

US EPA ARCHIVE DOCUMENT

Environmental Technology Verification Coatings and Coating Equipment Program (ETV CCEP)

UV-Curable Coatings – Generic Verification Protocol

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SI to English Conversions

SI Unit	English Unit	Multiply SI by factor to obtain English
°C	°F	1.80, then add 32
L	gal, liq (U.S.)	0.2642
m	ft	3.281
kg	lbm	2.205
kPa	psi	0.14504
cm	in.	0.3937
mm	mil (1 mil = 1/1000 in.)	39.37
m/s	ft/min	196.9
kg/L	lbm/gal, liq (U.S.)	8.345

List of Abbreviations and Acronyms

ACGIH	American Conference of Governmental Industrial Hygienists
ACS	American Chemical Society
ANSI	American National Standards Institute
AOAC	Association of Official Analytical Chemists
ASQC	American Society for Quality Control
ASTM	American Society for Testing and Materials
CCEP	Coatings and Coating Equipment Program
CTC	Concurrent Technologies Corporation
DFT	dry film thickness
DI	deionized
DOI	distinctness-of-image
EP	empty pan
EPA	U.S. Environmental Protection Agency
ES	empty syringe
ETF	Environmental Technology Facility
ETV	Environmental Technology Verification
FBO	Federal Business Opportunities (FedBizOpps.gov)
FS	full syringe
GVP	Generic Verification Protocol
HAP	hazardous air pollutant
ID	identification
IR	infrared
ISO	International Standardization Organization
MEK	methyl ethyl ketone
NDCEE	National Defense Center for Environmental Excellence
NIST	National Institute for Standards and Technology
P2	pollution prevention
PEA	Performance Evaluation Audit
PS	mass of pan with deposited solids
QA/QC	quality assurance/quality control
QMP	Quality Management Plan
RFT	Request for Technologies
RPD	Relative Percent Difference
RSD	Relative Standard Deviation
SOP	Standard Operating Procedures
SRM	standard reference material
TBD	to be determined
TQAPP	Testing and Quality Assurance Project Plan
UV	ultraviolet
VOC	volatile organic compound

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1.0 INTRODUCTION

1.1 Purpose of the UV-Curable Coatings Generic Verification Protocol

The primary purpose of this document is to establish the Generic Verification Protocol (GVP) for ultraviolet (UV)-curable coatings, to be referred to as the UV-Curable Coatings GVP. The secondary purpose is to establish the generic format and guidelines for product specific Testing and Quality Assurance Project Plans (TQAPPs) that relate to this GVP.

Environmental Technology Verification Coatings and Coating Equipment Program (ETV CCEP) pilot product-specific TQAPPs will establish the specific data quality requirements for all technical parties involved in each project. A defined format, as described below, is to be used for all ETV CCEP UV-Curable Coatings TQAPPs to facilitate independent reviews of project plans and test results, and to provide a standard platform for communicating with stakeholders and participants.

1.2 Quality Assurance for the ETV CCEP

Projects conducted under the auspices of the ETV CCEP will meet or exceed the requirements of the American National Standards Institute/American Society for Quality Control (ANSI/ASQC), Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs, ANSI/ASQC E-4 (1994) standard.¹ This GVP will ensure that project results are compatible with and complementary to similar projects. All ETV CCEP UV-Curable Coatings TQAPPs are adapted from this standard, the ETV Program Quality Management Plan (QMP), and the ETV CCEP QMP.^{2,3} These TQAPPs will contain sufficient detail to ensure that measurements are appropriate for achieving project objectives, that data quality are known, and that the data are reproducible and legally defensible.

1.3 Organization of the UV-Curable Coatings GVP

This GVP contains the sections outlined in the ANSI/ASQC E-4 standard. As such, this GVP identifies processes to be used, test and quality objectives, measurements to be made, data quality requirements and indicators, and procedures for the recording, reviewing and reporting of data.

The major technical sections discussed in this GVP are as follows:

- Project Description
- Project Organization and Responsibilities
- Quality Assurance (QA) Objectives
- Site Selection and Sampling Procedures
- Analytical Procedures and Calibration
- Data Reduction, Validation and Reporting
- Internal Quality Control (QC) Checks
- Performance and System Audits

- Calculation of Data Quality Indicators
- Corrective Action
- Quality Control Reports to Management
- Appendices

1.4 Formatting

In addition to the technical content, this GVP also contains standard formatting elements required by the ANSI/ASQC E-4 standard and Concurrent Technologies Corporation (CTC) deliverables. Standard format elements include, at a minimum, the following:

- Title Page
- TQAPP Approval Form
- Table of Contents
- Document Control Identification (in the plan header):
 - Section No. _____
 - Revision No. _____
 - Date: _____
 - Page: __ of __

1.5 Approval Form

Key ETV CCEP personnel will indicate their agreement and common understanding of the project objectives and requirements by signing the TQAPP Approval Form for each piece of equipment tested. Acknowledgment by each key person indicates commitment toward implementation of the plan. Figure 1 shows the Approval Form format to be used.

