymouth, MA, USA)	Cleaning and Decontamination using Strippable and Protective Coatings at the Idaho National Engineering and Environmental Laboratory, INEEL/CON-98-00797
Bartlett TLC Free Bartlett Services, Inc. (Plymouth, MA, USA)	Assessment of Strippable Coatings for Decontamination and Decommissioning, DOE/EW/55094—32, January 1998 Bartlett Services Inc. Stripcoat TLC Free Radiological Contamination Strippable Coating, EPA/600/R-08/099, September 2008
InstaCote ML Instacote (Erie, MI, USA)	Assessment of Strippable Coatings for Decontamination and Decommissioning, DOE/EW/55094—32, January 1998
Orion Isotron Corp. (Seattle, WA, USA)	Isotron Corp. Orion Radiological Decontamination Strippable Coating, EPA/600/R-08/100, September 2008
Tech Sol 8001 Technical Solutions & Systems, Inc. (Elizabethton, TN, USA)	Assessment of Strippable Coatings for Decontamination and Decommissioning, DOE/EW/55094—32, January 1998
Tech Sol 8002 Technical Solutions & Systems, Inc. (Elizabethton, TN, USA)	Assessment of Strippable Coatings for Decontamination and Decommissioning, DOE/EW/55094—32, January 1998

Table 2-2. Selection of sequestration coatings identified as potential candidates for method verification

Coating	Company	Web Address	Application	Advertised Shelf Life	Notes
ORION	Isotron Corporation Seattle, WA, USA	www.isotron.net	Brush, airless spray, roller	10 months at 75 °F	Produced in Japan under trade name "Extract"
Stripcoat TLC	Bartlett Services, Inc Plymouth, MA, USA	www.bartlettnuclear.com	Spray	7 to 30 days	Not applicable in subfreezing temperatures (water-based)
ALARA 1146	Carboline St. Louis, MO, USA	www.carboline.com	Spray recommended	n/a	Licensed for production in Japan by Carboline Water-based
RADBLOCK	Isotron Corporation Seattle, WA, USA	www.isotron.net	---	10 months at 75 °F	Coming soon (website checked 8/13/12) Gemini. Electrosorb, and WaterDecon
CC-STRIP	InstaCote Erie, MI, USA	www.instacote.com/cc-strip.htm	Sprayers and rollers	n/a	Does not penetrate pores; CC Wet is required as the first step, CC Wet is water-based Other products: CC Wet, CC Fix, CC Fix LV, CC Epoxy SP Only CC Strip is strippable
Spray Poly	Foster Specialty Construction Brands, Inc St. Aurora, IL, USA	www.fosterproducts.com	---	6 months	UV sensitive: used for asbestos remediation
Multisurface	Protectapeel Corby, Northamptonshire. NN174AR, UK	www.protecapeel.co.uk	Airless gun or roller	12 months between 5-25 °C	Not tested for radiological contamination Other products: SC1074, Sc1090, E105NF, E106
Proprietary Process	Salvarem-The Nuvia Group Z.A. Beaumont CEDEX-France	www.nuvia-india.com	Various surfaces, wet application	n/a	Nuvia Group France Three processes: FORAL, Forac, FORNET for radioactive cleanup
Contrad 70	Decon Labs, Inc. Prussia, PA, USA	www.deconlabs.com	Wet rinse	n/a	Also Decon 90 surface wash
RDS 2000	Kärcher Futuretech GmbH Schwaikheim Germany	www.rkb.us	n/a	n/a	Two component liquid agent, foaming unit required , foam applied to surfaces
KATS 9500 OPC	Kats Coatings Rockwall, TX, USA	www.katscoatings.com	Sprayers	n/a	Non-porous surfaces, Not tested for radiological contamination, three products: 9501, 9502, 9503
Intergard 10220	AkzoNobel Sassenheim, The Netherlands	http://www.akzonobel.com	n/a	n/a	Water-based polyurethane Cooperation with DSTL (UK) for absorptive coating

Red Shading – Shelf life inadequate
Purple Shading – Application conditions unacceptable
Green Shading – Selected for testing

3.0 Review of existing test methods

To determine if there were any existing test methods that could potentially be applicable to weatherability and shelf life determination, an extensive review of industry (primarily ASTM) methods and military specifications was conducted, focused on paints and coatings. Initial results indicated that potential candidates would likely be polymeric, paint-like substances. The review was focused on identifying methods used for paint testing that might be useful to evaluate shelf life and weatherability of sequestration coatings. In addition to shelf life and weatherability, ASTM E2731–09 specifies the following physical/mechanical parameters for a sequestration coating:

- Tensile Strength 35 kg/cm^2 (500 psi)
- Adhesion >3.5 kg/cm^2 (>50 psi) on concrete
- Abrasion <50-mg (0.002-oz) loss
- Tear Strength 14 kg/cm^2 (200 psi)
- Flammability - does not burn.

ASTM E2731–09 specifies a required shelf life of a minimum of five years. In addition, the coating must meet the above physical parameters for a period of one year under weathering conditions described as exposure to a variety of outdoor environmental conditions such as ultraviolet (UV) exposure, precipitation, high and low temperatures, and common bacteria.

It became apparent that performance in terms of weatherability and shelf life are inextricably tied to the measurable physical/mechanical requirements such as those listed above. Essentially, to evaluate weatherability performance, the coating must continue to exhibit its physical/mechanical attributes after being exposed to the weathering environment. Similarly, to evaluate the ability of a coating to meet the shelf life requirement, the coating must, after storage for the specified five-year period, continue to exhibit these same physical/mechanical attributes.

While some test methods require removal of the coating from the test substrate, other methods require that testing to be performed on the test substrate. In addition, both accelerated and non-accelerated test methods were used to identify potential degradation of a coating.

4.0 Test method development

The shelf life and the weatherability test methods were produced by incorporating selected portions of existing coating and paint test methods with some modification to suit the particularities of a capture coating. Table 4-1 provides a list of six properties initially selected for evaluating the potential shelf life of a coating. Table 4-1 lists a preferred method as well as alternate methods, if available. Table 4-2 provides a list of five properties initially selected for evaluating the potential weatherability of a coating.

Table 4-1. Existing test methods selected for incorporation into the shelf life test method

Property Describing Shelf Life	Preferred Method	Alternate Methods
Freeze/Thaw Resistance of Organic Coatings	ASTM D2337/ MIL-PRF-32239	N/A
Settling Properties	ASTM D1309/869	N/A
Freeze/Thaw Resistance of Aqueous-Based Coatings	ASTM D2243	N/A
Accelerated Storage Stability	MIL-PRF-32239	N/A
Density	ASTM D1475	N/A
Viscosity	ASTM D1200	ASTM D4287; ASTM D7395

N/A = not applicable

Table 4-2. Existing test methods selected for incorporation into the weatherability test method

Property Describing Weatherability	Preferred Method	Alternate Methods
Water resistance at 100% RH	ASTM D2247-02	ASTM D4585-07
Tensile strength	ASTM D2370-98 (organic-based coatings) ASTM D6083 - 05e1 (water-based coatings)	N/A
Abrasion resistance	ASTM D4060-07	ASTM D968[a]
Natural sunlight and soak-freeze-thaw tolerance	ASTM D5722-08	ASTM D660 and/or D662 and/or D714 and/or D772 and/or D4214 and/or D610[b] ASTM D6695-08 or MIL-PRF-32239[c]
Adhesion strength (on concrete)	ASTM D7234-05	ASTM D4541 - 09e1[d]

N/A= Not Applicable

[a] For organic coatings only

[b] As a follow up to D5722-08; not meant to replace D5722-08

[c] Only for the concentrated natural light part of D5722-08

[d] Only for surfaces other than concrete

For the purpose of developing the shelf life test protocol, successful weatherability performance was not considered to be a prerequisite. Likewise, weatherability of a coating can be tested whether or not the coating meets the shelf life requirements.

The two draft test methods were subjected to an internal peer review process, performed by subject matter experts in related fields, as well as personnel with experience in the field of materials testing and protocol development. Comments received from the review were addressed and the final methods developed prior to method verification. The final test methods were submitted for verification to an outside testing subcontractor (KTA-Tator Laboratories, Pittsburgh, PA, USA) with the requirement that they be strictly followed, to capture any weaknesses or errors. Communications were maintained between the test crew and the procedure writing crew during the entire verification process to assure communication of errors/omissions and changes. In addition, two visits were made to the laboratory to observe the testing of the coatings in progress and to facilitate further refinement of the test methods.

5.0 Verification of draft test methods

One containment coating, ALARA 1146, was selected for verification of the shelf life and weatherability test methods. The tests identified in Tables 4-1 and 4-2 were performed using this coating. ALARA 1146 was applied according to manufacturer's recommended procedures to both concrete and steel coupons for the purposes of method verification. These coupons were specifically manufactured to replicate two types of building materials commonly found in the urban environment. The coupons were clean and were not contaminated. As a result of these verification tests and observations, various changes were required for both test methods, as shown in Table 5-1. Final modified methods are shown in Table 5-2. Following method verification, both test methods were finalized and are incorporated into this report as Appendix A, Shelf Life Test Method and Appendix B, Weatherability Test Method. The results of the verification tests using ALARA 1146 are presented in Appendix C.

Table 5-1. Changes made following verification testing

<table>
<tr><th></th><th>Method Changed</th><th>Property</th><th>Method</th><th>Reason for Change</th></tr>
<tr><td rowspan="2">1</td><td rowspan="2">Shelf Life Method</td><td>Freeze/Thaw Resistance of Organic Coatings</td><td>D2337</td><td rowspan="2">These methods are essentially identical (one hour difference in cycle duration). Method D2243 for aqueous coatings has been deleted, and Method D2337 will be used for all coatings.</td></tr>
<tr><td>Freeze/Thaw Resistance of Aqueous-Based Coatings</td><td>D2243</td></tr>
<tr><td>2</td><td>Shelf Life Method</td><td>Viscosity</td><td>D1200
D2196</td><td>Method 1200 requires use of a #4 Ford cup, and the coating would not flow through this device. This method was replaced with Method D2196, which uses a Brookfield viscometer.</td></tr>
<tr><td>3</td><td>Weatherability Method</td><td>Water Resistance at 100% Relative Humidity (RH)</td><td>D2247</td><td>This method evaluates durability of a coating to water degradation at 100% RH. We had specified duration of 300 hours. However, the laboratory noted degradation by 200 hours. The required duration was changed to 200 hours for future tests.</td></tr>
</table>

Table 5-2. Final shelf life and weatherability methods

Property Describing Shelf Life	Method Selected
Freeze/Thaw Resistance of Organic Coatings	ASTM D2337
Settling Properties	ASTM D1309/869
Accelerated Storage Stability	MIL-PRF-32239
Density	ASTM D1475
Viscosity	ASTM D2196
Property Describing Weatherability	**Method Selected**
Water resistance	D2247-02 [a]
Tensile strength	D2370-98 (organic-based coatings) D6083 - 05e1 (water-based coatings)
Abrasion resistance	D4060-07
Natural sunlight and soak-freeze-thaw tolerance	D5722-08
Adhesion strength	D7234-05

[a] modified per Table 5-1.

6.0 Tests of additional coatings using the verified test methods

Three additional coatings were identified and tested using the verified test methods. The coatings identified and tested included (1) Instacote CC Strip (base coat) followed by CC Fix, (2) CC Strip (base coat) followed by SBR-10F, and (3) AkzoNobel Intergard 10220. All three coatings were applied to both concrete and steel coupons and allowed to cure for seven days prior to testing. The application of the coatings, as well as all testing was conducted at ambient laboratory temperature and humidity levels.

The Instacote coatings were used as two-part coatings due to the durability of the CC Fix and SBR-10F (as top coat) and the removability of CC Strip (as base coat). CC Strip is a flexible, strippable (peelable), protective coating used in various industries. This combination of applying a durable coating on top of a strippable coating was suggested to achieve the removability characteristics desired in these candidate coatings, as well as the durability of the coating surface. After simple laboratory tests at Instacote, Inc., the manufacturer reported that the coating system was still both strippable and durable.

The base coats of CC Strip were applied to the metal and concrete coupons in a horizontal orientation and allowed to cure. The recommended cure time for CC Strip is 24 hours at standard temperature and humidity. Higher humidity will require a longer cure time. The coupons and panels were allowed to dry for a minimum of 24 hours before application of the top coats. After curing, the CC Fix and SBR-10F top coats were applied to the coupons and allowed to dry. The Instacote coatings were brushed on (per the manufacturer) using a commercial-off-the-shelf (COTS) paint brush for both the base coat and the durable top coat.

After curing, both Instacote systems appeared to cover the substrate completely and were smooth and well-flowed, despite the base coat of CC Strip having some slight imperfections or ridges. The Instacote SBR-10 is a two-part resin and isocyanate with solvent. When mixed together and allowed to stand, this combination appeared to produce a slightly exothermic reaction. The pot life for SBR-10 is approximately 10 minutes with a cure time of 1.5 hours. Drying time increases with cooler temperatures.

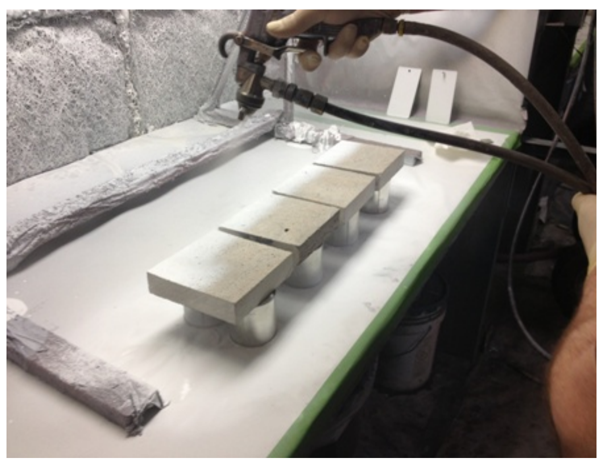

Figure 6-1. Intergard 10220 being applied to concrete coupons

The Intergard 10220 was applied by spraying the coating (per the manufacturer) onto the coupons in a horizontal orientation. Intergard 10220 required a 20% dilution with de-ionized (DI) water to facilitate spraying. The appearance of the cured Intergard 10220 was as a smooth, well-flowed liquid on both the metal and concrete coupons. Some small air bubbles appeared on the concrete coupons as the coating was curing. To assure a more uniform coating these bubbles were manually deflated. The cured Intergard coated coupons showed minor imperfections. In addition, the Intergard metal panels showed micro-cracking at the voids that remained from the bubbles in the film. AkzoNobel was willing to provide only very limited information on the characteristics and previous uses of Intergard 10220, citing as reason that it was originally developed for the U.S. military.

The detailed test results for Instacote CC Strip/CC Fix are presented in Appendix D, for Instacote CC Strip/SBR-10F in Appendix E, and for Intergard 10220 in Appendix F.

7.0 Quality Assurance and Quality Control (QA/QC)

This project was performed according to an approved Quality Assurance Project Plan (QAPP) titled *Identification and Development of Standard Methods for Evaluation of the Performance of Sequestration Coatings (August 2012)*. The QAPP was developed according to the NHSRC Quality Management Plan (QMP).The objective as stated in the QAPP was to verify the test methods for shelf life and weatherability and test three commercial coatings for shelf life and weatherability performance. Development of the test methods assumed the following efforts would be required:

(a) Revision of the draft test protocols for clarity, omissions, etc., which might occur before and/or after the testing.

(b) Potential revisions to the specific methods specified within each draft test protocol, based on laboratory feedback and how well the accuracy, precision, and completeness criteria would be met.

The QA/QC checks described in this section pertain to step (b) above and include quantitative acceptance criteria for shelf life and weatherability. These critical measurements are summarized in Section 7.1 and discussed in detail in Appendix G. The acceptance criteria for determining whether or not they meet the QA objectives of this study are summarized in Section 7.2 and discussed in detail in Appendix G.

7.1 Quantitative acceptance criteria for QA objectives

The test results will be used to identify any deficiencies of a tested containment coating to be applied as intended and will establish and verify the ability of a coating to possess successful shelf life and weatherability performance. Tables 7-1 and 7-2 provide the critical measurement properties for testing the shelf life and weatherability of a coating, respectively.

Table 7-1. Critical measurement properties for testing of shelf life

Property Describing Shelf Life	Preferred Method	Alternate Methods
Freeze/Thaw Resistance of Organic Coatings	ASTM D2337	N/A
Settling Properties	ASTM D1309 and ASTM D869	N/A
Accelerated Storage Stability	MIL-PRF-32239	N/A
Density	ASTM D1475	N/A
Viscosity	ASTM D2196	ASTM D4287 or ASTM D7395

N/A=Not Applicable

Table 7-2. Critical measurement properties for testing of weatherability

Property Describing Weatherability	Preferred Method	Alternate Methods
Water resistance	ASTM D2247-02	ASTM D4585-07
Tensile strength	ASTM D2370-98 (organic-based coatings) ASTM D6083-05e1 (water-based coatings)	N/A
Abrasion resistance	ASTM D4060-07	ASTM D968[a]
Natural sunlight and soak-freeze-thaw tolerance	ASTM D5722-08	ASTM D660 and/or D662 and/or D714 and/or D772 and/or D4214 and/or D610[b] ASTM D6695-08 or MIL-PRF-32239[c]
Adhesion strength	ASTM D7234-05	ASTM D4541 - 09e1[d]

N/A = Not Applicable

[a] For organic coatings only.

[b] As a follow up to D5722-08; not meant to replace D5722-08.

[c] Only for the concentrated natural light part of D5722-08; MIL-PRF = Military Performance Standard

[d] For surfaces other than concrete.

7.2 Data Quality Indicators

Data quality indicators (DQIs) for the critical measurements were used to determine if the collected data met the QA objectives. The mean value of at least four replicate tests was used to assess pass/fail for that portion of the test. A list of the DQIs is given in Table 7-3. The DQIs established in the QAPP were selected without specific knowledge of the coatings and their characteristics. One objective of the verification of the test methods was to verify that these were realistic goals for the coatings being tested.

The equipment used to make the critical measurements was calibrated as per the requirements of the respective ASTM methods.

7.3 Assessment of DQI Goals

The representativeness of the experiments was ensured by the careful selection of the concrete and steel coupons and experimental conditions. The parameters that were used to assess whether the data met the quality assurance objectives (as detailed in Table 7-3) include precision, accuracy, and completeness of the collected data. A detailed discussion of these measures is included in Appendix G.