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2.0 PROJECT DESCRIPTION

2.1 General Overview

Organic finishing processes are used by many industries for the protection and decoration of their products. Organic coatings contribute nearly 20 percent of total stationary area source volatile organic compound (VOC) emissions as well as a significant percentage of air toxic emissions. Alternatives, such as UV-curable coatings, are continually being developed by many sources in an effort to reduce any detrimental effects to the environment. Often these UV-curable coatings are slow to penetrate the market because potential users, especially an ever-growing number of small companies, do not have the resources to test UV-curable coatings on their particular application and may be constructively skeptical of the UV-curable coating provider's claims. If an unbiased, third party facility could provide pertinent test data, environmentally friendlier coatings would penetrate the industry faster and accelerate environmental improvements. UV-curable coatings require a specific type of energy (i.e., light energy with a wavelength of approximately 400 nm, or in the UV spectrum) to initiate chemical cross-linking of the coatings components. Thermal energy alone may cause any volatiles to evaporate from the coating, but thermal energy alone will not cause the coating to cure. Typical curing equipment includes a source of UV light and a mechanism to convey the 'wet' surfaces past the UV source.

The ETV CCEP is a partnership between the U.S. Environmental Protection Agency (EPA) and the National Defense Center for Environmental Excellence (NDCEE) Program and is managed by CTC of Johnstown, PA. It has been established to provide unbiased, third party environmental performance data. The ETV CCEP has been tasked to develop, and subsequently utilize, a series of standardized protocols to verify the performance characteristics of coatings and coating equipment. This GVP enables verification of the performance of UV-curable coatings.

To maximize the ETV CCEP's exposure to the coatings industry, the data from the verification testing will be made available on the Internet at the EPA's ETV Program website (<http://www.epa.gov/etv/>) under the P2 Innovative Coatings and Coating Equipment Pilot as well as through other sources (e.g., publications, seminars). This will help establish the ETV CCEP's reputation in the private sector. A long-range goal of this initiative is to become a vital resource to the industry and, thus, self-sustaining through private support. This is in addition to its primary objective of improving the environment by rapidly introducing more environmentally friendly coating technologies into the industry.

2.1.1 Coating Application and Curing Test Location

CTC, through NDCEE, does not currently possess equipment to cure UV-reactive coatings. The coating application and curing of these materials must be conducted offsite. Regardless of the test location, arrangements will be made to ensure the requirements of the TQAPP, ETV CCEP QMP, and ETV Program QMP and all associated QA procedures are completed. ETV CCEP staff will conduct a site survey and pretest audit of the offsite test location and equipment to ensure that all the QA/QC requirements are met. The ETV CCEP

staff will also oversee all coating application and curing procedures, transport the standard test panels from the test location and CTC as needed, measure all process variables, conduct any offsite laboratory analyses, and package the standard test panels for transport to the NDCEE Environmental Coatings Laboratory. The ASTM D 5403 analysis of total volatile content will be used to determine the environmental impact of the UV-curable coating. The ETV CCEP personnel will determine processing volatiles at the offsite test location, but the determination of potential volatiles will be completed at CTC.

2.1.2 NDCEE Environmental Coatings Laboratory Facilities

In support of the ETV CCEP, the NDCEE's extensive state-of-the-art Environmental Coatings Laboratory facilities will be available to evaluate the cured standard test panels. Laboratory facilities available from the NDCEE are described in Table 1.

Table 1. Testing and Laboratories and Representative Laboratory Equipment Holdings

Laboratory	Focus	Laboratory Equipment
Environmental Testing	1) Identification and quantification of biological, organic, and inorganic chemicals and pollutants to all media. 2) Industrial process control chemical analysis.	Hewlett Packard 5972A GC/MS Varian Liberty 110 Sequential ICP P-E 4100ZL Graphite Furnace Mitsubishi GT06 Autotitrator P-E Headspace GC/ECD/FID TOC/Flashpoint/pH/Conductivity Graseby 2010 Isokinetic Stack Analyzer Graseby 2800 VOST Stack Sampler Questron Q-Wave 1000 Microwave Leeman PS200/AP200 Mercury Stations Millipore TCLP/ZHE Extraction Station Lachat Quickchem Flow Injection Analyzer
Destructive and Nondestructive Evaluation	Evaluation of product and process performance, and surface cleanliness.	Optically Stimulated Electron Emission X-ray/Magnetic/Eddy Current Thickness Salt Spray Corrosion Chamber Microhardness/Tensile/Fatigue/Wear
Materials and Mechanical Testing	Measurement of service and processing material and mechanical properties.	Noran and CAMScan Electron Microscopes Leco 2001 Image Analysis System Nikon and Polaroid Light Optical Microscopes EDAX Energy Dispersive Spectrometer Single Crystal Imaging Metallography Polishing/Grinding/Etching MTS Machines Tinius Olsen Testers Impact Testers
Powder Metallurgy	Investigation of powder properties.	Horiba LA900 Laser Particle Size Analyzer Autopore II 9020 Mercury Porosimeter Accupyc 1330 Pycnometer Gemini II 2370 Surface Area Analyzer
Intelligent Processing of Materials	Development and evaluation of embedded process sensors.	TEC Model 1600 Stress Analyzer Spectraphysics Argon & ND:YAg Lasers Resonance Frequency System
Risk & Environment Analysis	Management, monitoring, and evaluation of material and process alternatives from health and safety perspective.	Biosym: molecular modeling software MOPAC, Extend, HSC Chemistry, Riskpro, Sessoil, and GIS software packages
Calibration Laboratory	Calibration of equipment, sensors, and components to nationally traceable standards.	Transmation Signal Calibrator (milliamps,millivolts) Thermalcal Dry Block Calibrator (Temperature) Druck Pressure Calibrator (Pressure) Fluke Digital Multimeter (Voltage)

2.1.3 *Statement of Project Objectives*

The overall objective of the ETV CCEP is to verify pollution prevention (P2) characteristics and performance of coatings and coating equipment technologies, and to make the results of the verification tests available to prospective technology users. The ETV CCEP aspires to increase the use of more environmentally friendly technologies in products finishing in an effort to reduce emissions.

2.2 Technical/Experimental Approach and Guidelines

The following tasks are proposed for tests completed according to this GVP:

- Develop product-specific TQAPP
- Conduct verification and baseline (as needed) tests
- Prepare the Verification Report and Data Notebook
- Prepare the Verification Statement for approval and distribution

Table 2 describes the general guidelines and procedures that will be applied to each TQAPP.

Table 2. Overall Guidelines and Procedures Applied to this GVP

- A detailed description of each part of the test will be given.
- Critical and noncritical factors will be listed. Noncritical factors will be held constant throughout the testing. Critical factors will be listed as control (process) factors or response (coating product quality) factors.
- The product-specific TQAPPs will identify the testing site.
- The testing will be under the control and close supervision of ETV CCEP representatives to ensure the integrity of the third party testing.
- The QA portions of this GVP will be strictly adhered to.
- A statistically significant number of samples will be analyzed for each critical response factor. Variances (or standard deviations) of each critical response factor will be reported for all results.

2.2.1 *Test Approach*

The following approach will be used for this GVP:

- The vendor will identify the performance parameters to be verified and recommend the optimum equipment settings for application and curing;
- The ETV CCEP will obtain enough test panels for the verification and baseline tests;
- The ETV CCEP will obtain the baseline coatings (as appropriate);
- The vendor will provide the UV-curable coating being verified;

- Data such as dry film thickness (DFT), gloss, and visual appearance will be collected, following American Society for Testing and Materials (ASTM) methods, or equivalent (see Appendix B);
- A statistically valid test program that efficiently accomplishes the required objectives will then be used to analyze the test results.

2.2.2 *Verification Test Objectives*

The objectives of the verification tests performed per this protocol are to determine the total volatile content per ASTM D 5403 and to verify the quality and durability of UV-curable coatings. The coated test panels will be checked for DFT, visual appearance, and at least three of the following analyses: gloss, color, distinctness-of-image (DOI), adhesion, corrosion resistance (salt spray), direct impact resistance, flexibility (mandrel bend), pencil hardness, humidity resistance, weather resistance, abrasion resistance, and chemical resistance [methyl ethyl ketone (MEK) rub]. The cost associated with each analysis (except the mandatory DFT and visual appearance) will be presented to the participating vendors. The coating vendors will then choose which optional tests they want to have performed on the panels prepared using their coating. The coating vendor must choose a minimum of three optional tests. The total cost for completing each verification test and the vendor's share of that cost will depend on the number and type of analyses chosen. Additional pretreatment processes or tests that are either listed above or requested by the vendor may be included at the expense of the UV-curable coating vendor.

2.2.3 *Test Panels*

The actual test panels may be fabricated from steel, stainless steel, glass, plastic, alloys, wood, or composites based on the UV-curable coating vendor's recommendations. All steel panels will be commercially available and pretreated with zinc phosphate prior to coating application unless otherwise specified in the individual TQAPP. Details concerning panel characteristics, pretreatment, and pretreatment analysis will be identified in each product-specific TQAPP. The quality of any substrate pretreatment will be evaluated before shipment to the test site to ensure that the substrate panels meet specifications. However, the default standard test panel, as is shown in Appendix A, *Default Standard Test Panel*, will be 30.5 cm (12 in.) long and 10.2 cm (4 in.) wide with 0.6-cm (0.25-in.) hole punched in one end so that it may be suspended from a hook. Other parts may be treated and tested at the expense of the UV-curable coating vendor.