Table 7-3. DQIs for critical measurements

Measurement Parameter	Test Method	Pass Value	Accuracy	Precision/ Repeatability	Completeness %
Shelf Life					
Freeze/Thaw Resistance of Organic Coatings	ASTM D2337	Less than 10% difference in viscosity	±15%	±20%	90
Settling Properties	ASTM D1309 or ASTM 869	At least 8 on the scale from 10 to 0*	N/A	N/A	90
Accelerated Storage Stability	MIL-PRF-32239	Less than 10% difference in viscosity and density compared to before accelerated test	±15%	±20%	90
Density	ASTM D1475	Less than 10% difference	±15%	±20%	90
Viscosity	ASTM D2196	Less than 10% difference	±15%	±20%	90
Weatherability					
Water resistance	ASTM D2247-02	<10% adhesion loss	N/A	N/A	90
Tensile Strength	ASTM D2370-98 (organic-based coatings) ASTM D6083 - 05e1 (water-based coatings)	>35 kg/cm^2 (kilograms/centimeter squared) (500 psi)	±15%	±20%	90
Abrasion resistance	ASTM D4060-07	<10% weight loss	±15%	±20%	90
Natural sunlight and soak-freeze-thaw tolerance	ASTM D5722-08	Note surface blemishes	N/A	N/A	90
Adhesion strength	ASTM D7234-05	>3.5 kg/cm^2 (>50 psi)	±15%	±20%	90

* “8” signifies a definite feel of settling; no resistance to sidewise movement. This is a very subjective test and the ratings are listed as word expressions; there are no units.

N/A=Not Applicable

8.0 Summary of test data

8.1 Summary

Each of the four coatings was subjected to the individual test methods recommended for each material property within both the shelf life and weatherability test methods. These tests were performed by KTA-Tator Laboratories. The data resulting from each of these tests are shown in the tables within each of the following sections.

8.2 Adhesion

Adhesion (pull-off strength) was measured in accordance with ASTM D7234, “Standard Test Method for Pull-Off Adhesion Strength of Coatings on Concrete Using Portable Pull-Off Adhesion Testers”, or ASTM D4541, “Pull-Off Strength of Coatings Using Portable Adhesion Testers”, Annex A4, “Self-Aligning Adhesion Tester Type IV”. Per the shelf life test method, either adhesion test method is acceptable. However ASTM D7234 is listed as the preferred method. For verification purposes since both methods are listed as acceptable, both methods were exercised. Both methods use the same equipment to remove the pull stubs and the results were reported as pounds per square inch (psi). However, the size of the pull stub differs. ASTM D7234 for concrete specifies a 2” pull stub. Coatings on the steel panels were evaluated per ASTM 4541 using a ½” pull stub. Both of these methods include initial performance under nominal laboratory conditions of temperature and humidity (72 ± 5 °F and 50 ± 5 %RH) and under exposure to high humidity conditions (72 ± 5 °F and 95 ± 5 %RH) for 200 hours. Figure 8-1 shows a concrete coupon and pull stubs following the adhesion test.

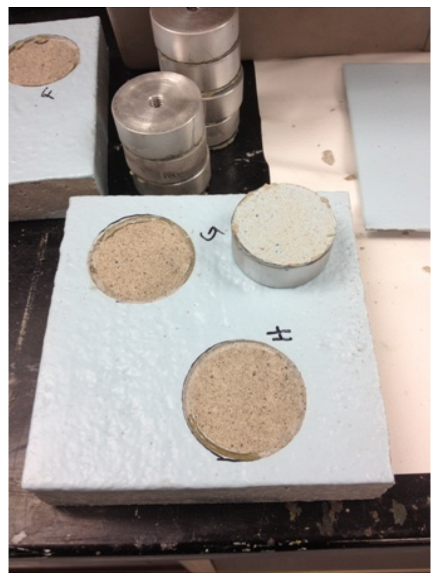

Figure 8-1. Adhesion test coupon

The results shown in Tables 8-1 through 8-4 are presented in the following sequence: concrete-initial, concrete after 200 hours humidity, steel, and steel after 200 hours humidity.

Table 8-1. Adhesion – concrete initial

Stub ID	Pull-Off Strength (psi)	Pull-Off Strength (psi)	Pull-Off Strength (psi)	Pull-Off Strength (psi)
	ALARA 1146	*CC Strip/CC Fix**	*CC Strip/SBR 10**	*Intergard 10220**
1	209	324	203	496
2	204	191	242	254
3	193	401	216	455
4	178	375	229	471
5	204	*	*	*
6	188	*	*	*
7	204	*	*	*
8	214	*	*	*
Average	199	323	223	419
Criteria	>50 psi			
Result	Pass	Pass	Pass	Pass

* Per QAPP only four replicates were used

Table 8-2. Adhesion – concrete after 200-hr humidity

Stub ID	Pull-Off Strength (psi)	Pull-Off Strength (psi)	Pull-Off Strength (psi)	Pull-Off Strength (psi)
	ALARA 1146	*CC Strip/CC Fix***	*CC Strip/SBR 10***	*Intergard 10220***
1	178	71	168	433
2	115	46	117	407
3	229	*	163	445
4	159	*	137	344
5	38*	**	N/A	**
6	127	**	**	**
7	121	**	**	**
8	127	**	**	**
Average	151	29	146	407
Criteria	>50 psi			
Result	Pass	Fail	Pass	Pass

* pull stub removed as test started

** Per QAPP only four replicates were used

Table 8-3. Adhesion – steel initial

Stub ID	Pull-Off Strength (psi)	Pull-Off Strength (psi)	Pull-Off Strength (psi)	Pull-Off Strength (psi)
	ALARA 1146	***CC Strip/CC Fix***	***CC Strip/SBR 10***	***Intergard 10220***
A	2522	1141	610.7	1670
B	2522	1161	610.7	1874
C	2382	1141	610.7	1711
D	2100	1182	610.7	1711
E	2382	1059	569.9	2038
F	2522	1059	488.3	1874
Average	2405	1124	584	1813
Criteria	>50 psi			
Result	Pass	Pass	Pass	Pass

Table 8-4. Adhesion – steel after 200-hr humidity

Stub ID	Pull-Off Strength (psi)	Pull-Off Strength (psi)	Pull-Off Strength (psi)	Pull-Off Strength (psi)
	ALARA 1146	***CC Strip/CC Fix***	***CC Strip/SBR 10***	***Intergard 10220***
A	<200*	284.2	**	1386
B	<200*	**	508.7	1631
C	<200*	**	569.9	1631
D	<200*	202.6	549.5	1018
E	<200*	202.6	529.1	1345
F	1397	202.6	406.7	1549
Average	--[a]	223[b]	513[c]	1427
Criteria	>50 psi			
Result	--	Pass	Pass	Pass

* pull stub removed as test started; due to rusted panel

** pull stub removed as test started

[a] not able to calculate average

[b] average calculated using stubs A,D,E,F

[c] average calculated using stubs B,C,D,E,F

8.3 Taber abrasion resistance

Taber abrasion resistance was determined in accordance with ASTM D4060, "Test Method for Abrasion Resistance of Organic Coatings by the Taber Abraser." Four 4" x 4" panels were weighed and then subjected to 1000 cycles using a 1000 g load and CS-17 abrasion wheel. Post-abrasion weights were acquired for the samples and the weight loss (in mg) reported. The test results for the four coatings are shown in Table 8-5.

8.4 Tensile strength

Tensile strength was determined in accordance with ASTM D2370, "Standard Test Method for Tensile Properties of Organic Coatings." The resulting samples were maintained at ambient laboratory conditions (72 ± 5 °F and 50 ± 5 %RH) for a minimum of 40 hours before testing. The samples were cut to a width of ½" using a double blade cutter and were tested for tensile strength using a Tinius-Olsen Universal Testing Machine (Tinius Olsen, Inc. Horsham, PA, USA). The results of the testing are shown in Table 8-6 for each of the four coatings.

8.5 Stormer viscosity

Stormer viscosity was determined in accordance with ASTM D562, "Standard Test Method for Consistency of Paints Measuring Krebs Unit (KU) Viscosity using a Stormer-Type Viscometer." Stormer-Type viscometers are produced by various manufacturers and are used to determine the viscosity of paints, similar to the coatings tested. The Stormer viscometer measurements were taken to satisfy the requirements of the freeze/thaw resistance testing in ASTM D2337 for organic coatings in the shelf life test method and also for aqueous coatings in ASTM D2243. Both ASTM methods, D2337 and D2243 were used only during the verification, using ALARA 1146. The remaining coatings were tested using only ASTM D 2337. The results of the Stormer-Type viscometer measurements are shown in Table 8-7 for all four coatings.

Table 8-5. Taber abrasion resistance

Replicate ID	ALARA 1146			CC Strip/CC Fix			CC Strip/SBR-10			Intergard 10220		
	Initial Weight (g)	Final Weight (g)	Weight Loss (mg)	Initial Weight (g)	Final Weight (g)	Weight Loss (mg)	Initial Weight (g)	Final Weight (g)	Weight Loss (mg)	Initial Weight (g)	Final Weight (g)	Weight Loss (mg)
A	70.6396	70.5242	115.4	65.0575	64.9883	69.2	66.3012	66.2403	60.9	65.1257	65.1182	7.5
B	71.1786	71.0676	111.0	65.4911	65.4199	71.2	65.2493	65.1860	63.3	64.9805	64.9667	13.8
C	70.6337	70.5195	114.2	65.3762	65.3066	69.6	66.5958	66.5353	60.5	65.3345	65.3189	15.6
D	70.9035	70.7887	114.8	64.8932	64.8194	73.8	66.5056	66.4495	56.1	65.1114	65.1027	8.7
Average			114			71			60			11
Criteria	<50 mg			<50 mg			<50 mg			<50 mg		
Result			Fail			Fail			Fail			Pass

Table 8-6. Tensile strength

Replicate ID	ALARA 1146			CC Strip/CC Fix			CC Strip/SBR-10			Intergard 10220		
	Force (pounds [lb])	Tensile Strength (psi)	Percent Elongation (%)	Force (lb)	Tensile Strength (psi)	Percent Elongation (%)	Force (lb)	Tensile Strength (psi)	Percent Elongation (%)	Force (lb)	Tensile Strength (psi)	Percent Elongation (%)
1	13.28	1451	230	3.00	211	1144	12.56	581	1144	11.82	1876	764
2	13.43	1476	206	3.34	246	1358	13.01	602	1065	13.13	1945	696
3	13.31	1471	202	2.71	206	1704	11.55	531	1065	12.26	1946	658
5	14.51	1603	214	2.99	251	1022	13.05	585	1200	12.37	1918	668
8	11.96	1429	206	3.62	296	1201	14.32	663	1124	12.26	1916	628
Average	1486			242			592			1920		
Criteria	>500 psi			>500 psi			>500 psi			>500 psi		
Result	Pass			Fail			Pass			Pass		

Table 8-7. Stormer viscosity (Krebs Units)

Sample ID		Initial	After Freeze/Thaw (Cycle 1)	After Freeze/Thaw (Cycle 2)*	Avg	% Diff	Criteria	Result
ALARA 1146	ALARA 1146	105	93.7	72.4	99.33	11%	< 10%	Fail
CC Strip/CC Fix	CC VIS-Strip	102.9	Solid	--	--	--	--	--
	CC Fix	67.2	Solid	--	--	--	--	--
CC Strip/SBR-10	VIS-Strip	102.9	Solid	--	--	--	--	--
	SBR-10 Part A	49.6	50.9	--	50.2	2.6%	< 10%	Pass
	SBR-10 Part B	90.7	87.5	--	89.1	3.6%	< 10%	Pass
	SBR-10 Mixed	55.6	55.0	--	55.3	1.1%	< 10%	Pass
Intergard 10220	Intergard 10220	138	Solid	--	--	--	--	--

* Cycle 2 performed for Alara 1146 only

8.6 Brookfield viscosity

The viscosity was determined using a Brookfield Viscometer (Brookfield Engineering Laboratories, Inc. Middleboro, MA, USA) (preferred method) in accordance with ASTM D2196, “Standard Test Method for Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield-Type) Viscometer.” The spindle type and speed used are largely a trial and error process with the objective being to choose a combination that will produce a display reading between 10% and 100% torque. The two spindles and speeds shown were used during the method verification and were, therefore, used for all subsequent coatings tested. The results for all four coatings are shown in Tables 8-8 through 8-11. Figure 8-2 shows SBR-10 being measured for viscosity using a Brookfield Viscometer.

Figure 8-2. Brookfield viscosity measurement of SBR-10

Table 8-8. ALARA 1146 Brookfield viscosity (centipoises [cps])

Sample ID	Spindle 4 Speed-20 rpm**	Spindle 5 Speed-20 rpm
ALARA 1146-Initial	7175	7400
ALARA 1146-After Storage Stability	3400	3450
Viscosity Change (%)	71%	73%
Viscosity Change Criteria	<10%	<10%
Result	Fail	Fail

**Revolutions per minute = rpm.

Table 8-9. CC Strip/CC Fix Brookfield viscosity (cps)

Sample ID	Spindle 4 Speed - 20 rpm		Spindle 5 Speed - 20 rpm	
	Initial	Post	Initial	Post
CC Strip	16950	No Reading*	10300	No Reading*
Sample ID	Spindle 4 Speed - 100 rpm		Spindle 5 Speed - 100 rpm	
	Initial	Post	Initial	Post
CC Fix	865	820	425	380
Viscosity Change (%)	9.9%		11.2%	
Viscosity Change Criteria	<10%		<10%	
Result	Pass		Fail	

* Sample solidified after evaluation using MIL-PRF-32239

Table 8-10. CC Strip/SBR-10 Brookfield viscosity (cps)

Sample ID	Spindle 4 Speed - 20 rpm		Spindle 5 Speed - 20 rpm	
	Initial	**Post Cycle 3**	**Initial**	**Post Cycle 3**
CC Strip (Initial)	16950	No Reading*	5000	No Reading*
Sample ID	**Spindle 2 Speed - 50 rpm**		**Spindle 2 Speed - 100 rpm**	
	Initial	**Post Cycle 3**	**Initial**	**Post Cycle 3**
SBR-10 Part A - After Storage Stability (Cycle 3)	60	48	60	62
Viscosity Change(%)	12 (22.2%)		2 (3.3%)	
Viscosity Change Criteria	<10%		<10%	
Result	Fail		Pass	
Sample ID	**Spindle 3 Speed - 50 rpm**		**Spindle 4 Speed - 50 rpm**	
	Initial	**Post Cycle 3**	**Initial**	**Post Cycle 3**
SBR-10 Part B - After Storage Stability (Cycle 3)	1190	1085	1180	1040
Viscosity Change (%)	5 (9.2%)		40 (12.6%)	
Viscosity Change Criteria	<10%		<10%	
Result	Pass		Fail	
Sample ID	**Spindle 3 Speed - 100 rpm**		**Spindle 4 Speed - 100 rpm**	
	Initial	**Post Cycle 3**	**Initial**	**Post Cycle 3**
SBR-10 Mixed - After Storage Stability (Cycle 3)	710	230	760	200
Viscosity Change (%)	480 (102%)		560 (117%)	
Viscosity Change Criteria	<10%		<10%	
Result	Fail		Fail	

* Sample solidified after evaluation using MIL-PRF-32239

Table 8-11. Intergard 10220 Brookfield viscosity (cps)

Sample ID	Spindle 5 Speed - 10 rpm		Spindle 6 Speed - 20 rpm	
	Initial	Post Cycle 3	Initial	Post Cycle 3
Intergard 10220	30000	(Too thick to read*)	24000	41250
Viscosity Change (%)	--		[17,250] 52.9%	
Viscosity Change Criteria	<10%		<10%	
Result	--		Fail	

* Spindle and speed adjustments did not lead to a comparable result

8.7 Density

Density was determined in accordance with ASTM D1475, "Standard Test Method for Density of Liquid Coatings, Inks, and Related Products." The instrument used was a Gardco U.S. Standard Cup (Paul N. Gardner Company, Inc., Pompano Beach, FL, USA). The liquid material was tested prior to any exposure and after the storage stability tests. The replicate average results for all four coatings are shown in Table 8-12; results are reported as pounds per gallon (lb/gal).

Table 8-12. Density

Sample ID		Initial (lb/gal)	After Storage Stability (Cycle 3) (lb/gal)	Density Change (%)	Density Change Criteria (%)	Result
ALARA 1146	ALARA 1146	8.65	9.095	5.1	<10%	Pass
CC Strip/CC Fix	CC Strip	8.978	8.988	0		Pass
	CC Fix	8.668	8.668	0		Pass
CC Strip/SBR-10	CC Strip	8.718	8.715	0		Pass
	SBR-10 Part A	8.874	8.880	0		Pass
	SBR-10 Part B	8.836	8.806	0		Pass
	SBR-10 Mixed	8.978	8.988	0		Pass
Intergard 10220	Intergard 10220	9.082	8.650	[4.8]		Pass

9.0 Summary of results

Tables 9-1 and 9-2 present a summary of the test results for each coating in pass/fail format. None of the four products tested passed all of the performance requirements successfully to meet both the five-year shelf life and weatherability requirements. As specified in ASTM E-2731, the overarching sequestration coating performance specification, the inability to pass all performance requirements does not necessarily eliminate a particular product from consideration as a potential deployable asset. More importantly, the data derived from subjecting potential products to the test methods described herein, as well as those listed in the larger E-2731 specification, can be helpful to assist emergency planning and response organizations as they make decisions regarding preparation for and response to a radiological contamination event. A summary of the results for testing under the Shelf Life Test Method is presented in Table 9-1 with results for Weatherability testing in Table 9-2. Some of the performance requirements may prove difficult for any given product to meet. However, as mentioned in ASTM E-2731-09, a product that meets some, but not all, of the performance requirements may have value for a particular response and recovery scenario. The data derived from this testing may be used as a guide by which to evaluate such products.

Table 9-1. Summary of results for shelf life

	Freeze/Thaw Resistance	**Settling**	**Accelerated Storage Stability**	**Density**	**Viscosity**
	ASTM D2337	**ASTM D1309 D869**	**MIL-PRF-32239**	**ASTM D1475**	**ASTM D2196**
ALARA 1146	Fail	Pass	Fail-Viscosity Pass-Density	Pass	Pass
CC Strip/CC Fix	Fail	Fail-CC Strip, Pass-CC Fix	CC Strip: Fail-Viscosity Pass-Density CC Fix: Pass-Viscosity Pass-Density	Pass	Pass
CC Strip/SBR-10	Fail-CC Strip Pass-SBR-10	Fail-CC Strip Pass-SBR-10	CC Strip: No Viscosity Pass-Density SBR-10: Fail-Viscosity Pass-Density	Pass	Pass
Intergard 10220	Fail	Fail	Fail-Viscosity Pass-Density	Pass	Pass

Table 9-2. Summary of results for weatherability

	Water Resistance	Tensile Strength	Abrasion Resistance	Natural Sunlight Soak-Freeze-Thaw Tolerance*	Adhesion Strength
	ASTM D2247	**ASTM D2370**	**ASTM D4060**	**ASTM D5722**	**ASTM D7234**
ALARA 1146	Pass-Concrete Fail-Steel	Pass	Fail	--	Pass-Concrete Pass-Steel
CC Strip/CC Fix	Pass-Concrete Pass-Steel	Fail	Fail	--	Pass-Concrete Pass-Steel
CC Strip/SBR-10	Pass-Concrete Pass-Steel	Pass	Fail	--	Pass-Concrete Pass-Steel
Intergard 10220	Pass-Concrete, Pass-Steel	Pass	Pass	--	Pass

* Testing not conducted

Appendix A - Shelf Life Test Method

Testing Procedure for Assessing Shelf Life of Containment Coatings

1. Scope

1.1 This specification is intended to provide a basis for testing of materials used to immobilize radioactive contamination and facilitate subsequent decontamination. These materials are herein named containment coatings (CCs).

1.2 This specification provides a set of test methods to be used to evaluate properties of CCs intended for use to prevent the spread of radioactive contamination. Some of the test procedures are provided as accelerated methods. However, other test procedures are available to evaluate the candidate coating on a non-accelerated basis.

1.3 The values stated in SI units are to be regarded as standard. The values given in parentheses are mathematical conversions that are provided for information only and are not considered standard.

1.4 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use. A CC must be qualified nonhazardous as defined by US EPA and US DOT. CC must also be nontoxic as an aerosol, vapor, liquid, or solid after application and curing.

1.5 User should refer to **ASTM E2731-09**: Standard Specification for Materials to Mitigate the Spread of Radioactive Contamination after a Radiological Dispersion Event

2. Referenced Documents

2.1 *ASTM Standards:*

E2731-09 Standard Specification for Materials to Mitigate the Spread of Radioactive Contamination after a Radiological Dispersion Event

D562-01(2005) Standard Test Method for Consistency of Paints Measuring Krebs Unit (KU) Viscosity Using a Stormer-Type Viscometer

D869-85R04 Standard Test Method for Evaluating Degree of Settling of Paints

D1309-93R04 Settling Properties of Traffic Paints During Storage

D1475-98R08 Density of Liquid Coatings, Inks, Related Products

D2196-10 Viscosity by Brookfield viscometer

D2337-01R05 Freeze-Thaw Stability of Multicolor Lacquers

D4287-00R05 High-Shear Viscosity Using a Cone/Plate Viscometer

D7395-07 Cone/Plate Viscosity at a 500 s-1 Shear Rate

2.2 Military Standards

MIL-PRF-32239 Coating System, Advanced Performance – Accelerated Storage Stability

3. Terminology

3.1 *Definitions:*

containment coating—film-forming product used to physically or chemically hold or bind radioactive particulates; containment does not mean affecting the radioactivity or the decay process of the radioactive contamination

containment coating film—material that results from the application of the containment coating

immobilize—to fix in place; to prevent movement or re –formation of aerosol of particulates due to mechanical or environmental forces such as by tracking, precipitation, or wind

weatherability – the ability of a containment coating to withstand weathering without degradation of performance. Weathering is defined as exposure to a variety of outdoor environmental conditions; for example, ultraviolet (UV) exposure, water, high and low temperatures, and common bacteria

4. Significance and Use

4.1 This specification establishes test methodology for a CC that is intended to immobilize dispersible radioactive contamination deposited on buildings and equipment as might result from a radiological dispersal device (RDD) event.

4.2 The intended use of this the CC addressed in this specification is primarily in an urban environment; however, it may be used in other environments such as suburban or rural areas.

4.3 The CC is intended to be removable during subsequent decontamination and recovery operations. It is intended to prevent the radioactive contamination from further migration, re-suspension into the air; and spreading as a result of external forces.

5.0 Minimum Performance Criteria

Minimum performance criteria for CCs have been established by **E2731–09** Standard Specification for Materials to Mitigate the Spread of Radioactive Contamination after a Radiological Dispersion Event. **E2731–09** specifies the following physical parameters for a CC: Tensile Strength 35 kg/cm^2 (500 psi), Adhesion >3.5 kg/cm^2 (>50 psi) on concrete, Abrasion <50-mg (0.002-oz) loss, Tear Strength 14 kg/cm^2 (200 psi), Flammability - does not burn. **E2731–09** also specifies a required shelf life of a minimum of 5 years and the CC, or stabilizer film, must meet the above physical parameters for a period of one year under weather conditions as described in 3.1. Any CC being evaluated for approval which meets some, but not all, of these performance criteria may still be considered to have value. However, all CCs must meet the requirements as specified in **E2731-09** before being considered for the tests as stated in this protocol.

6.0 Shelf Life

The following six test methods, or a combination of, shall be used to simulate and evaluate the effect of shelf life on properties of the CC. Test methods for viscosity shown in 6.6 and 6.7 are offered as alternatives to 6.5 depending upon shear tolerance. If an uncontrolled climate storage facility is used, the CC must meet the requirements of 6.2. This would be used for organic coatings. If a climate-controlled facility is used, the CC must meet the requirements of 6.3. This would be used for aqueous-based coatings. It should be noted that some of the tests require the coating to be tested on the substrate, whereas, other tests require the coating to be tested attached to the substrate. However, the CC must meet the applicable tests chosen in addition to the other tests as presented below. These test methods will be used to identify potential degradation of a coating by performing the accelerated and non-accelerated test(s). The test method results will identify any deficiencies of a coating to be applied as intended and will establish and verify the ability of a coating to possess successful shelf life performance. For each test method below, the equipment needed is listed first followed by the methodology of the test. Table 1 below provides a menu of properties for testing the shelf life of a CC. For each property, the table gives a preferred method as well as alternate methods, when available.

Property Describing Shelf Life	**Preferred Method**	**Alternate Methods**
Freeze/Thaw Resistance of Coatings	ASTM D2337[A]	N/A
Settling Properties	ASTM D1309/869	N/A
Accelerated Storage Stability	MIL-PRF-32239	N/A
Density	ASTM D1475	N/A
Viscosity	ASTM D2196	ASTM D562; ASTM D4287; ASTM D7395

[A] Although ASTM D2337 is titled "Freeze/Thaw Resistance of Organic Coatings", this method will be used for both lacquers or organic coatings as well as aqueous-based coatings.

6.1 Freeze-Thaw Resistance of Coatings[1] **-modified**

To be used for all coatings (lacquers or organic coatings as well as aqueous-based coatings).

6.1.1 Equipment Needed

Cold box capable of -3.9 °C (25 °F); Brooksfield viscometer, accurate timer

6.1.2 Methodology

ASTM D2337-01R05 will be modified as stated. Perform thirty cycles of 16 hours at -3.9 ± 5 °C (25 ± 5 °F) followed by 8 hours at room temperature, 22.2 ± 3 °C (72 ± 5 °F). Following the completion of above described thirty cycles, perform **ASTM D2196** or other suitable method to check viscosity (see 6.5 below). CC manufacturer shall specify the recommended viscosity range when tested in accordance with 6.5.

6.2 Settling Properties[2]

Accelerated method (modified) that simulates in 2 weeks the settling that might occur during 12 months of storage.

6.2.1 Equipment Needed

Freezer capable of -15 to -12.2 °C (5 to 10 °F) temperature range , oven capable of up to at least 71.1 °C (160 °F), spatula

[1] **D2337-01R05** Freeze-Thaw Stability of Multicolor Lacquers

[2] **D1309-93R04** Settling Properties of Traffic Paints During Storage

6.2.2 Methodology

Accelerated test with a 14 day cycle; Freezer, set to -13 °C (8.6 °F) -8 AM; Oven-10 AM; Freezer-12 PM; Oven-2 PM, followed by a 16-hr cycle at room temperature for five days. From 4 PM Friday until 8 AM Monday, leave at room temperature. Freezer-8 AM Monday and repeat cycle for 7 more days. Each time can of coating is removed from the oven, tap sharply on bench-top. At completion of exposure, allow to cool at room temperature and determine degree of settling per **ASTM D869**[3]**.**

Use a standard 500-ml (1 pint) friction top paint container. Prepare a spatula with square-end blade 12.1 cm (4 ¾ in) length and approximately 2.1 cm (13/16 in) width weighing 45 ± 1 g (1.6 oz) will be used to examine the paint for settling. Drop spatula in coating to determine degree of settling per **ASTM D869**. The conditions of the CC should be designated as following:

Rating	**Description of Paint Condition**
10	Perfect suspension. No change from the original condition of the paint.
8	A definite feel of settling and a slight deposit brought up on spatula. No significant resistance to sidewise movement of spatula.
6	Definite cake of settled pigment. Spatula drops through cake to bottom of container under its own weight. Definite resistance to sidewise motion of spatula. Coherent portions of cake may be removed on spatula.
4	Spatula does not fall to bottom of container under its own weight. Difficult to move spatula through cake sidewise and slight edgewise resistance. Paint can be remixed readily to a homogeneous state.
2	When spatula has been forced through the settled layer it is very difficult to move spatula sidewise. Definite edgewise resistance to movement of spatula. Paint can be remixed to a homogeneous state.
0	Very firm cake that cannot be reincorporated with the liquid to form a smooth paint by stirring manually.

6.3 Accelerated Storage Stability[4]

6.3.1 Equipment Needed

Oven capable of 60 °C (140 °F)

6.3.2 Methodology

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[3] **D869-85R04** Standard Test Method for Evaluating Degree of Settling of Paint

[4] **MIL-PRF-32239** Coating System, Advanced Performance – Accelerated Storage Stability

Store at 60 °C (140 °F) for seven days, cool to room temperature 22.2 °C (72 °F) and check density (**D1475**) and viscosity (**D2196**). Compare density and viscosity to values before subjecting to accelerated test. See 6.4 and 6.5. below, respectively. Any difference in values before and after accelerated test could indicate degraded CC during extended storage conditions.

6.4 Density[5]

6.4.1 Equipment Needed

Standardized cup, balance, Type II water

6.4.2 Methodology

Use standardized cup, fill with water and determine volume; fill with liquid and weigh to determine density

6.5 Viscosity[6]

6.5.1 Equipment Needed

Brookfield viscometer, spindles, Brookfield reference chart (spindle/speed), thermometer

6.5.2 Methodology

Use a quart can or a large beaker (600 ml or greater) that will accommodate the spindle guard. Add sufficient coating to cover the 'mark' on the spindle. The spindle and speed are selected such that the generated value is between 20 and 80 on the dial. If the value is lower than 20 or higher than 80, disregard and select a different spindle/speed until the generated value falls between 20 and 80 on the dial.

6.6 High-shear Viscosity[7]

To be used with liquids that can stand high shear rate

6.6.1 Equipment Needed

Cone/plate viscometer

6.6.2 Methodology

Evaluate flow properties (viscosity) of CC under high shear conditions similar to brushing, spraying, or roll coating. Repeat viscosity determination with two specimens. If the two viscosity determinations differ by less than 7 %, calculate the mean and report as the high-

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[5] **D1475-98R08** Density of Liquid Coatings, Inks, Related Products

[6] **D2196-10** Viscosity by Brookfield viscometer.

[7] **D4287-00R05** High-Shear Viscosity Using a Cone/Plate Viscometer

shear viscosity for the material. If the values differ by more than 7 %, make a third determination. If no two readings are within 7 % of each other, the material is not suitable for testing by this method. For CCs that cannot withstand shear in **D4287**, use **D7395**.

6.7 Low Shear Viscosity - Cone/Plate Viscosity[8]

To be used with liquids at a shear rate of 500 s^{-1}; liquids that cannot stand shear rate as prescribed in **D4287**

6.7.1 Equipment Needed

Cone/plate viscometer

6.7.2 Methodology

Evaluate flow properties (viscosity) of CC under low shear conditions similar to procedure used in 6.6. Repeat viscosity determination with two specimens. If the two viscosity determinations differ by less than 7 %, calculate the mean and report as the high-shear viscosity for the material. If the values differ by more than 7 %, make a third determination. If no two readings are within 7 % of each other, the material is not suitable for testing by this method.

[8] **D7395-07** Cone/Plate Viscosity at a 500 s-1 Shear Rate

Appendix B - Weatherability Test Method

Testing Procedure for Assessing Weatherability of Containment Coatings

1. Scope

1.1 This specification is intended to provide a basis for testing of materials used to immobilize radioactive contamination and facilitate subsequent decontamination. These materials are herein named containment coatings (CCs).

1.2 This specification provides a set of test methods to be used to evaluate properties of CCs intended for use to prevent the spread of radioactive contamination. At this time this specification describes a test method to evaluate the weatherability of CCs. Some of the test procedures are provided as accelerated methods. However, other test procedures are available to evaluate the candidate coating on a non-accelerated basis.

1.3 The values stated in SI units are to be regarded as standard. The values given in parentheses are mathematical conversions that are provided for information only and are not considered standard.

1.4 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use. A CC must be qualified nonhazardous as defined by US EPA and US DOT. CC must also be nontoxic as an aerosol, vapor, liquid, or solid after application and curing.

1.5 User of this specification should refer to **ASTM E2731 – 09**: Standard Specification for Materials to Mitigate the Spread of Radioactive Contamination after a Radiological Dispersion Event.

2. Referenced Documents

2.1 ASTM Standards:

E2731 – 09 Standard Specification for Materials to Mitigate the Spread of Radioactive Contamination after a Radiological Dispersion Event

D610 Standard Practice for Evaluating Degree of Rusting on Painted Steel Surfaces

D660 Standard Test Method for Evaluating Degree of Checking of Exterior Paints

D662 Standard Test Method for Evaluating Degree of Erosion of Exterior Paints

D714 Standard Test Method for Evaluating Degree of Blistering of Paints

D772 Standard Test Method for Evaluating Degree of Flaking (Scaling) of Exterior Paints

D2247-02 Standard Practice for Testing Water Resistance of Coatings in 100% Relative Humidity

D2370-98 Standard Test Method for Tensile Properties of Organic Coatings

D4060-07 Standard Test Method for Abrasion Resistance of Organic Coatings by the Taber Abraser

D4214 Standard Test Methods for Evaluating the Degree of Chalking of Exterior Paint Films

D4585-07 Standard Practice for Testing Water Resistance of Coatings Using Controlled Condensation

D5722-08 Standard Practice for Performing Accelerated Outdoor Weathering of Factory-Coated Embossed Hardboard Using Concentrated Natural Sunlight and a Soak-Freeze-Thaw Procedure

D6083-05 Standard Specification for Liquid Applied Acrylic Coating Used in Roofing

D6695-08 Standard Practice for Xenon-Arc Exposures of Paint and Related Coatings

D7234-05 Adhesion Strength of Coatings on Concrete

2.2 Military Standards

MIL-PRF-32239 Coating System, Advanced Performance, for all Aerospace Applications Weather Resistance: Xenon Arc vs. UV

3. Terminology

3.1 Definitions:

containment coating—film-forming product used to physically or chemically hold or bind radioactive particulates; containment does not mean affecting the radioactivity or the decay process of the radioactive contamination

containment coating film—material that results from the application of the containment coating *immobilize*—to fix in place; to prevent movement or re –formation of aerosol of particulates due to mechanical or environmental forces such as by tracking, precipitation, or wind

weatherability – The ability of a containment coating to withstand weathering without degradation of performance. Weathering is defined as exposure to a variety of outdoor environmental conditions; for example, ultraviolet (UV) exposure, water, high and low temperatures, and common bacteria

4. Significance and Use

4.1 This specification establishes test methodology for a CC that is intended to immobilize dispersible radioactive contamination deposited on buildings and equipment as might result from a radiological dispersal device (RDD) event.

4.2 The intended use of this the CC addressed in this specification is primarily in an urban environment; however, it may be used in other environments such as suburban or rural areas.

4.3 The CC is intended to be removable during subsequent decontamination and recovery operations. It is intended to prevent the radioactive contamination from further migration, re-suspension into the air; and spreading as a result of external forces.

5. Minimum Performance Criteria

Minimum performance criteria for CCs have been established by **E2731 – 09** Standard Specification for Materials to Mitigate the Spread of Radioactive Contamination after a Radiological Dispersion Event. **E2731 – 09** specifies the following physical parameters for a CC: Tensile Strength 3447 kPa (500 psi), Adhesion >345 kPa (>50 psi) on concrete, Abrasion <50-mg loss (<0.002-oz), Tear 1379 kPa (200 psi), Flammability - does not burn. **E2731 – 09** also specifies a required shelf life of a minimum of 5 years and the CC, or stabilizer film, must meet the above physical parameters for a period of one year under weather conditions as described in 3.5. Any CC being evaluated for approval which meets some, but not all, of these performance criteria may still be considered to have value. The CC will be applied per the manufacturer's recommendations. It is assumed that the thickness of the coating will be adequate enough to allow for removal from the surface to facilitate testing and perform as a film as indicated in **E-2731-09**. It should be pointed out that while some test methods require removal of the CC from panel, other methods allow for testing to be performed on a panel. All materials to be tested will be performed on a standardized block of concrete prepared according to Quality Assurance Project Plan, Revision 2 "Assessment of Water Wash Down for Mitigation for Cesium Chloride Contamination Part II Pressure Washing", November 24, 2008 unless the test method specifies otherwise.

6.0 Weatherability

Weatherability tests as specified below shall be conducted with the CC that has been tested according to E2731-09 and meets its requirements with regard to shelf life, working life, cure time, immobilization of radioactive particles, and non-flammability. Specifically, weatherability of the CC will be tested following its guaranteed shelf life time. The guaranteed shelf life time prior to weatherability testing will be accomplished by either the actual storage time on the shelf or by accelerated shelf life tests as prescribed by the Shelf Life Method. For each weatherability test method below, the equipment needed is listed first followed by the methodology of the test. The weatherability testing will be comprised of the following:

- Water Resistance at 100% RH
- Tensile Properties
- Abrasion Resistance
- Natural Sunlight and Soak-Freeze-Thaw Procedure
- Adhesion strength of coatings on concrete

For a CC to be considered passing the weatherability test, it must satisfy the requirements of each of the five methods above. Table 1 below provides a menu of properties for testing of weatherability of a CC. For each property to be tested, the table gives a preferred method as well as alternate methods, where available.

Property Describing Weatherability	Preferred Method	Alternate Methods
Water resistance	D2247-02	D4585-07
Tensile strength	D2370-98 (organic-based coatings) D6083 - 05e1 (water-based coatings)	N/A
Abrasion resistance	D4060-07	D968 *
Natural sunlight and soak-freeze-thaw tolerance	D5722-08	D660 and/or D662 and/or D714 and/or D772 and/or D4214 and/or D610 ** D6695-08 or MIL-PRF-32239 ***
Adhesion strength	D7234-05	D4541 - 09e1 ****

N/A= Not Applicable

* for organic coatings only

** as a follow up to D5722-08; not meant to replace D5722-08

*** only for the concentrated natural light part of D5722-08

**** for surfaces other than concrete

6.1 Water Resistance[1]

Useful service of a CC may be predicted by knowing its resistance to water degradation. This test method evaluates durability of a CC at 100% RH.