2.2.4 *Coating Specification*

The UV-curable coatings submitted for verification testing should provide an environmental benefit over the existing coatings currently in use in each UV-curable coating's target industry. The stakeholders group will also review the UV-curable coatings to determine their status as innovative coatings.

Each coating vendor will supply its test coating and respective specifications for the verification test. In addition, each vendor will supply a sufficient amount of coating to complete

the verification tests, the exact preparation instructions, and the instructions/parameters for applying the coating. The application procedures and conditions must be typical of the actual target industry.

2.2.5 *UV Curing Apparatus*

A suitable UV curing coating application apparatus, based on suggestions from the UV-curable coating provider, will be used to apply the UV-curable coating to test panels (and any other part requested by the coating provider in the TQAPP). A thickness range will be designated for each UV-curable coating as well as curing conditions.

Before the test, a set of dummy panels will be coated to ensure that the equipment parameters are set correctly. The fluid delivery pressure will be monitored periodically throughout the test. The paint usage may be determined through gravimetric means.

To help ensure proper equipment setup and operation, the UV-curable coatings vendors will be invited to participate in the startup phase of the testing and to observe the testing of their coatings. Each product-specific TQAPP will provide background to vendors for their test.

2.2.6 *Coating Baseline Test*

A coating baseline test may be performed for a coating that is submitted for verification as appropriate. The coating baseline will be used to determine the relative environmental and performance benefits of the UV-curable coating being verified. The coating baseline panels will also be evaluated for DFT, visual appearance, and the same optional tests chosen by the coating vendor for the verification test.

The coating baseline will use an existing coating and application method that is consistent with the verified technology's target industry. The coating baseline testing will be designed and performed by the ETV CCEP personnel. Certain operating parameters used for the coating baseline will be identical to the parameters used for the UV-curable coating verification test. Other parameters will be developed from the application equipment's or coating manufacturer's recommendations and experimental trials performed by the ETV CCEP.

2.2.7 *Design of Experiment*

This test protocol will verify the performance of UV-curable coatings submitted in response to the associated Federal Business Opportunities (FedBizOpps.gov) (FBO) notice or Request for Technologies (RFT). A mean value and variance (or standard deviation) will be reported for each critical response factor. If a UV-curable coating vendor makes a claim about a particular coating characteristic, the vendor of the coating will be asked to submit a confidence limit and specification limit (acceptable quality limit) for that claim for verification purposes. If the vendor does not submit a confidence and specification limit, a default 95% confidence limit will be applied. Any claims made by the coating vendor regarding particular coating characteristics will be used in the design of experiments. The appropriate number of test panels to be coated and analyzed is based on the confidence limit, specification limit, and the

appropriate statistical test to be applied to the results (i.e., Student's T-Test, Chi Square Test, or F-Test). Typically, as a default scenario, each verification test will consist of five runs with one rack of eight panels in a single row per run. The statistical analyses for all response factors will be carried out using Minitab statistical software.

Prior to the verification test, setup panels will be coated to ensure that the equipment parameters are correct. In actual verification testing, one panel per run will be removed for pretreatment analysis, and a predetermined number of panels (five runs with one rack of eight panels) will be coated to determine the P2 benefit and finish quality. Specifically, the standard test panels coated during the verification test will be analyzed for their chemical and physical properties as well as appearance.

If requested in the RFT or FBO response, the coating vendor can supply five additional parts to be coated during each verification test run. Fixturing of parts will be determined after the coating vendor submits parts, and vendors are bound by the part size and weight restrictions of the offsite test facility.

2.2.8 Performance Testing

UV-curable coating vendors will provide the ETV CCEP with coating specifications and appropriate equipment settings. The ETV CCEP will not attempt to optimize test settings during the actual test runs; however, the coating vendors will be given the opportunity to do so during the startup phase of the testing. The ETV CCEP will provide the UV-curable coating vendors with a list of key noncritical test factors that may affect the test results). Depending on the nature of the vendor's coating technology, this list may not address all of the factors that could impact the test results.

All testing will be conducted on the coated standard panels. All such tests will be performed per ASTM procedures and provide insight to the chemical and physical properties of the coatings. A comparison will be made from panel to panel and run to run.