6.1.1 Equipment Needed

Enclosed fog chamber, thermostatically controlled heated water tank, thermometer

6.1.2 Methodology

Coated samples and control panel with known durability are placed at 15 degrees from vertical in an enclosed fog chamber maintained at 38°C (100 °F) for a period of 300 hours. Color change, blisters, loss of adhesion, softening, and embrittlement are observed after no less than 5 min and no more than 10 min after removal from test. The CC shall be considered to be water

[1] **D2247-02** Standard Practice for Testing Water Resistance of Coatings in 100% Relative Humidity. Alternatively, **D4585-07** Standard Practice for Testing Water Resistance of Coatings Using Controlled Condensation may be used.

resistant if no more than 10% loss of adhesion is measured following the water resistance test and utilizing procedures described in **D7234-05** Standard Test Method for Pull-Off Adhesion Strength of Coatings on Concrete Using Portable Pull-Off Adhesion Testers. The remaining 90% (or more) of the original adhesion strength following water resistance tests as prescribed by D2247-02 will be sufficient to maintain the integrity of the CC on the coated surface.

6.2 Tensile Properties[2]

To determine tensile properties of organic coatings when tested as free films, **D2370-98** Standard Test Method for Tensile Properties of Organic Coatings shall be used. However, for water-based coatings, **D6083-05** shall be used. **D6083-05** clarifies test conditions regarding testing tensile strength of water-dispersed protective roof coatings. The tensile strength determined by D2370-98 (organic coatings) or by D6083 (water-based coatings) shall be at least 20% more than the adhesion strength as determined by D7234-05 Standard Test Method for Pull-Off Adhesion Strength of Coatings on Concrete Using Portable Pull-Off Adhesion Testers. Tensile strength at least 20% larger than the adhesion strength is necessary to assure efficient removal of the CC from the coated surface.

6.2.1 Equipment Needed for D2370-98

Stationary micrometer, tensile tester with load capacities from 100 to 2000 g (3.5 to 70.5 oz), precision specimen cutter, dental tin foil, sheet of polyethylene

6.2.2 Methodology for D2370-98

Free unsupported films of uniform thickness of the CC to be tested are prepared. Thickness of films is measured and samples are conditioned for at least 24 hours at 22.2 ± 3 °C (72 ± 5 °F) and 50 % RH. Next, samples are mounted in the tensile tester with pre-set target elongation and with preset load application rate. Pull in kg (lb.) needed to rupture the film is measured.

6.2.3 Equipment Needed for D6083-05

The same equipment as for the D2370-98 above.

6.2.4 Methodology for D6083-05

Film measuring 75 mm (2.9 in) long by 13 mm (0.5 in) wide tested according to D2370-98 at 22.2 ± 3 °C (72 ± 5°F) at 50 % relative humidity.

6.3. Abrasion Resistance[3]

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[2] **D2370-98** Standard Test Method for Tensile Properties of Organic Coatings and **D6083 - 05**Standard Specification for Liquid Applied Acrylic Coating Used in Roofing

[3] **D4060-07** Standard Test Method for Abrasion Resistance of Organic Coatings by the Taber Abraser

D4060-07 shall be used for testing of organic coatings applied at uniform thickness to plane, rigid surface and properly cured. **D4060-07** shall be also used for testing of water-based coatings. **D4060** was used in the past to determine abrasion resistance of water-based coatings as well.[4] To pass the test, the sample shall not lose more than 10% of its original weight. It is assumed that weight loss of up to 10% will not compromise integrity of the CC.

6.3.1 Equipment needed

Taber abraser, revolutions counter, abrasive wheels, resurfacing medium, vacuum suction

6.3.2 Methodology

Samples coated with a CC are conditioned, weighted, then abrased on a revolving wheel for a number of cycles. Following abrasion, sample is weighted again. Alternatively, **D968** Test Methods for Abrasion Resistance of Organic Coatings by Falling Abrasive, may be used for organic coatings only.

6.4. Natural Sunlight and Soak-Freeze-Thaw Procedure[5]

Note: The method was originally developed for performing accelerated outdoor weathering of factory-coated embossed hardboard.

6.4.1 Equipment Needed

Fresnel reflecting concentrator accelerated weathering machine described in **G90**, freezer capable of sustaining minus 20 °C (minus 4 °F) temperature

6.4.2 Methodology

Sample and control panel with known durability are exposed to concentrated natural sunlight in Fresnel machine (with periodic daytime surface water spray). Following this exposure to concentrated natural sunlight, the soak-freeze-thaw procedure is implemented. The soak-freeze-thaw cycle is comprised of immersion in DI water at 25 °C (77 °F) for 1 hour, freezing at minus 20 °C (minus 4 °F) for 12-15 hours, and thawing at room temperature for a minimum of 1 hour; 30 cycles are applied.

The following test methods may be used to determine changes in samples exposed to the soak-freeze-thaw cycles:

- **D660** (Standard Test Method for Evaluating Degree of Checking of Exterior Paints)
- **D662** (Standard Test Method for Evaluating Degree of Erosion of Exterior Paints)

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[4] **WATER-BASED ACRYLIC FLOOR COATING K0500 Series, Product Data Sheet by Krylon Industrial Coatings**

[5] **D5722-08** Standard Practice for Performing Accelerated Outdoor Weathering of Factory- Coated Embossed Hardboard Using Concentrated Natural Sunlight and a Soak-Freeze-Thaw Procedure

- **D714** (Standard Test Method for Evaluating Degree of Blistering of Paints)
- **D772** (Standard Test Method for Evaluating Degree of Flaking (Scaling) of Exterior Paints)
- **D4214** (Standard Test Methods for Evaluating the Degree of Chalking of Exterior Paint Films)

- **D610** (Standard Practice for Evaluating Degree of Rusting on Painted Steel Surfaces)

For the above six test methods, the CC will be deemed as passing the test method only if less than 5% of surface is affected. The 5% rate is considered to not significantly deteriorate the integrity of the CC.

Xenon Arc[6] Method D6695

This method may be used alternatively to the procedure involving exposure of the sample to concentrated natural sunlight only as described above in **D5722-08** Standard Practice for Performing Accelerated Outdoor Weathering of Factory- Coated Embossed Hardboard Using Concentrated Natural Sunlight and a Soak-Freeze-Thaw Procedure. The **D6695** method is used to test accelerated exposure of general coatings which is associated with sunlight, moisture, and heat.

Equipment Needed
Xenon-arc apparatus (conforming to requirements of G151 and G155), humidity chamber.

Methodology
After coating has been applied to a flat panel, the sample is conditioned at 23 °C (73.4 °F) and 50 %RH for at least 7 days. Exposure to xenon-arc light for specified time is described in Table 1 of **D6695-08** (for general coatings, Cycle 1 is used with continuous light for 102 min at 50% RH). Corresponding typical irradiances are given in Table 1 of **D6695-08**. The test methods listed above in 6.4.2 above may be used to determine changes in exposed samples.

Another alternative to the concentrated natural light part of D5722-08 only may be Xenon Arc testing described by **MIL-PRF-32239[7].** Equipment needed for this test is Xenon-arc weather-o-meter type Q-Sun/3000 or Q-Sun/1000. In this method, test panels are exposed for 3000 hours in a Xenon-arc weather-o-meter cycling between 102 minutes of light only and 18 minutes of light and DI water spray. After the exposure samples are examined for conformance to 3.6.9 Flexibility, Class 2: High Flexibility Coating System. For the CC to pass this test, it will exhibit no cracking when tested according to 4.6.15.2 Low Temperature Flexibility of the MIL-PRF-32239.

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[6] **D6695-08** Standard Practice for Xenon-Arc Exposures of Paint and Related Coatings

[7] **MIL-PRF-32239** Performance Specification-Coating System, Advanced Performance, for Aerospace Applications Weather Resistance: Xenon Arc vs. UV

6.5 Adhesion strength of coatings on concrete[8]

This test method describes procedures for evaluating the pull-off adhesion strength of a coating applied to concrete. The test determines the greatest perpendicular force (in tension) that a surface of coating can bear before a plug of coating is detached from the surface. This greatest perpendicular force determined by **D7234-05** must be at least 345 kPa (50 psi), as required by E2731-09.

Alternatively, **ASTM D4541-09e1** Standard Test Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers may be used for substrates other than concrete. The method consists of several “sub-methods” (B through F) applicable to various substrates, e.g., plastic, wood, etc. Sub-method A, previously addressing concrete substrate has been withdrawn. According to **ASTM D4541-09e1**, the standard was developed for metal substrates, but may be appropriate for other rigid substrates such as plastic and wood. This test method uses a portable pull-off adhesion tester capable of applying a concentric load and counter load to a single surface so that coatings can be tested even though only one side is accessible.

6.5.1 Equipment Needed
Pull-off adhesion tester, force applicator, and timer for control of the rate of stress application.

6.5.2 Methodology
CCs thicker than 0.5 mm (0.02 in) must be scored in the direction normal to the surface of the CC. The adhesive is then applied between the surface of the CC to be tested and the fixture. After the adhesive has cured, adhesion tester is applied to the fixture. The load is applied in a smooth and gradual manner until the failure (separation of the CC from the surface). The force attained at failure is recorded and pull-off adhesion strength is calculated. Finally, the substrate failure or other modes of failure are classified.

[8] **D7234-05** Standard Test Method for Pull-Off Adhesion Strength of Coatings on Concrete Using Portable Pull-Off Adhesion Testers

Appendix C - KTA Test Report – ALARA 1146

August 8, 2013
Via Email: *brent.hall@arcadis-us.com*

Mr. Brent Hall
ARCADIS U.S., Inc.
4915 Prospectus Drive, Suite F
Durham, NC 27713-4408

SUBJECT: Laboratory Testing of Strippable Coating; KTA-Tator, Inc. Project No. 320715-R2

Dear Mr. Hall:

In accordance with KTA Proposal PN120083 and ARCADIS U.S. Inc. (ARCADIS) Work Authorization No. D12-0178, KTA-Tator, Inc. (KTA) has tested a coating material to several different requirements to determine the resulting physical properties. This report describes the laboratory techniques used and contains the results of the testing.

SAMPLES

The samples listed in Table 1, "Samples" were received from ARCADIS on the dates listed. It should be noted that at no time did KTA personnel witness the acquisition or manufacturing of the samples.

Table 1 – Samples

Sample ID	Sample Description	Date Received
KTA-1	Three 1-gallon cans of Alara 1146, Lot # L787-072, Blue 0100. ***Arrived damaged and leaking.***	October 31, 2012
KTA-2	Two 1-gallon cans of Alara 1146, Lot #L787-161, Blue 0100. ***Not damaged or leaking.***	November 20, 2012
KTA-3	Thirteen concrete panels for application of coating and testing measuring 6″x6″.	October 31, 2012

LABORATORY INVESTIGATION

The laboratory investigation consisted of exposure of the liquid material to different thermal cyclic conditions to simulate storage and freeze/thaw possibilities followed by evaluations of viscosity, density and settling. The coating film was tested to determine the resistance to humidity and abrasion. The film was also tested to determine the tensile adhesion value and the tensile strength. Since the first shipment of material was leaking, the cans were not used for testing. All of the testing was performed using Lot #L787-161.

<u>Thermal Cycling</u>

Four different conditions of thermal cycling or exposure were evaluated for this testing protocol. Each condition is described in a paragraph below. The coating was evaluated by different methods at the conclusion of the exposure. The results are addressed in the sections explaining the test methods employed for the evaluations. Each of the cyclic tests was assigned a number and a short designation for each is referenced in the correspondence results tables.

Cycle 1 – Freeze/Thaw of Solvent based materials

Thermal cycling was performed in accordance with ASTM D2337-01(10), "Standard Test Method for Freeze-Thaw Stability of Multicolor Lacquers". This test method was performed to determine the resistance of the solvent based liquid coating material to cycles of freezing and thawing. The duration of the test was 30 cycles. The cycles occurred on weekdays. The can remained in the freezer during the weekends. The thermal cycling conditions for this test consisted of sixteen hours at 25 ± 3°F and eight hours at room temperature (72 ± 5°F). The liquid material was evaluated by Stormer viscosity after exposure. A visual examination of the coating revealed that the coating was lumpy initially, but could be mixed to a consistent fluid. A ¼" to ½" layer of material at the base of the can remained thick and did not mix in well during hand mixing of the coating.

Cycle 2 – Freeze/Thaw of Water based materials

Thermal cycling was performed in accordance with ASTM D2243-95(08), "Standard Test Method for Freeze-Thaw Resistance of Water-Borne Coatings". This test method was performed to determine the resistance of the water based liquid coating material to cycles of freezing and thawing. The duration of the test was 30 cycles. The cycles occurred on weekdays. The can remained in the freezer during the weekends. The thermal cycling conditions for this test consisted of seventeen hours at 25 - 30°F and seven hours at room temperature (72 ± 5°F). The liquid material was evaluated by Stormer viscosity after exposure. The visual examination of the coating revealed that there was some separation of a yellow translucent material. Mixing incorporated the liquid, but the resulting material was not as thick as the original coating material and had a sandy appearance.

Cycle 3 – Storage Exposure

Thermal cycling was performed in accordance with ASTM D1309-93(10), "Standard Test Method for Settling Properties of Traffic Paints During Storage". This test method was performed to determine the resistance of the liquid coating material to cycles of freezing and heating. The duration of the test was 14 cycles. The cycles occurred on weekdays. The can remained in the at room temperature in the evenings and on the weekends. The thermal cycling conditions for this test consisted of two hours at 5 - 10°F, followed by two hours at 160°F. These cycles were repeated twice, then followed by 16 cycle at room temperature. The 8 hour cycle was completed during work hours. The liquid was evaluated for settling after exposure.

The immediate visual evaluation revealed skinning of the coating material that had been in contact with the interior surface of the lid and the sides of the can until the top surface of the liquid material (about ½″ to ¾″ down the side of the can). The liquid material was examined for settling and the results are included below

Cycle 4 – Storage Stability

Thermal cycling was performed in accordance with military specification MIL-PRF-32239, "Standard Method for Accelerated Storage Stability". This test method was performed to determine the resistance of the water based liquid coating material to extended heat exposure. The can remained in the oven set at 140°F for the 7 day duration of the test. The liquid material was requested to be evaluated for changes in viscosity using the Ford Cup, but the initial coating was too viscous for evaluation using the Ford Cup # 4. Subsequently, the viscosity was evaluated by Brookfield viscosity after exposure. Additionally, the density of the material was evaluated after exposure. A visual examination of the material reveals a slight separation of a translucent layer. Mixing revealed that the layer was incorporated and the resulting material appeared much like the unexposed coating material.

Stormer Viscosity

Stormer viscosity was determined in accordance with ASTM D562-10, "Standard Test Method for Consistency of Paints Measuring Krebs Unit (KU) Viscosity Using a Stormer-Type Viscometer." Readings were taken once the samples were brought to 25 ± 0.2°C, and recorded in Krebs units (KU). Baseline viscosity readings were obtained on liquid material that was not exposed to any of the conditions listed above. The results can be found in Table 2, "Results of Stormer Viscosity."

Table 2 – Results of Stormer Viscosity Evaluation

Sample ID	Initial	After Freeze/Thaw (Cycle 1)	After Freeze/Thaw (Cycle 2)
Alara 1146	105 KU	93.7 KU	72.4 KU

Ford Cup Viscosity

Ford cup viscosity was requested for the sample that was scheduled to undergo Cycle 4, storage stability. The initial sample was set for testing in accordance with ASTM D1200-10, "Standard Test Method for Viscosity by Ford Viscosity Cup." For this type of viscosity measurement, the liquid sample was stirred and its temperature was brought to 25 ± 0.2°C. Ford Cup #4 was filled with the prepared sample with the orifice at the bottom of the cup plugged with the analyst's finger. The finger was removed and a timer was simultaneously started. The viscosity is determined by the time of the first break in the stream, using a stopwatch. The liquid coating supplied for this testing did not flow through the orifice, leading to continual breaks in the stream. The coating material did not empty from the cup in less than 20 minutes and the testing was discontinued. ARCADIS was contacted and a mutual decision to evaluate the viscosity using the Brookfield viscometer was accepted.

Brookfield Viscosity

The Brookfield viscosity was determined in accordance with ASTM D2169-10, "Standard Test Method for Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield type) Viscometer." The temperature of the sample was raised to 25 ± 0.2°C. No spindle number or speed was specified for this product, so spindle 4 and 20 RPM speed were selected. The second set of tests was performed using spindle 5 and a speed of 20 RPM, for confirmation. For this testing, the spindle was lowered into the sample until the immersion mark on the spindle shaft touched the sample. The viscometer was turned on and the pointer was allowed to make several rotations until it had stabilized. Once the pointer stabilized, the viscometer was stopped by simultaneously pressing down on the power and hold switches. The viscosity reading was multiplied by the spindle factor, and recorded in centipoise (cps) units. The same procedure was used for the second set of conditions. The average result is value reported. The results can be found in Table 3, "Results of Brookfield Viscosity."

Table 3 – Results of Brookfield Viscosity Evaluation

Sample ID	Spindle 4 Speed – 20 rpm	Spindle 5 Speed – 20 rpm
Alara 1146 – Initial	7175 cps	7400 cps
Alara 1146 – After Storage Stability (Cycle 4)	3400 cps	3450 cps

Density

Density was determined in accordance with ASTM D1475-98(08), "Standard Test Method for Density of Liquid Coatings, Inks, and Related Products." A calibrated cup was weighed empty and then weighed full of liquid coating. The air bubbles were eliminated from the coatings as much as feasible by gently tapping the cup. The liquid material was tested prior to any exposure and then after the storage stability described in Cycle 4. Calculations were performed, and the results of the testing are listed in Table 4, "Density Results."

Table 4 – Density Results

Sample ID	Initial	After Storage Stability (Cycle 4)
Alara 1146	8.65 lb/gal	9.095 lb/gal

Degree of Settling

The degree of settling was evaluated in accordance with ASTM D869-85(11), "Standard Test Method for Evaluating Degree of Settling of Paint. This method requires that the coating material not be shaken or stirred after exposure, prior to the settling evaluation. In this case, the coating had skinned over where it had been in contact with the interior surface of the lid. The skin was removed and a blunt tipped spatula was dropped into the coating. The spatula hit the base of the can with a metallic sound, indicating that no settling had occurred in the 14 days of cyclic exposure represented in Cycle 3 – Storage Exposure. This testing would receive a settling value of 10.

Humidity Resistance

The coating was applied over both concrete and steel panels and permitted to dry for a minimum of 7 days prior to testing. The resulting panels were tested for humidity resistance in accordance with ASTM D2247-11, "Standard Practice for Testing Water Resistance of Coatings in 100% Relative Humidity." The coated panels were placed on the exterior of an enclosed chamber containing a heated, saturated mixture of air and water vapor. At an internal condition of 100°F and 100% relative humidity, the temperature difference between the specimen surface and the surrounding vapor causes the formation of condensation on the interior (painted surface) of the panels. The samples were subjected to 200 hours of humidity exposure. Initially, the exposure was scheduled for a duration of 300 hours, but the panels were displaying cracks and discoloration. The exposure was discontinued at the request of ARCADIS. After the exposure time has elapsed, the coated panels were evaluated for loss of tensile adhesion. The results are listed in the section titled Tensile Adhesion Strength.

Tensile Adhesion Strength

Tensile adhesion (pull-off strength) was measured in accordance with ASTM D4541-09e1, "Pull-Off Strength of Coatings Using Portable Adhesion Testers," Annex A4, "Self-Aligning Adhesion Tester Type IV" or ASTM D7234-12, "Standard Test Method for Pull-Off Adhesion Strength of Coatings on Concrete Using Portable Pull-Off Adhesion Testers." Both of the methods use the same equipment to remove the pull stubs. The main difference between the test methods is the size of the pull stubs. The ASTM D7234-12 for concrete specifies the 2″ pull stub. The coating on the steel panels was evaluated using the ½″ pull stub.

For both methods, the testing surfaces were wiped clean and abraded gently using fine sandpaper. Pull stubs with an abraded test surface were attached to the coating using a two component epoxy adhesive (Araldite 2011), which was allowed to cure for a minimum of twenty-four hours at ambient laboratory conditions (70 ± 2°F and 50 ± 5% relative humidity). The 2″ pull-stubs were then detached using a self-aligning pneumatic adhesion tester (PATTI-Pneumatic Adhesion Tensile Testing Instrument) employing the F8 piston (range: 25-255 psi). The ½″ pull stubs were detached using the F4 piston, which has a range of 202-2039 psi for the small diameter pull stub. The pressure required to remove each pull-stub was recorded along with the location of break and approximate percentage of each. The location of break is defined as adhesive (a split between layers), cohesive (within a layer), or glue failure (coating strength exceeds glue strength). The results of the testing can be found in Table 5, "Results of Tensile Adhesion Testing."

Table 5 – Results of Tensile Adhesion Testing

Coating System	Pull Stub ID	Pull-Off Strength (psi)	Location of Break	Average Pull-Off Strength (psi)
Alara 1146 to Concrete	1	209	100% adhesive at the concrete interface	199
	2	204	100% adhesive at the concrete interface	
	3	193	100% adhesive at the concrete interface	
	4	178	100% adhesive at the concrete interface	
	5	204	100% adhesive at the concrete interface	
	6	188	100% adhesive at the concrete interface	
	7	204	100% adhesive at the concrete interface	
	8	214	100% adhesive at the concrete interface	
Alara 1146 to Concrete (After 200 hours humidity)	1	178	80% adhesive to concrete, 20% cohesive	151*
	2	115	85% adhesive to concrete, 15% cohesive	
	3	229	90% adhesive to concrete, 10% cohesive	
	4	159	60% adhesive to concrete, 40% cohesive	
	5	38	95% adhesive to concrete, 5% cohesive	
	6	127	95% adhesive to concrete, 5% cohesive	
	7	121	90% adhesive to concrete, 10% cohesive	
	8	127	95% adhesive to concrete, 5% cohesive	
Alara 1146 to Steel	A	2522	100% adhesive at the steel interface	2405
	B	2522	100% adhesive at the steel interface	
	C	2382	100% adhesive at the steel interface	
	D	2100	100% adhesive at the steel interface	
	E	2382	100% adhesive at the steel interface	
	F	2522	100% adhesive at the steel interface	
Alara 1146 to Steel (After 200 hours humidity)	A	< 200**	100% adhesive at the steel interface, rusted	< 1397 (only 1 value reported)
	B	< 200**	100% adhesive at the steel interface, rusted	
	C	< 200**	100% adhesive at the steel interface, rusted	
	D	< 200**	100% adhesive at the steel interface, rusted	
	E	< 200**	100% adhesive at the steel interface, rusted	
	F	1397	100% adhesive at the steel interface, rusted	

* *Value from pull stub #5 not used for average.* ** *Pull stub removed as test started.*

Taber Abrasion Resistance

Taber abrasion resistance was determined in accordance with ASTM D4060-10, "Test Method for Abrasion Resistance of Organic Coatings by the Taber Abraser." Four replicate 4″ x 4″ panels were weighed then subjected to 1000 cycles using a 1000g load and CS-17 abrasion wheels. Post weights were acquired for the samples, and the weight loss (in mg) reported. The results of the testing are contained in Table 6, "Taber Abrasion Resistance Results."

Table 6 – Taber Abrasion Resistance Results

Coating System	Replicate ID	Initial Weight	Final Weight	Weight Loss (mg)	Average Weight Loss (mg)
Alara 1146	A	70.6396	70.5242	115.4	113.8
	B	71.1786	71.0676	111.0	
	C	70.6337	70.5195	113.9	
	D	70.9035	70.7887	114.8	

Tensile Strength

Tensile strength was determined in accordance with ASTM D2370-98(10), "Standard Test Method for Tensile Properties of Organic Coatings." The samples were cut to a width of ½″ using a double blade cutter. The resulting samples were maintained at ambient laboratory conditions (70 ± 2° F and 50 ± 5% RH) for a minimum of 40 hours before testing. The samples were tested for tensile strength using a Tinius Olsen Universal Testing Machine. The cross-head speed used for testing was 2.0 in/min. The gage length of 1 – 2″ was evaluated for three samples. A gage length of 1″ was selected due to the strength and elongation of the material. The force (in pounds) required to break the sample and the sample dimensions were used to calculate the tensile strength of each free film of coating in pounds per square inch (psi). The ultimate length at break was used to calculate the elongation. The method requires testing of 10 specimens. The five specimens with the highest combination of properties were selected for reporting. The results of the testing can be found in Table 7, "Results of Tensile Strength Testing."

Table 7 – Results of Tensile Strength Testing

Sample ID	Replicate ID	Force (lbs)	Tensile Strength (psi)	Average Tensile Strength (psi)	Percent Elongation (%)	Average Percent Elongation (%)
Alara 1146	1	13.28	1451	1486	230	212
	2	13.43	1476		206	
	3	13.31	1471		202	
	5	14.51	1603		214	
	8	11.96	1429		206	

If you have any questions or comments regarding this report, please contact me by telephone at 412-788-1300, extension 183, or by email at vsherbondy@kta.com.

Sincerely,

KTA-TATOR, INC.

Valerie D. Sherbondy
Senior Chemist

VDS/RNR:jsc
JN320715-R2
CIN: 205716

cc: Ms. Kim Egler of ARCADIS *<kim.egler@arcadis-us.com>*

R2 – A revision was issued to reference the four replicate taber panels in the text of the report.

(320715-R2 Arcadis.doc)

Appendix D - KTA Test Report – Instacote CC Strip/CC Fix

June 6, 2013
Via Email: *brent.hall@arcadis-us.com*

Mr. Brent Hall
ARCADIS U.S., Inc.
4915 Prospectus Drive, Suite F
Durham, NC 27713-4408

SUBJECT: Laboratory Testing of Strippable Coating;
KTA-Tator, Inc. Project No. 320715-A3-R1

Dear Mr. Hall:

In accordance with KTA Proposal PN131249 and ARCADIS U.S. Inc. (ARCADIS) Work Authorization No. D12-0178, KTA-Tator, Inc. (KTA) has tested a coating material to several different requirements to determine the resulting physical properties. This report describes the laboratory techniques used and contains the results of the testing.

SAMPLES

The samples listed in Table 1, "Samples" were received from InstaCote on the dates listed. It should be noted that at no time did KTA personnel witness the acquisition or manufacturing of the samples.

Table 1 – Samples

Sample ID	Sample Description	Date Received
KTA-5	Three 1-gallon cans of InstaCote CC FIX, Batch # 101612100-1	January 30, 2013
KTA-6	One 5-gallon pail of InstaCote CC Hi VIS-Strip #1 VIS-Strip, Batch #012413-4-2	January 30, 2013

LABORATORY INVESTIGATION

The laboratory investigation consisted of exposure of the liquid materials to different thermal cyclic conditions to simulate storage and freeze/thaw possibilities followed by evaluations of viscosity, density and settling. The applied coating system was tested to determine the resistance to humidity and the tensile adhesion value (Table 5), while the topcoat was tested to determine abrasion resistance (Table 6). A free-film sample of the coating system was also tested to determine the tensile strength (Table 7).

Thermal Cycling

Three different conditions of thermal cycling or exposure were evaluated for this testing protocol. Each condition is described in a paragraph below. The coating was evaluated by different methods at the conclusion of the exposure. The results are addressed in the sections explaining the test methods employed for the evaluations. Each of the cyclic tests was assigned a number and a short designation for each is referenced in the corresponding results tables.

Cycle 1 – Freeze/Thaw Stability

Freeze/ thaw thermal cycling was performed in accordance with ASTM D2337-01(2010), "Standard Test Method for Freeze-Thaw Stability of Multicolor Lacquers." This test method was performed to determine the resistance of the liquid coating material to cycles of freezing and thawing. The duration of the test was 30 cycles. The cycles occurred on weekdays. The can remained in the freezer during the weekends. The thermal cycling conditions for this test consisted of sixteen hours at 25 ± 3°F and eight hours at room temperature (72 ± 5°F). The liquid material was scheduled to be evaluated by Stormer viscosity after exposure. A visual examination of the coating materials revealed that both coatings were solid and Stormer viscosity readings could not be obtained.

Cycle 2 – Storage Exposure

Storage exposure was performed in accordance with ASTM D1309-93(2010), "Standard Test Method for Settling Properties of Traffic Paints During Storage". This test method was performed to determine the resistance of the liquid coating material to cycles of freezing and heating. The duration of the test was 14 cycles, which occurred on weekdays. The can remained at room temperature in the evenings and on the weekends. The thermal cycling conditions for this test consisted of two hours at 5 – 10°F, followed by two hours at approximately 160°F. These cycles were repeated twice, then followed by 16 hours at room temperature (72 ± 5°F). The 8 hour cycle was completed during work hours. The liquids were scheduled for evaluation of settling after exposure. The CC VIS-Strip had solidified and the degree of settling could not be evaluated for settling. The CC Fix product remained liquid and was evaluated for settling.

Cycle 3 – Storage Stability

Storage stability was performed in accordance with military specification MIL-PRF-32239, "Standard Method for Accelerated Storage Stability". This test method was performed to determine the resistance of the water based liquid coating material to extended heat exposure. The can remained in the oven set at approximately140°F for the 7 day duration of the test. At the completion of the exposure, the liquid material was scheduled for evaluation of viscosity using a Brookfield viscometer. Additionally, the density of the materials were to be evaluated after storage stability exposure. A visual examination of the materials revealed that the majority of the VIS-Strip material had solidified during the exposure. The small amount of liquid VIS-Strip product was used to perform density testing. Not enough liquid was present for a viscosity evaluation. The CC FIX material had remained liquid and fluid enough for testing. A photograph of the coating material can be found in the Photographic Appendix.

Stormer Viscosity

Stormer viscosity was determined in accordance with ASTM D562-10, “Standard Test Method for Consistency of Paints Measuring Krebs Unit (KU) Viscosity Using a Stormer-Type Viscometer.” Readings were taken once the samples were brought to 25 ± 0.2°C, and recorded in Krebs units (KU). Baseline viscosity readings were obtained on liquid material that was not exposed to any of the conditions listed above. The results can be found in Table 2, “Results of Stormer Viscosity Evaluation.”

Table 2 – Results of Stormer Viscosity Evaluation

Sample ID	Initial	After Freeze/Thaw (Cycle 1)
CC VIS-Strip	102.9 KU	Solid
CC Fix	67.2 KU	Solid

Brookfield Viscosity

The Brookfield viscosity was determined in accordance with ASTM D2196-10, “Standard Test Method for Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield type) Viscometer.” The temperature of the sample was raised to 25 ± 0.2°C. Previous testing was conducted using spindle 4 and 20 RPM speed, so the testing started with that selection. The second set of tests was performed using spindle 5 and a speed of 20 RPM, for confirmation. The selection was not appropriate for the CC FIX product and different combinations were used and are listed in the table. For this testing, the spindle was lowered into the sample until the immersion mark on the spindle shaft touched the sample. The viscometer was turned on and the pointer was allowed to make several rotations until it had stabilized. Once the pointer stabilized, the viscometer was stopped by simultaneously pressing down on the power and hold switches. The viscosity reading was multiplied by the spindle/speed factor, and recorded in centipoise (cps) units. The same procedure was used for the second set of conditions. The average value is reported. The results can be found in Table 3, “Results of Brookfield Viscosity Evaluation.”

Table 3 – Results of Brookfield Viscosity Evaluation

Sample ID	Spindle 4 Speed – 20 rpm		Spindle 5 Speed – 20 rpm	
	Initial	Post	Initial	Post
CC VIS-Strip	16,950 cps	No reading	10,300 cps	No reading
Sample ID	Spindle 4 Speed – 100 rpm		Spindle 5 Speed – 100 rpm	
	Initial	Post	Initial	Post
CC Fix	865 cps	820 cps	425 cps	380 cps

Density

Density was determined in accordance with ASTM D1475-98(08), "Standard Test Method for Density of Liquid Coatings, Inks, and Related Products." A calibrated cup was weighed empty and then weighed full of liquid coating. The air bubbles were eliminated from the coatings as much as feasible by gently tapping the cup. The liquid materials were tested prior to any exposure and then after the storage stability described in Cycle 3. Calculations were performed, and the results of the testing are listed in Table 4, "Density Results."

Table 4 – Density Results

Sample ID	Initial	After Storage Stability (Cycle 3)
CC VIS-Strip	8.978 lb/gal	8.988 lb/gal
CC Fix	8.668 lb/gal	8.668 lb/gal

Degree of Settling

The degree of settling was evaluated in accordance with ASTM D869-85(11), "Standard Test Method for Evaluating Degree of Settling of Paint." This method requires that the coating material not be shaken or stirred after exposure, prior to the settling evaluation. The blunt tipped spatula was dropped into the CC Fix and coating, the spatula hit the base of the can with a metallic sound, indicating that no settling had occurred in the 14 days of cyclic exposure represented in Cycle 2 – Storage Exposure, indicating a rating of 10 for the CC Fix product. Since the CC VIS-Strip product was semi-solid, no ASTM D869 rating could be applied.

Humidity Resistance

The coating system was applied over both concrete and steel panels and permitted to dry for a minimum of 7 days prior to testing. The resulting panels were tested for humidity resistance in accordance with ASTM D 2247-11, "Standard Practice for Testing Water Resistance of Coatings in 100% Relative Humidity." The coated panels were placed on the exterior of an enclosed chamber containing a heated, saturated mixture of air and water vapor. At an internal condition of 100°F and 100% relative humidity, the temperature difference between the specimen surface and the surrounding vapor causes the formation of condensation on the interior (painted surface) of the panels. The samples were subjected to 200 hours of humidity exposure. Visually, these samples appeared to be failing. There was discoloration noted of both coating materials and large areas of lifted coating were apparent (see photographs). After the exposure time had elapsed, the coated panels were evaluated for loss of tensile adhesion. The results are listed in the section titled Tensile Adhesion Strength. Photographs of the panels can be found in the Photographic Appendix.

Tensile Adhesion Strength

Tensile adhesion (pull-off strength) was measured in accordance with ASTM D4541-09e1, "Pull-Off Strength of Coatings Using Portable Adhesion Testers," Annex A4, "Self-Aligning Adhesion Tester Type IV" or ASTM D7234-12, "Standard Test Method for Pull-Off

Adhesion Strength of Coatings on Concrete Using Portable Pull-Off Adhesion Testers." Both of the methods use the same equipment to remove the pull stubs. The main difference between the test methods is the size of the pull stubs. The ASTM D7234-12 for concrete specifies 2″ pull stubs. The coating on the steel panels was evaluated using ½″ pull stubs.

For both methods, the testing surfaces were wiped clean and abraded gently using fine sandpaper. Pull stubs with an abraded test surface were attached to the coating using a two component epoxy adhesive (Araldite 2011), which was allowed to cure for a minimum of twenty-four hours at ambient laboratory conditions (72 ± 5°F and 50 ± 5% relative humidity). The 2″ pull stubs on the concrete samples were then detached using a self-aligning pneumatic adhesion tester (PATTI-Pneumatic Adhesion Tensile Testing Instrument) employing the F8 piston (range: 25-255 psi) or the F20 piston (range 63-637 psi). The ½″ pull stubs were detached using the F4 piston, which has a range of 202-2039 psi for the small diameter pull stub. The pressure required to remove each pull stub was recorded along with the location of break and approximate percentage of each. The location of break is defined as adhesive (a split between layers), cohesive (within a layer), or glue failure (coating strength exceeds glue strength). The results of the testing can be found in Table 5, "Results of Tensile Adhesion Testing." Photographs of the panels can be found in the Photographic Appendix.

Table 5 – Results of Tensile Adhesion Testing

Coating System	Pull Stub ID	Pull-Off Strength (psi)	Location of Break	Average Pull-Off Strength (psi)
CC VIS-Strip with CC Fix to Concrete (Initial)	1	324	100% adhesive at the concrete interface	323
	2	191	100% adhesive at the concrete interface	
	3	401	100% adhesive at the concrete interface	
	4	375	100% adhesive at the concrete interface	
CC VIS-Strip with CC Fix to Concrete (After 200 hours humidity)	1	71	100% adhesive to concrete	58 (average of 2 values)
	2	46	100% adhesive to concrete	
	3	*	100% adhesive to concrete	
	4	*	100% adhesive to concrete	
CC VIS-Strip with CC Fix to Steel (Initial)	A	1141	100% adhesive at the steel interface	1124
	B	1161	100% adhesive at the steel interface	
	C	1141	100% adhesive at the steel interface	
	D	1182	100% adhesive at the steel interface	
	E	1059	100% adhesive at the steel interface	
	F	1059	100% adhesive at the steel interface	

Coating System	Pull Stub ID	Pull-Off Strength (psi)	Location of Break	Average Pull-Off Strength (psi)
CC VIS-Strip with CC Fix to Steel (After 200 hours humidity)	A	284.2	100% adhesive at the steel interface, rusted	223 (average of 4 values)
	B	*	100% adhesive at the steel interface, rusted	
	C	*	100% adhesive at the steel interface, rusted	
	D	202.6	100% adhesive at the steel interface, rusted	
	E	202.6	100% adhesive at the steel interface, rusted	
	F	202.6	100% adhesive at the steel interface, rusted	

* *Pull stub removed as test started.*

Taber Abrasion Resistance

Taber abrasion resistance of the CC Fix material was determined in accordance with ASTM D4060-10, "Test Method for Abrasion Resistance of Organic Coatings by the Taber Abraser." Duplicate 4″ x 4″ panels were weighed then subjected to 1000 cycles using a 1000g load and CS-17 abrasion wheels. Post weights were acquired for the samples, and the weight loss (in mg) reported. The results of the testing are contained in Table 6, "Taber Abrasion Resistance Results."