2.2.9 Quantitative Measurements

In order to evaluate the environmental benefit and the finish quality obtained by using the UV-curable coating, several measurements will be taken on the coating, and noncoated and coated test panels. Coating samples will be analyzed for total volatile content, which includes VOCs and hazardous air pollutants (HAPs). Noncoated panels will be checked for surface area and pretreatment. Coated panels will be checked for DFT and visual appearance.

2.2.10 Participation

The vendor of the technology being verified is welcome to participate in the startup phase and observe the verification testing. The ETV CCEP personnel will be responsible for

performing all necessary tests and verifications required for performance evaluation. For safety purposes, the vendor staff may operate the UV curing equipment.

2.2.11 Critical and Noncritical Factors

In a designed experiment, critical and noncritical control factors must be identified. In this context, the term “critical” does not convey the importance of a particular factor. (Importance can only be determined through experimentation and characterization of the total process.) Rather, this term displays its relationship within the design of experiments. For the purposes of this protocol, the following definitions will be used for critical control factors, noncritical control factors, and critical response factors.

- Critical control factor – a factor that is varied in a controlled manner within a design of an experiment to determine its effect on a particular outcome of a system.
- Noncritical control factors – factors that remain relatively constant or are randomized throughout the testing.
- Critical response factors – the measured outcomes of each combination of critical and noncritical control factors used in the design of experiments.

In the case of the verification testing of a coating, there is only one critical control factor, and that is the coating itself. All other processing factors are noncritical control factors; therefore, the multiple runs and sample measurements within each run for each critical response factor will be used to determine the amount of variation expected for each critical response factor. For example, for each coating application, parameters associated with pretreatment would remain constant, and, thus, be noncritical control factors; however, a parameter, such as adhesion, would be identified as a critical response factor and could vary from run to run.

Tables 3 through 5 identify the factors to be monitored during testing as well as their acceptance criteria (where appropriate), data quality indicators, measurement locations, and measurement frequencies. The values in the “Total Numbers” column are based on the default test scenarios.

Table 3. Critical Control Factors

Critical Control Factor	Resin Type	Solvent Type	Cure Method	Target Industry
UV-Curable Coating	TBD	TBD	TBD	TBD

TBD – To be determined

Table 4. Noncritical Control Factors

Noncritical Factor	Set Points/ Acceptance Criteria	Measurement Location	Frequency	Total Number for the Test
Application Method (Manufacturer/Model)	From coating and equipment providers	Factory floor ^a	Once per test	1
Input Air Pressure to Gun or Pot	From coating provider	Factory floor	Once per test	1
Product Involved in Testing	Standard Test Plan (material TBD) ^b	Factory floor	Default scenario in Section 5.2	40 panels
Coating Delivery Pressure	From coating provider	Factory floor	Once per run	5
Pretreatment Analysis	Varies <1.2 g/m ²	Coatings laboratory	Once per run	5
Surface Area of Test Panels	TBD	Factory floor	Once per test	1
Ambient Factory Relative Humidity	Varies <10% During test	Factory floor	Once per run	5
Ambient Factory Temperature	Varies <5 °C during test	Factory floor	Once per run	5
Booth Relative Humidity	Varies <10% During test	Factory floor	Once per run	5
Booth Temperature	Varies <5 °C during test	Factory floor	Once per run	5
Spray Booth Airflow (Face Velocity)	0.4—0.6 m/s (80–120 ft/min)	Factory floor	Once per run	5
Temperature of Panels, as Coated	Varies <5 °C during test	Factory floor	Once per run	5
Distance to Panels	Varies <1.3 cm (<0.5 in.) during test	Factory floor	Once per test	1
Horizontal Gun Traverse Speed	TBD	Factory floor	Once per test	1
Vertical Drop Between Passes	TBD	Factory floor	Once per test	1
Volatile Content of Applied Coating	Varies <5% for each coating	Coatings laboratory	Once per run	5
Density of Applied Coating	Varies <50 g/L during test	Coatings laboratory	Once per run	5
Weight % Solids of Applied Coating	Varies <5% during test	Coatings laboratory	Once per run	5
Coating Temperature, as Applied	Varies <5 °C during test	Coatings laboratory	Once per run	5
Coating Viscosity, as Applied	Varies <5 seconds during test	Coatings laboratory	Once per run	5
Cure Time	TBD	Factory floor	Once per run	5

^a At offsite test facility

^b TBD – To be determined

2.2.12 Determination of Total Volatile Content of the UV-Curable Coating

This verification test will use ASTM D 5403, Test Method A, which will determine the Total Volatile Content by the following procedure:

- Test substrates will be heavy gage aluminum foil
- Test substrates will be in the shape of square pans with approximately 2 cm tall sides
- Test substrates will measure approximately 10 cm by 30 cm
- Weigh prepared test substrate (without coating) [A]
- Deposit UV-curable coating onto test substrates to a maximum of 1 mil wet film thickness using a syringe (minimum of 0.2 g)
- Weigh the coated test substrate [B]
- Cure coated substrate according to manufacturer's specifications
- Weigh the cured test substrate [C]
- Heat cured test substrate at 110 ± 5 °C for 60 minutes
- Weigh test substrate after cooling [D]

$$\% \text{ Processing Volatiles} = 100 \cdot [(B - C) / (B - A)]$$

$$\% \text{ Potential Volatiles} = 100 \cdot [(C - D) / (B - A)]$$

$$\text{Total Volatile Content} = \% \text{ Processing Volatiles} + \% \text{ Potential Volatiles}$$

2.3 Schedule

ETV CCEP uses standard tools for project scheduling. Project schedules are prepared in Microsoft Project. Project schedules show the complete work breakdown structure of the project, including technical work, meetings and deliverables. Table 6 shows the estimated schedule for the testing of UV-curable coatings.

Table 6. Estimated Schedule as of 9/26/2003

ID	Name	Duration	Start Date	Finish Date
Task 1	Approval of TQAPP	10 days	TBD	TBD
Task 2	Verification Testing	10 days	TBD	TBD
Task 3	Complete Data Analyses	20 days	TBD	TBD
Task 4	Prepare Verification Report	30 days	TBD	TBD
Task 5	Approval of Verification Report	30 days	TBD	TBD
Task 6	Issue Verification Statement	15 days	TBD	TBD

3.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

ETV CCEP, through its agreement with CTC, performs verification testing of environmentally beneficial technologies. The laboratory supports the ETV CCEP project manager and the ETV CCEP project leader by providing test data. Laboratory analysts report to the ETV CCEP laboratory leader. The ETV CCEP laboratory leader and organic finishing engineer coordinate with the ETV CCEP project leader on testing schedules. The ETV CCEP project leader is the conduit between the laboratory and the ETV CCEP project manager. The ETV CCEP project leader answers directly to the ETV CCEP project manager. For the ETV CCEP, the ETV CCEP project leader will be responsible for preparing the TQAPPs, Verification Report and Statement, and Data Notebook for each test.

The ETV CCEP QA officer, who is independent of both the laboratory and the program, is responsible for administering ETV and ETV CCEP QMP policies and CTC policies developed by its quality committee. These policies provide for, and ensure that quality objectives are met for each project. The policies are applicable to laboratory testing, factory demonstration processing, engineering decisions, and deliverables. The ETV CCEP QA officer reports directly to CTC senior management and is organizationally independent of the project or program management activities.

The project organization chart, showing lines of responsibility and the specific CTC personnel assigned to this project, is presented in Figure 4. A summary of the responsibilities of each CTC participant, his/her applicable experience, and his/her anticipated time dedication to the project during testing and reporting is given in Table 7.