Table 6 – Taber Abrasion Resistance Results

Coating System	Replicate ID	Initial Weight	Final Weight	Weight Loss (mg)	Average Weight Loss (mg)
CC Fix	A	65.0575	64.9883	69.2	71.0
	B	65.4911	65.4199	71.2	
	C	65.3762	65.3066	69.6	
	D	64.8932	64.8194	73.8	

Tensile Strength

Tensile strength was determined in accordance with ASTM D2370-98(10), "Standard Test Method for Tensile Properties of Organic Coatings." The samples were cut to a width of ½″ using a double blade cutter. The resulting samples were maintained at ambient laboratory conditions (72 ± 5° F and 50 ± 5% RH) for a minimum of 40 hours before testing. The samples were tested for tensile strength using a Tinius Olsen Universal Testing Machine. The cross-head speed used for testing was 2.0 in/min. A gage length of 1″ was selected due to the expected strength and elongation of the material and previous testing performed using this protocol. The force (in pounds) required to break the sample and the sample dimensions were used to calculate the tensile strength of each free-film of coating in pounds per square inch (psi). The ultimate length at break was used to calculate the elongation. The method requires testing of 10 specimens. The five specimens with the highest combination of properties were selected for reporting as outlined in the ASTM method. The results of the testing can be found in Table 7, "Results of Tensile Strength Testing."

Table 7 – Results of Tensile Strength Testing

Sample ID	Replicate ID	Force (lbs)	Tensile Strength (psi)	Average Tensile Strength (psi)	Percent Elongation (%)	Average Percent Elongation (%)
CC VIS-Strip with CC Fix	1	3.00	211	242	1144	1286
	2	3.34	246		1358	
	3	2.71	206		1704	
	5	2.99	251		1022	
	8	3.62	296		1201	

If you have any questions or comments regarding this report, please contact me by telephone at 412-788-1300, extension 183, or by email at vsherbondy@kta.com.

Sincerely,

KTA-TATOR, INC.

Valerie D. Sherbondy
Senior Chemist

Appendix: Photographs
VDS/RNR:kdw
JN320715-A3-R1
CIN: 205716

cc: Ms. Kim Egler of ARCADIS <*kim.egler@arcadis-us.com*>

R1 – A revision was issued to include edits and to add a photographic appendix.

(320715-A3-R1 Arcadis.doc)

APPENDIX

PHOTOGRAPHIC APPENDIX

VIS STRIP Cycle 1 (close) – Freeze/Thaw Stability

CC FIX Panels Humidity on Bottom

CC FIX System on Steel (Humidity Top) – Humidity

CC FIX over VIS Strip over Concrete – Adhesion (Humidity Panels on Bottom)

CC FIX with VIS Strip Over Steel – Adhesion

Appendix E - KTA Test Report – Instacote CC Strip/SBR-10

July 2, 2013
Via Email: *brent.hall@arcadis-us.com*

Mr. Brent Hall
ARCADIS U.S., Inc.
4915 Prospectus Drive, Suite F
Durham, NC 27713-4408

SUBJECT: Laboratory Testing of Strippable Coating – InstaCote SBR-10F;
KTA-Tator, Inc. Project No. 320715-A2-R2

Dear Mr. Hall:

In accordance with KTA Proposal PN131249 and ARCADIS U.S. Inc. (ARCADIS) Work Authorization No. D12-0178, KTA-Tator, Inc. (KTA) has tested a coating material to several different requirements to determine the resulting physical properties. This report describes the laboratory techniques used and contains the results of the testing.

SAMPLES

The samples listed in Table 1, "Samples" were received from InstaCote on the dates listed. It should be noted that at no time did KTA personnel witness the acquisition or manufacturing of the samples.

Table 1 – Samples

Sample ID	Sample Description	Date Received
KTA-6	One 5-gallon pail of InstaCote CC Hi VIS-Strip #1 VIS-Strip, Batch #012413-4-2	January 30, 2013
KTA-8	One gallon InstaCote SBR 10F with solvent, Part A, no batch number	March 6, 2013
	One gallon InstaCote SBR 10F with solvent, Part B Aspartic Ester, Resin, "Clear," no batch number	

LABORATORY INVESTIGATION

The laboratory investigation consisted of exposure of the liquid materials to different thermal cyclic conditions to simulate storage and freeze/thaw possibilities followed by evaluations of viscosity, density and settling. The applied coating system was tested to determine the resistance to humidity and the tensile strength (Table 5), while the topcoat was tested to determine the abrasion resistance (Table 6). A free-film sample of the coating system was also tested to determine the tensile strength (Table 7).

Thermal Cycling

Three different conditions of thermal cycling or exposure were evaluated for this testing protocol. Each condition is described in a paragraph below. The coatings were evaluated by different methods at the conclusion of the exposure. The results are addressed in the sections explaining the test methods employed for the evaluations. Each of the cyclic tests was assigned a number and a short designation for each is referenced in the corresponding results tables.

Cycle 1 – Freeze/Thaw Stability

Freeze/thaw thermal cycling was performed in accordance with ASTM D2337-01(2010), "Standard Test Method for Freeze-Thaw Stability of Multicolor Lacquers." This test method was performed to determine the resistance of the liquid coating material to cycles of freezing and thawing. The duration of the test was 30 cycles. The cycles occurred on weekdays. The can remained in the freezer during the weekends. The thermal cycling conditions for this test consisted of sixteen hours at 25 ± 3°F and eight hours at room temperature (72 ± 5°F). The liquid material was evaluated by Stormer viscosity after exposure. A visual examination of the coating revealed that the SBR-10F coating was similar in appearance to the unexposed samples. The InstaCote CC VIS-Strip was solid after exposure and a Stormer viscosity reading could not be obtained. A photograph of the coating material can be found in the Photographic Appendix.

Cycle 2 – Storage Exposure

Storage exposure was performed in accordance with ASTM D1309-93(2010), "Standard Test Method for Settling Properties of Traffic Paints During Storage." This test method was performed to determine the resistance of the liquid coating material to cycles of freezing and heating. The duration of the test was 14 cycles, which occurred on weekdays. The can remained in the at room temperature for the evenings and on the weekends. The thermal cycling conditions for this test consisted of two hours at 5 – 10°F, followed by two hours at approximately 160°F. These cycles were repeated twice, then followed by 16 hours at room temperature (72 ± 5°F). The 8 hour cycle was completed during work hours. The liquids were scheduled to be evaluated for settling after exposure. The VIS-Strip product was semi-solid after this exposure and a settling evaluation could not be performed. Components A and B of the InstaCote SBR 10F were liquid and the settling evaluation was performed.

Cycle 3 – Storage Stability

Storage stability was performed in accordance with military specification MIL-PRF-32239, "Standard Method for Accelerated Storage Stability." This test method was performed to determine the resistance of the water based liquid coating material to extended heat exposure. The can remained in the oven set at 140°F for the 7 day duration of the test. At the completion of the exposure, the liquid materials were scheduled for evaluation of viscosity using a Brookfield viscometer. Additionally, the density of the material was to be evaluated after exposure. A visual examination revealed that the majority of the VIS-Strip had partially solidified during this exposure and that the individual components of the SBR 10 appeared unaffected. There was enough liquid VIS-Strip material to perform the density testing, but not enough volume for viscosity testing.

Stormer Viscosity

Stormer viscosity was determined in accordance with ASTM D562-10, "Standard Test Method for Consistency of Paints Measuring Krebs Unit (KU) Viscosity Using a Stormer-Type Viscometer." Readings were taken once the samples were brought to 25 ± 0.2^{o}C, and recorded in Krebs units (KU). Baseline viscosity readings were obtained on liquid material that was not exposed to any of the conditions listed above. Initial readings were reported for the mixed material. Fifteen minutes after mixing, the viscosity was approximately 65.5 KU both before and after exposure. The results can be found in Table 2, "Results of Stormer Viscosity Evaluation."

Table 2 – Results of Stormer Viscosity Evaluation

Sample ID	Initial	After Freeze/Thaw (Cycle 1)
VIS-Strip	102.9 KU	Solid
SBR-10 Part A	49.6 KU	50.9 KU
SBR-10 Part B	90.7 KU	87.5 KU
SBR-10 Mixed	55.6 KU	55.0 KU

Brookfield Viscosity

The Brookfield viscosity was determined in accordance with ASTM D2196-10, "Standard Test Method for Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield type) Viscometer." The temperature of the sample was raised to 25 ± 0.2°C. Since previous testing for a similar protocol had been performed using spindle 4 and 20 RPM speed, this combination was utilized for the evaluation. These products did not produce acceptable results with that combination and the various combinations used for evaluation are listed below. For this testing, the spindle was lowered into the sample until the immersion mark on the spindle shaft touched the sample. The viscometer was turned on and the pointer was allowed to make several rotations until it had stabilized. Once the pointer stabilized, the viscometer was stopped by simultaneously pressing down on the power and hold switches. The viscosity reading was multiplied by the spindle/speed factor, and recorded in centipoise (cps) units. The same procedure was used for the second set of conditions. The average value is reported. The results can be found in Table 3, "Results of Brookfield Viscosity Evaluation."

Table 3 – Results of Brookfield Viscosity Evaluation

Sample ID	Spindle 4 Speed – 20 rpm		Spindle 5 Speed – 20 rpm	
	Initial	Post Cycle 3	Initial	Post Cycle 3
VIS-Strip (Initial)	16950 cps	no reading	5000 cps	no reading

Sample ID	Spindle 2 Speed – 50 rpm		Spindle 2 Speed – 100 rpm	
	Initial	Post Cycle 3	Initial	Post Cycle 3
SBR-10 Part A – After Storage Stability (Cycle 3)	60 cps	48 cps	60 cps	62 cps
Sample ID	Spindle 3 Speed – 50 rpm		Spindle 4 Speed – 50 rpm	
	Initial	Post Cycle 3	Initial	Post Cycle 3
SBR-10 Part B – After Storage Stability (Cycle 3)	1190 cps	1085 cps	1180 cps	1040 cps
Sample ID	Spindle 3 Speed – 100 rpm		Spindle 4 Speed – 100 rpm	
	Initial	Post Cycle 3	Initial	Post Cycle 3
SBR-10 Mixed – After Storage Stability (Cycle 3)	710 cps	230 cps	760 cps	200 cps

Density

Density was determined in accordance with ASTM D1475-98(08), "Standard Test Method for Density of Liquid Coatings, Inks, and Related Products." A calibrated cup was weighed empty and then weighed full of liquid coating. The air bubbles were eliminated from the coatings as much as feasible by gently tapping the cup. The liquid material was tested prior to any exposure and then after the storage stability described in Cycle 3. Calculations were performed, and the results of the testing are listed in Table 4, "Density Results."

Table 4 – Density Results

Sample ID	Initial	After Storage Stability (Cycle 3)
SBR-10 Part A	8.718 lb/gal	8.715 lb/gal
SBR-10 Part B	8.874 lb/gal	8.880 lb/gal
SBR-10 Mixed	8.836 lb/gal	8.806 lb/gal
VIS-Strip	8.978 lb/gal	8.988 lb/gal

Degree of Settling

The degree of settling was evaluated in accordance with ASTM D869-85(11), "Standard Test Method for Evaluating Degree of Settling of Paint." This method requires that the coating material not be shaken or stirred after exposure, prior to the settling evaluation. In this case, the CC VIS-Strip coating had solidified and no testing could be performed. Testing of the InstaCote SBR 10F Components A and B was performed. The spatula hit the base of the can with a metallic sound, indicating that no settling had occurred in the 14 days of cyclic exposure represented in Cycle 2 – Storage Exposure. The SBR-10F Components A and B would each receive a settling value of 10.

Humidity Resistance

The coating was applied over both concrete and steel panels and permitted to dry for a minimum of 7 days prior to testing. The resulting panels were tested for humidity resistance in accordance with ASTM D2247-11, "Standard Practice for Testing Water Resistance of Coatings in 100% Relative Humidity." The coated panels were placed on the exterior of an enclosed chamber containing a heated, saturated mixture of air and water vapor. At an internal condition of 100°F and 100% relative humidity, the temperature difference between the specimen surface and the surrounding vapor causes the formation of condensation on the interior (painted surface) of the panels. The samples were subjected to 200 hours of humidity exposure. The coating on the concrete panels had lightened and become more opaque in appearance. Visually, the coating on the steel samples appeared similar to when the samples were placed in exposure. After the exposure time had elapsed, the coated panels were evaluated for loss of tensile adhesion. The results are listed in the section titled Tensile Adhesion Strength. Photographs of the panels can be found in the Photographic Appendix.

Tensile Adhesion Strength

Tensile adhesion (pull-off strength) was measured in accordance with ASTM D4541-09e1, "Pull-Off Strength of Coatings Using Portable Adhesion Testers," Annex A4, "Self-Aligning Adhesion Tester Type IV" or ASTM D7234-12, "Standard Test Method for Pull-Off Adhesion Strength of Coatings on Concrete Using Portable Pull-Off Adhesion Testers." Both of the methods use the same equipment to remove the pull stubs. The main difference between the test methods is the size of the pull stubs. The ASTM D7234-12 for concrete specifies 2″ pull stubs. The coating on the steel panels was evaluated using ½″ pull stubs.

For both methods, the testing surfaces were wiped clean and abraded gently using fine sandpaper. Pull stubs with an abraded test surface were attached to the coating using a two component epoxy adhesive (Araldite 2011), which was allowed to cure for a minimum of twenty-four hours at ambient laboratory conditions (72 ± 5°F and 50 ± 5% relative humidity). The 2″ pull stubs on the concrete specimens were then detached using a self-aligning pneumatic adhesion tester (PATTI-Pneumatic Adhesion Tensile Testing Instrument) employing the F8 piston (range: 25 – 255 psi). The ½″ pull stubs were detached using the F4 piston, which has a range of 202 – 2039 psi for the small diameter pull stub. The pressure required to remove each pull stub was recorded along with the location of break and approximate percentage of each. The location of break is defined as adhesive (a split between layers), cohesive (within a layer), or glue failure (coating strength exceeds glue strength). The results of the testing can be found in Table 5, "Results of Tensile Adhesion Testing." Photographs of the panels can be found in the Photographic Appendix.

Table 5 – Results of Tensile Adhesion Testing

Coating System	Pull Stub ID	Pull-Off Strength (psi)	Location of Break	Average Pull-Off Strength (psi)
SBR-10/ VIS-Strip to Concrete (Initial)	1	203	100% adhesive between coats	223
	2	242	70% adhesive primer/topcoat; 30% adhesive to concrete	
	3	216	90% adhesive primer/topcoat; 10% adhesive to concrete	
	4	229	100% adhesive between coats	
SBR-10/ VIS-Strip to Concrete (After 200 hours humidity)	1	168	80% adhesive to concrete, 20% cohesive in coating	146
	2	117	85% adhesive to concrete, 15% cohesive in coating	
	3	163	90% adhesive to concrete, 10% cohesive in coating	
	4	137	60% adhesive to concrete, 40% cohesive in coating	
SBR-10 to Steel (Initial)	A	610.7	100% adhesive at the steel interface	584
	B	610.7	100% adhesive at the steel interface	
	C	610.7	100% adhesive at the steel interface	
	D	610.7	100% adhesive at the steel interface	
	E	569.9	100% adhesive at the steel interface	
	F	488.3	100% adhesive at the steel interface	
SBR-10 to Steel (After 200 hours humidity)	A	*	100% adhesive at the steel interface, rusted	513 (average of 5 values)
	B	508.7	100% adhesive at the steel interface, rusted	
	C	569.9	100% adhesive at the steel interface, rusted	
	D	549.5	100% adhesive at the steel interface, rusted	
	E	529.1	100% adhesive at the steel interface, rusted	
	F	406.7	100% adhesive at the steel interface, rusted	

* *Pull stub removed as test started.*

Taber Abrasion Resistance

Taber abrasion resistance of the SBR-10F was determined in accordance with ASTM D4060-10, "Test Method for Abrasion Resistance of Organic Coatings by the Taber Abraser." Duplicate 4″ x 4″ panels were weighed then subjected to 1000 cycles using a 1000g load and CS-17 abrasion wheels. Post weights were acquired for the samples, and the weight loss (in mg) reported. The results of the testing are contained in Table 6, "Taber Abrasion Resistance Results."

Table 6 – Taber Abrasion Resistance Results

Coating System	Replicate ID	Initial Weight	Final Weight	Weight Loss (mg)	Average Weight Loss (mg)
SBR-10	A	66.3012	66.2403	60.9	60.2
	B	65.2493	65.1860	63.3	
	C	66.5958	66.5353	60.5	
	D	66.5056	66.4495	56.1	

Tensile Strength

Tensile strength was determined in accordance with ASTM D2370-98(10), "Standard Test Method for Tensile Properties of Organic Coatings." The samples of the two coat system were cut to a width of ½″ using a double blade cutter. The resulting samples were maintained at ambient laboratory conditions (72 ± 5° F and 50 ± 5% RH) for a minimum of 40 hours before testing. The samples were tested for tensile strength using a Tinius Olsen Universal Testing Machine. The cross-head speed used for testing was 2.0 in/min. A gage length of 1″ was selected due to the strength and elongation of the material and the previous testing performed for this testing protocol. The force (in pounds) required to break the sample and the sample dimensions were used to calculate the tensile strength of each free-film of coating in pounds per square inch (psi). The ultimate length at break was used to calculate the elongation. The method requires testing of 10 specimens. The five specimens with the highest combination of properties were selected for reporting as outlined in the ASTM method. The results of the testing can be found in Table 7, "Results of Tensile Strength Testing."

Table 7 – Results of Tensile Strength Testing

Sample ID	Replicate ID	Force (lbs)	Tensile Strength (psi)	Average Tensile Strength (psi)	Percent Elongation (%)	Average Percent Elongation (%)
VIS-Strip with SBR-10	1	12.56	581	592	1144	1135
	2	13.01	602		1065	
	3	11.55	531		1065	
	5	13.05	585		1200	
	8	14.32	663		1124	

If you have any questions or comments regarding this report, please contact me by telephone at 412-788-1300, extension 183, or by email at vsherbondy@kta.com.

Sincerely,

KTA-TATOR, INC.

Valerie D. Sherbondy
Senior Chemist

Appendix: Photographs
VDS/RNR:kdw/jsc
JN320715-A2-R2
CIN: 205716

cc: Ms. Kim Egler of ARCADIS *<kim.egler@arcadis-us.com>*

R2 – A revision was issued to edit the photographic appendix.