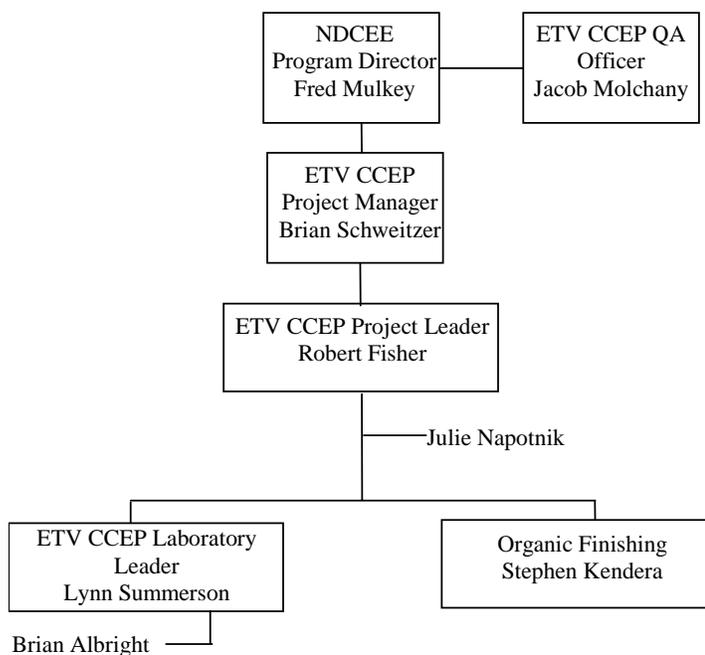


Figure 2. Project Organization Chart

Table 7. Summary of Current ETV CCEP Experience and Responsibilities

Key CTC Personnel and Roles	Responsibilities	Applicable Experience	Education	Time Dedication
Fred Mulkey – NDCEE Program Director	Manages NDCEE Program Accountable to CTC Technical Services Manager and CTC Corporate Management	Laboratory Chemist and Manager (15 years) Project Quality Assurance (15 years) Project Management (14 years) Registered Environmental Manager	M.S., Chemistry B.S., Chemistry	5%
Brian Schweitzer – Manager, Process Engineering/ ETV CCEP Project Manager	Responsible for overall ETV CCEP technical aspects, budget, and schedule issues on daily basis Accountable to NDCEE Program Director	Process Engineer (14 years) Project Manager, Organic Finishing (9 years)	B.S., Mechanical Engineering	25%
Jacob Molchany – ETV CCEP QA Officer	Responsible for overall project QA Responsible for establishing the QA audit checklist. Accountable to NDCEE Program Director	Industrial QA/QC and (14 years) Quality Mgmt. /ISO 9000 (8 years) Environmental Compliance and ISO 14000 Management Systems (8 years) Certified Hazardous Materials Mgr.	B.S., Industrial Engineering	5%
Robert Fisher – Staff Process Engineer/ ETV CCEP Project Leader	Technical project support Process design and development Accountable to ETV CCEP Project Manager Conducts site survey and oversees coating application / curing procedures.	Organic Finishing Regulations (9 years) Organic Finishing Operations (6 years) Professional Engineer	B.S., Chemical Engineering	50%
Julie Napotnik - Assistant Process Engineer/ ETV CCEP Project Team	Technical project support Process design and development Accountable to ETV CCEP Project Manager	Organic Coating Systems (3 years) Process Engineer (4 years)	B.S., Geo-Environmental Engineering	50%
Stephen Kendera – Sr. Organic Finishing Technician	Performs day-to-day operations of the Organic Finishing Line Accountable to Finishing Engineer	Industrial Paint and Coatings Experience (28 years)		10%
Lynn Summerson – ETV CCEP Laboratory Leader/Statistical Support Staff	Laboratory analysis / pre-test QA audit Accountable to ETV CCEP Project Manager	Industrial and Environmental Laboratory Testing (20 years)	M.S., Chemistry B.S., Chemistry	20%
Brian Albright – ETV CCEP Assistant Laboratory Analyst	QC Analysis Accountable to ETV CCEP Laboratory Leader	Environmental and QC Testing (7 years)	B.S., Chemistry	10%

The ETV CCEP personnel specified in Table 7 are responsible for maintaining communication with other responsible parties working on the project. The frequency and mechanisms for communication are shown in Table 8. In addition, the individuals listed in Table 9 will have certain responsibilities during the testing phase.

Each product-specific TQAPP will document the roles and responsibilities of offsite personnel.

Table 8. Frequency and Mechanisms of Communications

Initiator	Recipient	Mechanism	Frequency
NDCEE Program Director, ETV CCEP Project Manager, or ETV CCEP Project Leader	EPA ETV CCEP Project Manager	Written Report Verbal Status Report	Monthly Weekly
ETV CCEP Project Manager	NDCEE Program Director	Written or Verbal Status Report	Weekly
ETV CCEP Laboratory Leader	ETV CCEP Project Leader	Data Reports	As Generated
ETV CCEP Project Leader	ETV CCEP Project Manager	Written or Verbal Status Report	Weekly
ETV CCEP QA Officer	NDCEE Program Director	Quality Review Report	As Required
EPA ETV CCEP Project Manager	CTC	Onsite Visit	At Least Once per Year
Special Occurrence	Initiator	Recipient	Mechanism/ Frequency
Schedule or Financial Variances	NDCEE Program Director or ETV CCEP Project Manager	EPA ETV CCEP Project Manager	Telephone Call, Written Follow-up Report as Necessary
Major Quality Objective Deviation (will prevent accomplishment of verification cycle testing)	NDCEE Program Director or ETV CCEP Project Manager	EPA ETV CCEP Project Manager	Telephone Call with Written Follow-up Report

Table 9. Responsibilities During Testing

Position	Responsibility
ETV CCEP Project Manager	Overall coordination of project
ETV CCEP QA Officer	Audits of verification testing operations and laboratory analyses
ETV CCEP Project Leader	Overall coordination of testing, reporting, and data review
Statistical Support	Coordinates interpretation of test results

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4.0 QUALITY ASSURANCE OBJECTIVES

4.1 General Objectives

The overall objectives of this ETV CCEP GVP are to verify the performance of UV-curable coatings by establishing their environmental benefit and by documenting the applied coating's finish quality. These objectives will be met by controlling and monitoring the critical and noncritical factors, which are QA objectives for each technology-specific TQAPP based on this GVP. Tables 3 and 4 list the critical and noncritical control factors, respectively.

The analytical methods that will be used for coating evaluations are adapted from ASTM Standards, or equivalent. The QA objectives of the project and the capabilities of these test methods for product and process inspection and evaluation are synonymous because the methods were specifically designed for evaluation of the coating properties under investigation. The methods will be used as published, or as supplied, without major deviations unless noted otherwise. The specific methods to be used for this project are attached to this document as Appendix B (ASTM Methods).