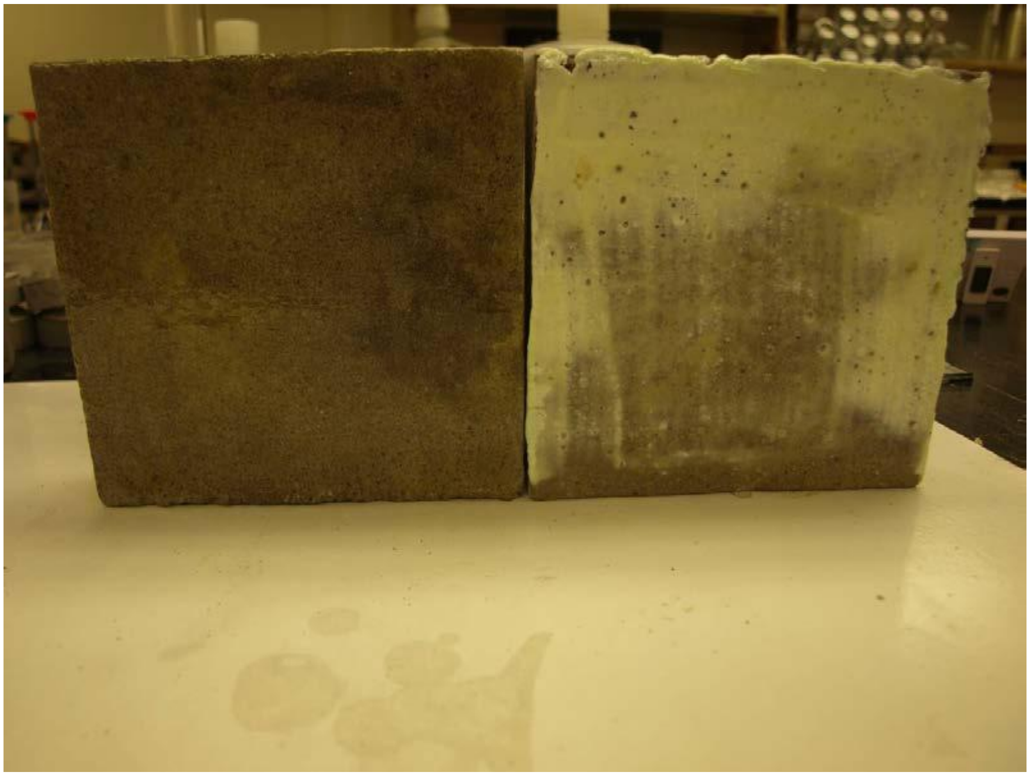
(320715-A2-R2 Arcadis.doc)

APPENDIX

PHOTOGRAPHIC APPENDIX

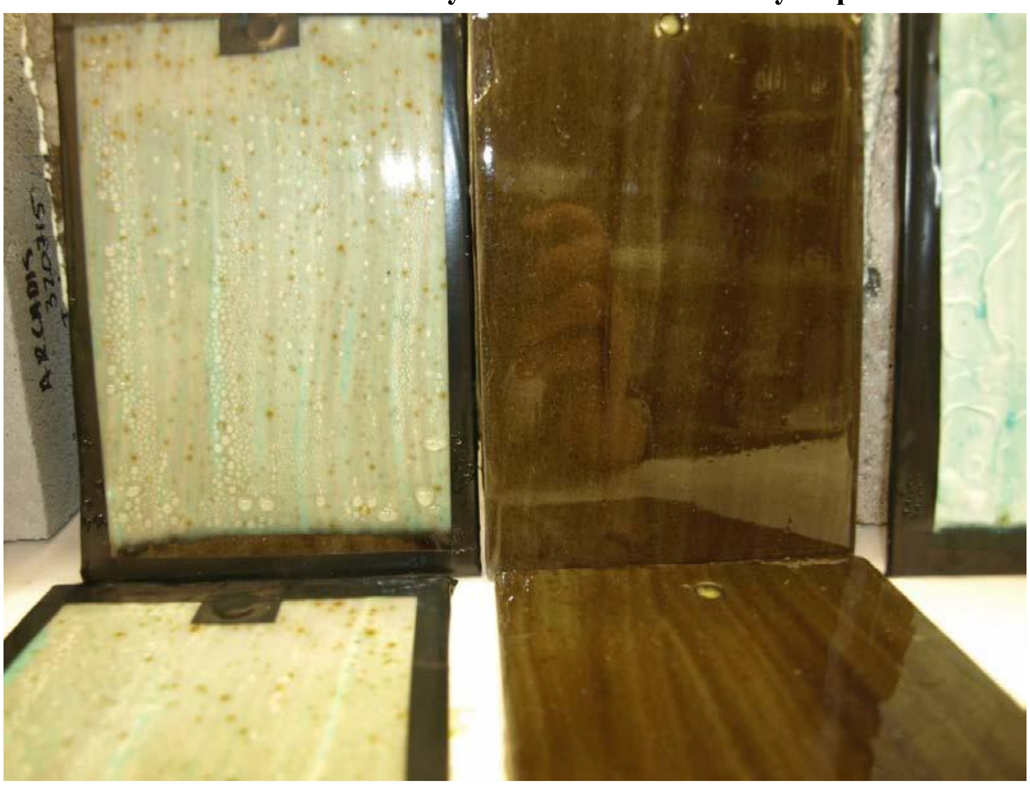
VIS STRIP Cycle 1 – Freeze/Thaw Stability

SBR-10F Panels Humidity vs Ambient – Humidity Exposure



SBR-10F Panels Humidity vs Ambient – Humidity Exposure (close view)

SBR-10F Steel – Humidity vs Ambient – Humidity Exposure



SBR-10F on Steel – Humidity vs Ambient – Humidity Exposure (Close View)

SBR-10WVIS Strip Over Concrete – Adhesion

SBR-10WVIS Strip Over Steel – Adhesion

Appendix F - KTA Test Report – Intergard 10220 Test Report

June 17, 2013
Via Email: *brent.hall@arcadis-us.com*

Mr. Brent Hall
ARCADIS U.S., Inc.
4915 Prospectus Drive, Suite F
Durham, NC 27713-4408

SUBJECT: Laboratory Testing of Strippable Coating – International Intergard 10220; KTA-Tator, Inc. Project No. 320715-A1-R2

Dear Mr. Hall:

In accordance with KTA Proposal PN131249 and ARCADIS U.S. Inc. (ARCADIS) Work Authorization No. D12-0178, KTA-Tator, Inc. (KTA) has tested a coating material to several different requirements to determine the resulting physical properties. This report describes the laboratory techniques used and contains the results of the testing.

SAMPLES

One five-liter can of International Intergard 10220, batch number 031004327, was received from ARCADIS on January 25, 2013. This sample was designated by KTA as Sample KTA-4. It should be noted that at no time did KTA personnel witness the acquisition or manufacturing of the samples.

LABORATORY INVESTIGATION

The laboratory investigation consisted of exposure of the liquid material to different thermal cyclic conditions to simulate storage and freeze/thaw possibilities followed by evaluations of viscosity, density and settling. The coating was tested to determine the resistance to humidity, tensile adhesion (Table 4) and abrasion (Table 5). A free-film was also prepared and tested to determine the tensile strength (Table 6).

Thermal Cycling

Three different conditions of thermal cycling or exposure were evaluated for this testing protocol. Each condition is described in a paragraph below. The coating was evaluated by different methods at the conclusion of the exposure. The results are addressed in the sections explaining the test methods employed for the evaluations. Each of the cyclic tests was assigned a number and a short designation for each is referenced in the corresponding results tables.

Cycle 1 – Freeze/Thaw Stability

Freeze/thaw thermal cycling was performed in accordance with ASTM D2337-01(2010), "Standard Test Method for Freeze-Thaw Stability of Multicolor Lacquers." This test method was performed to determine the resistance of the liquid coating material to cycles of freezing and thawing. The duration of the test was 30 cycles. The cycles occurred on weekdays. The can remained in the freezer during the weekends. The thermal cycling conditions for this test consisted of sixteen hours at 25 ± 3°F and eight hours at room temperature (72 ± 5°F). The liquid material was scheduled to be evaluated by Stormer viscosity after exposure. A visual examination of the coating at the conclusion of the cycling revealed that the coating was solid and Stormer viscosity could not be performed.

Cycle 2 – Storage Exposure

Storage exposure was performed in accordance with ASTM D1309 - 93(2010), "Standard Test Method for Settling Properties of Traffic Paints During Storage." This test method was performed to determine the resistance of the liquid coating material to cycles of freezing and heating. The duration of the test was 14 cycles, which occurred on weekdays. The can remained in the at room temperature in the evenings and on the weekends. The thermal cycling conditions for this test consisted of two hours at 5 – 10°F, followed by two hours at 160°F. These cycles were repeated twice, then followed by 16 hours at room temperature (72 ± 5°F). The 8 hour cycle was completed during work hours. The liquid was scheduled to be evaluated for settling after exposure. The immediate visual evaluation after cycling revealed that the coating material was solid and no settling evaluation could be performed.

Cycle 3 – Storage Stability

Storage stability was evaluated in accordance with military specification MIL-PRF-32239, "Standard Method for Accelerated Storage Stability." This test method was performed to determine the resistance of the water based liquid coating material to extended heat exposure. The can remained in the oven set at approximately 140°F for the 7 day duration of the test. At the completion of the exposure, the liquid material was evaluated for changes in viscosity using a Brookfield viscometer. Additionally, the density of the material was evaluated after the storage stability exposure. A visual examination of the material revealed that the coating was thicker than the original material, but still fluid.

Stormer Viscosity

Stormer viscosity was determined in accordance with ASTM D562-10, "Standard Test Method for Consistency of Paints Measuring Krebs Unit (KU) Viscosity Using a Stormer-Type Viscometer." Readings were taken once the samples were brought to 25 ± 0.2^{o}C, and recorded in Krebs units (KU). Baseline viscosity readings were obtained on liquid material that was not exposed to any of the conditions listed above. The sample exposed to the conditions of Cycle 1 was solid and a post evaluation of viscosity could not be performed. The results can be found in Table 1, "Results of Stormer Viscosity Evaluation."

Table 1 – Results of Stormer Viscosity Evaluation

Sample ID	Initial	After Freeze/Thaw (Cycle 1)
International 10220	138 KU	Solid

Brookfield Viscosity

The Brookfield viscosity was determined in accordance with ASTM D2196-10, "Standard Test Method for Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield type) Viscometer." The temperature of the sample was raised to 25 ± 0.2°C. No spindle number or speed was specified, so spindle 4 and 20 RPM speed were initially used to provide consistency with other materials tested under a similar protocol. This selection was not appropriate for this material, so the initial viscosity values were obtained using spindle 5 and a speed of 10 RPM, followed by spindle 6 at 20 RPM, for confirmation. For this testing, the spindle was lowered into the sample until the immersion mark on the spindle shaft touched the sample. The viscometer was turned on and the pointer was allowed to make several rotations, until it had stabilized. Once the pointer stabilized, the viscometer was stopped by simultaneously pressing down on the power and hold switches. The viscosity reading was multiplied by the spindle/speed factor, and recorded in centipoise (cps) units. The same procedure was used for the second set of conditions. The average value is reported. The results can be found in Table 2, "Results of Brookfield Viscosity Evaluation."

Table 2 – Results of Brookfield Viscosity Evaluation

Sample ID	Spindle 5 Speed – 10 rpm	Spindle 6 Speed – 20 rpm
International 10220 – Initial	30,000 cps	24,000 cps
International 10220 – After Storage Stability (Cycle 3)	Too thick to read*	41,250 cps

* *Spindle and speed adjustments did not lead to a comparable result.*

Density

Density was determined in accordance with ASTM D1475-98(08), "Standard Test Method for Density of Liquid Coatings, Inks, and Related Products." A calibrated cup was weighed empty and then weighed full of liquid coating. The air bubbles were eliminated from the coatings as much as feasible by gently tapping the cup. The liquid material was tested prior to any exposure and then after the storage stability described in Cycle 3. Calculations were performed, and the results of the testing are listed in Table 3, "Density Results."

Table 3 – Density Results

Sample ID	Initial	After Storage Stability (Cycle 3)
International 10220	9.082 lb/gal	8.650 lb/gal

Degree of Settling

The degree of settling was evaluated in accordance with ASTM D869-85(11), "Standard Test Method for Evaluating Degree of Settling of Paint." This method requires that the coating material not be shaken or stirred after exposure, prior to the settling evaluation. In this case, the coating exposed to Cycle 2 conditions had solidified and no ASTM D869 rating could be applied.

Humidity Resistance

The coating was applied over both concrete and steel panels and permitted to dry for a minimum of 7 days prior to testing. The resulting panels were tested for humidity resistance in accordance with ASTM D2247-11, "Standard Practice for Testing Water Resistance of Coatings in 100% Relative Humidity." The coated panels were placed on the exterior of an enclosed chamber containing a heated, saturated mixture of air and water vapor. At an internal condition of 100°F and 100% relative humidity, the temperature difference between the specimen surface and the surrounding vapor causes the formation of condensation on the interior (painted surface) of the panels. The samples were subjected to 200 hours of humidity exposure. Visually, the applications over steel appeared rusted, but did maintain a flat film. The applications to the concrete panels appeared unchanged. After drying, small cracks were noted in the coating. After the exposure time had elapsed, the coated panels were evaluated for loss of tensile adhesion. The results are listed in the section titled Tensile Adhesion Strength. Photographs of the panels can be found in the Photographic Appendix.

Tensile Adhesion Strength

Tensile adhesion (pull-off strength) was measured in accordance with ASTM D4541-09e1, "Pull-Off Strength of Coatings Using Portable Adhesion Testers," Annex A4, "Self-Aligning Adhesion Tester Type IV" or ASTM D7234-12, "Standard Test Method for Pull-Off Adhesion Strength of Coatings on Concrete Using Portable Pull-Off Adhesion Testers." Both of the methods use the same equipment to remove the pull stubs. The main difference between the test methods is the size of the pull stubs. The ASTM D7234-12 for concrete specifies 2″ pull stubs. The coating on the steel panels was evaluated using ½″ pull stub.

For both methods, the testing surfaces were wiped clean and abraded gently using fine sandpaper. Pull stubs with an abraded test surface were attached to the coating using a two component epoxy adhesive (Araldite 2011), which was allowed to cure for a minimum of twenty-four hours at ambient laboratory conditions (72 ± 5°F and 50 ± 5% relative humidity). The 2″ pull stubs on the concrete specimens were then detached using a self-aligning pneumatic adhesion tester (PATTI-Pneumatic Adhesion Tensile Testing Instrument) employing the F8 piston (range: 25 – 255 psi) or the F20 piston (Range 63 – 637 psi). The ½″ pull stubs were detached using the F4 piston, which has a range of 202 – 2039 psi for the small diameter pull stub. The pressure required to remove each pull stub was recorded along with the location of break and approximate percentage of each. The location of break is defined as adhesive (a split between layers), cohesive (within a layer), or glue failure (coating strength exceeds glue strength). The results of the testing can be found in Table 4, "Results of Tensile Adhesion Testing." Photographs of the panels can be found in the Photographic Appendix.

Table 4 – Results of Tensile Adhesion Testing

Coating System	Pull Stub ID	Pull-Off Strength (psi)	Location of Break	Average Pull-Off Strength (psi)
International 10220 to Concrete (Initial)	1	496	100% adhesive at the concrete interface	419
	2	254	100% adhesive at the concrete interface	
	3	455	100% adhesive at the concrete interface	
	4	471	100% adhesive at the concrete interface	
International 10220 to Concrete (After 200 hours humidity)	1	433	80% adhesive to concrete, 20% cohesive in coating	407
	2	407	85% adhesive to concrete, 15% cohesive in coating	
	3	445	90% adhesive to concrete, 10% cohesive in coating	
	4	344	60% adhesive to concrete, 40% cohesive in coating	
International 10220 to Steel (Initial)	A	1670	100% adhesive at the steel interface	1813
	B	1874	100% adhesive at the steel interface	
	C	1711	100% adhesive at the steel interface	
	D	1711	100% adhesive at the steel interface	
	E	2038	100% adhesive at the steel interface	
	F	1874	100% adhesive at the steel interface	
International 10220 to Steel (After 200 hours humidity)	A	1386	100% adhesive at the steel interface, rusted	1427
	B	1631	100% adhesive at the steel interface, rusted	
	C	1631	100% adhesive at the steel interface, rusted	
	D	1018	100% adhesive at the steel interface, rusted	
	E	1345	100% adhesive at the steel interface, rusted	
	F	1549	100% adhesive at the steel interface, rusted	

Taber Abrasion Resistance

Taber abrasion resistance was determined in accordance with ASTM D4060-10, "Test Method for Abrasion Resistance of Organic Coatings by the Taber Abraser." Duplicate 4″ x 4″ panels were weighed then subjected to 1000 cycles using a 1000g load and CS-17 abrasion wheels. Post weights were acquired for the samples, and the weight loss (in mg) reported. The results of the testing are contained in Table 5, "Taber Abrasion Resistance Results."

Table 5 – Taber Abrasion Resistance Results

Coating System	Replicate ID	Initial Weight	Final Weight	Weight Loss (mg)	Average Weight Loss (mg)
International 10220	A	65.1257	65.1182	7.5	11.4
	B	64.9805	64.9667	13.8	
	C	65.3345	65.3189	15.6	
	D	65.1114	65.1027	8.7	

Tensile Strength

Tensile strength was determined in accordance with ASTM D2370-98(10), "Standard Test Method for Tensile Properties of Organic Coatings." The samples were cut to a width of ½″ using a double blade cutter. The resulting samples were maintained at ambient laboratory conditions (72 ± 5° F and 50 ± 5% RH) for a minimum of 40 hours before testing. The samples were tested for tensile strength using a Tinius Olsen Universal Testing Machine. The cross-head speed used for testing was 2.0 in/min. A gage length of 1″ was selected due to the strength and elongation of the material and to provide consistency with other test results from a similar testing protocol. The force (in pounds) required to break the sample and the sample dimensions were used to calculate the tensile strength of each free-film of coating in pounds per square inch (psi). The ultimate length at break was used to calculate the elongation. The method requires testing of 10 specimens. The five specimens with the highest combination of properties were selected for reporting, as outlined in the ASTM method. The results of the testing can be found in Table 6, "Results of Tensile Strength Testing."

Table 6 – Results of Tensile Strength Testing

Sample ID	Replicate ID	Force (lbs)	Tensile Strength (psi)	Average Tensile Strength (psi)	Percent Elongation (%)	Average Percent Elongation (%)
International 10220	1	11.82	1876	1920	764	682
	2	13.13	1945		696	
	3	12.26	1946		658	
	5	12.37	1918		668	
	8	12.26	1916		628	

If you have any questions or comments regarding this report, please contact me by telephone at 412-788-1300, extension 183, or by email at vsherbondy@kta.com.

Sincerely,

KTA-TATOR, INC.

Valerie D. Sherbondy
Senior Chemist

Appendix: Photographs
VDS/RNR:kdw
JN320715-A1-R1
CIN: 205716

cc: Ms. Kim Egler of ARCADIS <*kim.egler@arcadis-us.com*>

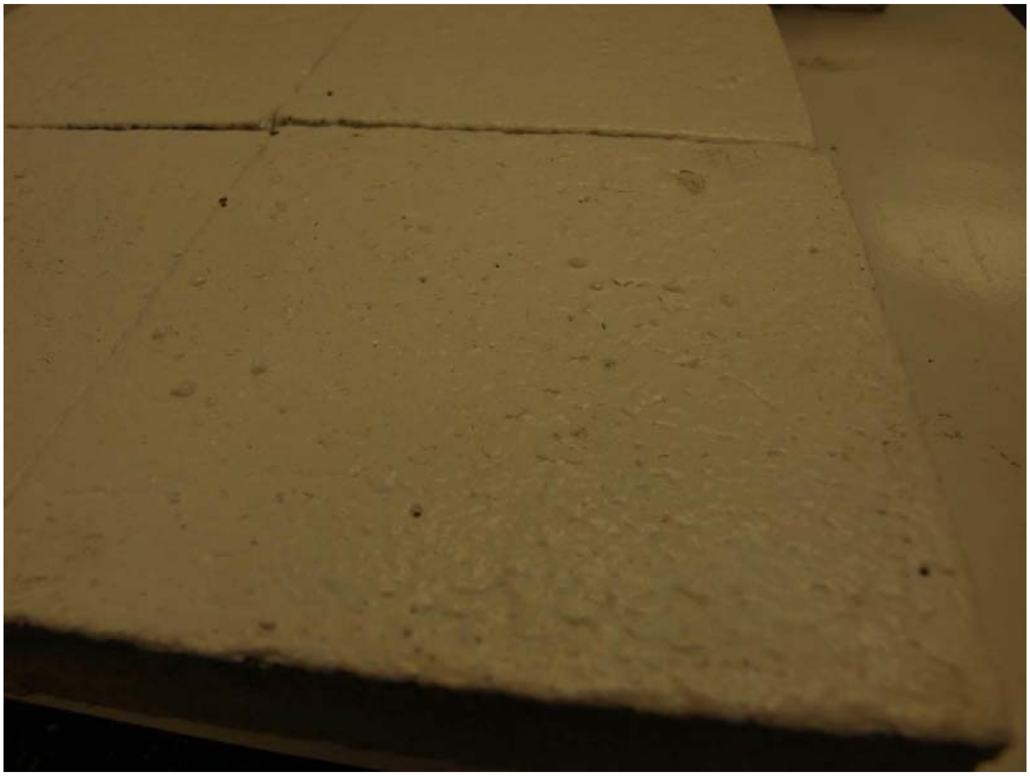
R1 – A revision was issued to correct the table reference in the Laboratory Investigation paragraph.

(320715-A1-R1 Arcadis.doc)

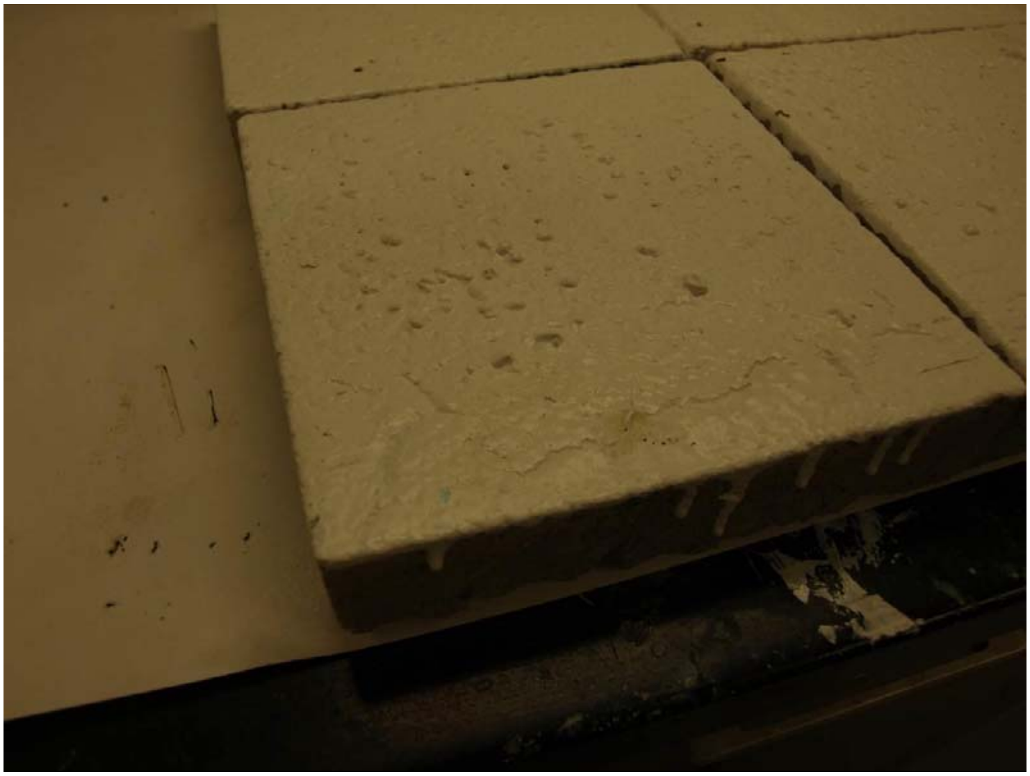
APPENDIX

PHOTOGRAPHIC APPENDIX

International 10220 Humidity vs Not – Humidity Exposure

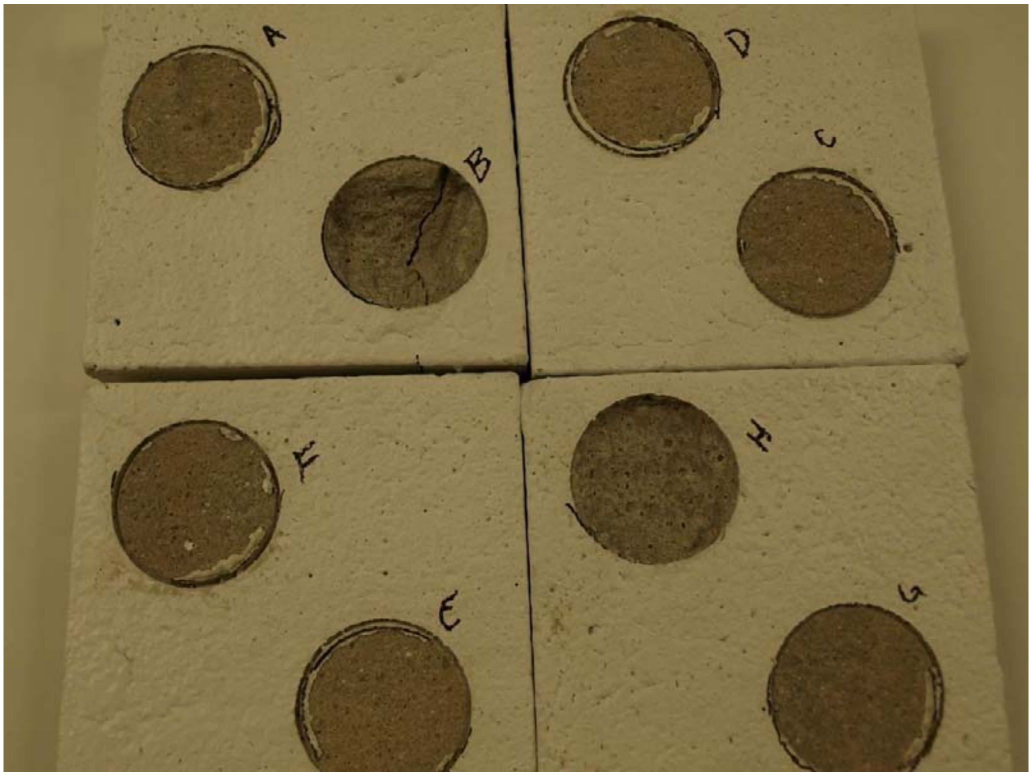


International 10220 Surface – Humidity Exposure

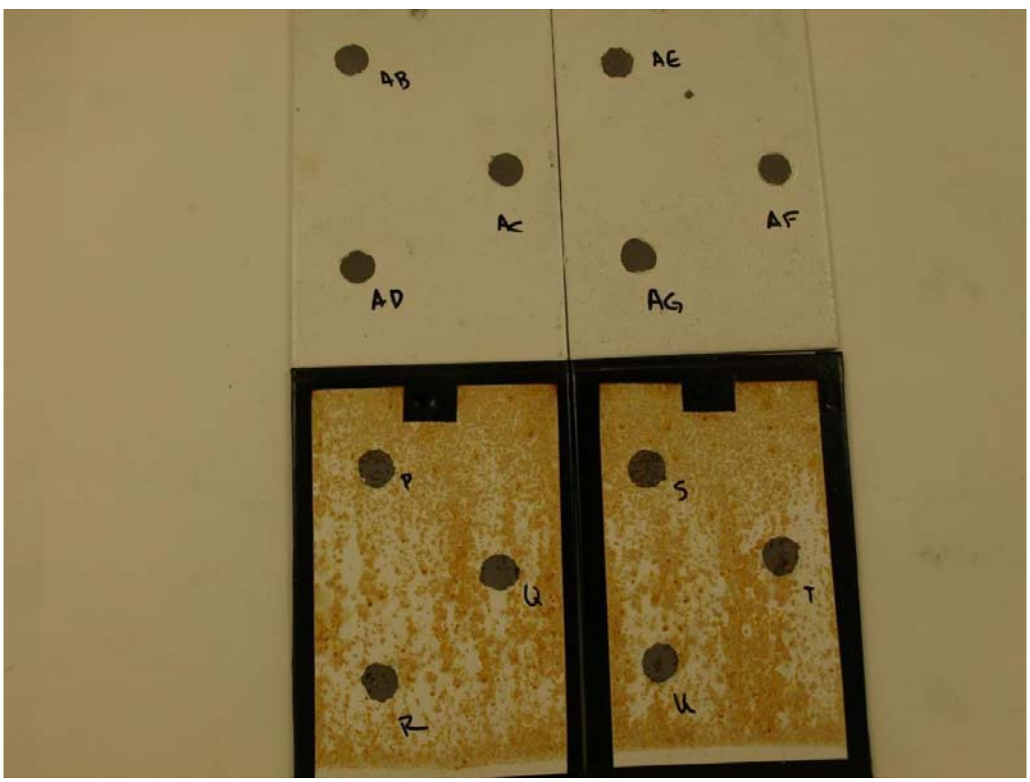


International Cracks from Humidity – Humidity Exposure

International 10220 Steel Humidity vs Not – Humidity Exposure



International 10220 Over Concrete – Adhesion



International 10220 Over Concrete – Adhesion

Appendix G - Data Quality Indicators

Appendix G - Data Quality Indicators

1.1 Data Quality Indicators

DQIs for the critical measurements were used to determine if the collected data met the QA objectives. The mean value of at least four replicate tests was used to assess pass/fail for that portion of the test. A list of the DQIs was given in Table 7-3. The DQIs established in the QAPP were selected without specific knowledge of the coatings and their characteristics. One objective of the validation of the test methods was to verify that these were realistic goals for the coatings being tested.

The equipment used to make the critical measurements was calibrated as per the requirements of the respective ASTM methods.

1.2 Assessment of DQI goals

The representativeness of the experiments was ensured by the careful selection of the concrete and steel coupons and experimental conditions. The parameters that were used to assess whether the data met the quality assurance objectives (as detailed in Table 7-3) include precision, accuracy, and completeness of the collected data.

Precision describes the closeness of data obtained using the same procedure over multiple experiments. There are three functions which are used to describe precision: standard deviation, variance, and coefficient of variance. The precision of a data set can be defined using the following equation:

$$\sigma = \sqrt{\frac{\sum_{i=1}^{N}(x_i - \mu)^2}{N}} \qquad \text{(G-1)}$$

where

N = the number of replicates in the data set,

x_i = the measured value in the data set, and

μ = the data set mean.

When applied to a smaller data set, a sample standard deviation is calculated which changes equation G-1 to the following:

$$s = \sqrt{\frac{\sum_{i=1}^{N}(x_i - \bar{x})^2}{N-1}} \qquad \text{(G-2)}$$

where

s = the sample standard deviation, and

x = the mean of the smaller data set.

Accuracy describes the closeness of the data to the true value. There are two functions frequently used to describe accuracy: absolute and relative error. Absolute error is the measured value minus the actual value, while the percent relative error is the same difference divided by the actual value and multiplied by 100.

The percent completeness of the data is simply the ratio of the number of valid data points taken to the total number of data points planned, multiplied by 100.

1.3 Shelf life DQI goals

All of the DQI goals related to shelf life, with the exception of settling properties, were based on viscosity and density DQI goals. These were the only measurements that had actual standards or instrument calibrations that could be used for the DQI assessment.

1.3.1 Density

Accuracy describes the closeness of the individual data point to the true value of the characteristic. Accuracy of the density measurement on the coatings was verified by determining the density of the ALARA 1146 coating using the specific gravity of the coating supplied by the manufacturer as the reference. As shown below, the accuracy was 3.0%, well below the DQI goal of a maximum of 15%.

ALARA 1146 calculated density	8.92 lb/gal (sp. gravity 1.07 × 8.34 lb/gal)
KTA-Tator measured density	8.65 lb/gal
Accuracy =	3.0%

The density cup used by the laboratory is verified every six months.

Precision of the density measurement is demonstrated by performing replicate measurements. The DQI goal for precision was 20%.. There were four density measurements performed for each of the four coatings. These measurements resulted in the following values for precision expressed as relative standard deviation (RSD), all of which were well within the precision goal of ±20%

ALARA 1146	0.01% RSD
Instacote SBR-10	0.29% RSD
Instacote CC Fix	0.01% RSD
Instacote CC Strip	0.01% RSD
Intergard 10220	0.02% RSD

Density measurements were 100% complete.

1.3.2 Viscosity

The Brookfield viscometer was calibrated January 26, 2011, and is accurate to within 1% of its full scale range when operated according to calibration conditions as specified in the Brookfield viscometer instruction manual. The calibration is verified every six months by KTA-Tator with a reference material with a known viscosity of 4960 cps. The most recent verification associated with these tests was performed in February 2013 and obtained values of 5200 cps and 5150 cps, both within the acceptance range of 5% set by ASTM D 2196. Precision for this measurement is represented by the percent difference between the replicate readings and is 1%, well within the DQI goal of 20%. Viscosity measurements were 100% complete for coatings that were not solidified during the test processes. The laboratory also took baseline readings of coating viscosity prior to exposure to any conditions.

The Stormer viscometer was verified on February 15, 2013, using a 73 KU (lot# 12201) and a 106 KU standard (lot# 11301). Verification was performed in duplicate to assess precision. Measured values for the 73.3 KU standard were 74.0 KU and 74.0 KU, well within the acceptance range of 73.3 KU ± 3.7 KU. Measured values for the 106.3 KU standard were 106.2 KU and 106.3 KU, well within the acceptance range of 106.3 KU ± 5.3 KU. All values were well within the DQI goals of 15% accuracy and 20% precision for a completeness of 100%.

1.3.3 Settling properties

No standards are available for determining the accuracy of this method. Precision can be demonstrated by doing replicate measurements but no DQI goals for precision were established. Settling properties were determined for all coatings that were not solid or semi-solid after cycles were completed so the completeness of this measurement was 100%.

1.4 Weatherability DQI goals

There are no standards that can be used to evaluate accuracy of these types of measurements. Evaluation of precision was limited to three characteristics related to weatherability where replicates are part of the method. These three characteristics are adhesion strength, abrasion resistance, and tensile strength. The DQI precision goal for each of these measurement parameters was 20%. Inability to meet this goal does not necessarily challenge the validity of the DQI goal, but rather sheds light on the potential shortcomings of the particular coating. This is information that would be taken into account by the end user when evaluating whether or not this coating is suitable for their intended use.

1.4.1 Water resistance

Panels were subjected to 200 hours of humidity exposure and tested in accordance with ASTM D2247-11. DQI goals in terms of accuracy and precision for water resistance were not established. This measurement was performed for all coatings and was 100% complete.

1.4.2 Tensile strength

Tensile strength was determined in accordance with ASTM D 2370-98 using a Tinius Olsen Universal Testing Machine. The method requires the testing of ten specimens and the five specimens with the highest combination of properties were selected for reporting. The accuracy DQI goal could not be assessed because there is no known standard. Precision between the five replicates has been assessed and compared to the established goal of 20%. This goal was met for tensile strength for all coatings and was therefore, 100% complete. The precision DQI goal was only slightly exceeded (20.5%) for Percent Elongation on the CC/Strip/CC Fix coating on concrete as can be seen in the assessment below.

- ALARA 1146 coating on concrete - the precision between five replicates for Tensile Strength testing and Percent Elongation was 4.6% RSD and 5.3% RSD, respectively.

- Intergard on concrete - the precision between five replicates for Tensile Strength testing and Percent Elongation was 1.5% RSD and 7.5% RSD respectively.

- CC Strip/CC Fix on concrete - the precision for five replicates for Tensile Strength was 15% RSD and 20.5% RSD for Percent Elongation.

- CC Strip/SBR 10 on concrete - the precision for five replicates for Tensile Strength was 8% RSD and 5.1% RSD for Percent Elongation.

1.4.3 Taber abrasion resistance

Taber abrasion resistance was determined in accordance with ASTM D4060-10. No known standards are available for assessing the accuracy of this method. Precision was assessed through replicate measurements and was met for three of the four coatings for a 75% completeness. This value does not meet the completeness goal of 90% established in the QAPP.

- ALARA 1146 coating on concrete – the precision between four replicates was 1.7% RSD.

- Intergard 10220 coating on concrete – the precision between four replicates was 34.3% RSD.

- CC Strip/CC Fix coating on concrete - the precision between four replicates was 2.9% RSD.

- CC Strip/SBR 10 coating on concrete - the precision between four replicates was 5% RSD.

1.4.4 Adhesion strength

Tensile adhesion (pull-off strength) of the coating on concrete and steel was measured in accordance with ASTM D4541-09 or ASTM D 7234-12. Both of the methods use the same equipment for testing, the only difference being the size of the pull stubs. Not standard is available for assessing the accuracy of this method. Precision was assessed by performing replicate tests and is detailed below. The DQI goal of 20% was exceeded for several of the coatings and may be an indication that the goal should be extended to account for the variability inherent in the method.

- ALARA 1146 coating on concrete pull off test for eight replicates was 6% precision initially and for seven replicates on the concrete after 200 hours in humidity, precision was 27.4% RSD. The pull off test for the ALARA 1146 on stainless steel panels of six (6) replicates was 6.8% RSD. The test could not be completed after the 200 hours humidity due to the panels being rusted.

- Intergard 10220 on concrete for four (4) replicates had a precision of 26.6% RSD. After 200 hours in humidity, the precision between four (4) replicates was 11.1% RSD. Intergard 10220 on stainless steel panels, six (6) replicates had a precision of 7.8% RSD and 16.4% RSD after 200 hour humidity for six (6) replicates.

- CC Strip/CC Fix on concrete for four (4) replicates initially had a 29% RSD precision. After 200 hours of humidity the precision was 30.2% RSD for duplicate tests. CC Strip/CC Fix on stainless steel panels, six (6) replicates initially had a precision of 4.7% RSD and for four (4) replicates after 200 hours humidity, precision was 18.3% RSD.

- CC Strip/SBR 10 on concrete for four (4) replicates initially had a 7.5% RSD precision. After 200 hours of humidity the precision was 16.3% RSD. CC Strip/SBR 10 on stainless steel panels, six (6) replicates had a precision of 8.5% RSD initially and five (5) replicates a 12.4% RSD precision.

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