4.2 Quantitative Quality Assurance Objectives

Quality assurance parameters such as precision and accuracy are presented in Tables 10 and 11. Table 10 presents the manufacturers' stated capabilities of the equipment used for measurement of noncritical control factors typically used by ETV CCEP. Control factors and equipment will be updated in product-specific TQAPPs should other equipment be used. The precision and accuracy parameters listed are relative to the true value that the equipment measures. Table 11 presents the precision and accuracy parameters for the critical response factors. The precision and accuracy are determined using duplicate analysis and known standards or spiked samples and must fall within the values found in the specific methods expressed.

The ETV CCEP will coordinate efforts to statistically interpret test results and QA objectives.

4.2.1 Accuracy

Standard reference materials, traceable to national sources such as the National Institute for Standards and Technology (NIST) for instrument calibration and periodic calibration verification, will be procured and utilized where such materials are available and applicable to this project. For reference calibration materials with certified values, acceptable accuracy for calibration verification will be within the specific guidelines provided in the method if verification limits are given. Otherwise, 80 to 120 percent of the true reference values will be used (see Tables 10 and 11). Reference materials will be evaluated using the same methods as for the actual test specimens.

Table 10. QA Objectives for Precision, Accuracy and Completeness for All Noncritical Control Factor Performance Analyses

Measurement	Method	Units	Precision	Accuracy	Completeness
Input Air Pressure to Gun or Pot	Pressure gauge	psig	± 0.5 psig	± 0.5%	90%
Product Involved in Testing	Test panels	N/A	N/A	N/A	100%
Coating Delivery Pressure	Pressure gauge	psig	± 0.5 psig	± 0.5%	90%
Pretreatment Analysis	ASTM B 767	g/m ²	± 0.005	± 0.01	90%
Surface Area of Test Panels	Ruler	cm ² (ft ²)	± 0.025 (± 0.0036)	± 0.025 (± 0.0036)	90%
Ambient Factory Relative Humidity	Thermal hygrometer	%	± 3% of full scale	± 3% of full scale	90%
Ambient Factory Temperature	Thermal hygrometer	°C	± 3% of full scale	± 3% of full scale	90%
Booth Relative Humidity	Thermal hygrometer	%	± 3% of full scale	± 3% of full scale	90%
Booth Temperature	Thermal hygrometer	°C	± 3% of full scale	± 3% of full scale	90%
Spray Booth Airflow (Face Velocity)	Per ACGIH	m/s (ft/min)	± 0.03* (± 5)	± 0.03* (± 5)	90%
Temperature of Panels, as Coated	Infrared (IR) thermometer	°C	± 0.13 °C	± 0.25 °C	90%
Distance to Panels	Ruler	cm (in.)	± 0.15 (± 0.06)	± 0.15 (± 0.06)	90%
Horizontal Gun Traverse Speed	Stopwatch	cm/s (in./s)	± 5%	± 5%	90%
Vertical Drop Between Passes	Ruler	cm (in.)	± 0.15 (± 0.06)	± 0.15 (± 0.06)	90%
Volatile Content of Applied Coating	ASTM D 3960	g/L (lb/gal)	± 0.6%	± 1.8%	90%
Density of Applied Coating	ASTM D 1475	g/L (lb /gal)	± 0.6%	± 1.8%	90%
Weight % Solids of Applied Coating	ASTM D 2369	%	± 1.5%	± 4.7%	90%
Coating Temperature, as Applied	Thermometer	°C	± 0.5 °C	± 0.2 °C	90%
Coating Viscosity, as Applied	ASTM D 1200	Seconds (#4 Ford Cup)	± 10%	± 10%	90%
Cure Time	Stopwatch	s	± 10%	± 10%	90%

ACGIH – American Conference of Governmental Industrial Hygienists, Inc.

* Accuracy and Precision stated by the manufacturer for velocities ranging from 20 to 100 ft/min

Table 11. QA Objectives for Precision, Accuracy and Completeness for All Critical Response Factor Performance Analyses

Measurement	Method	Units	Precision	Accuracy	Completeness
Total Volatile Content	ASTM D 5403, Method A	g/kg (lbm/lbm)	2.3% per ASTM D 5403	Not reported in ASTM D 5403	90%
Energy Usage of the UV Lamps	Calculated	KW	± 10%	± 10%	90%
Dry Film Thickness (DFT) – Magnetic	ASTM B 499	mils ^a	20%	10% true thickness	90%
Visual Appearance	N/A ^b	N/A	N/A	N/A	100%
Gloss	ASTM D 523	Gloss units	20% RPD ^c	± 0.3	90%
Color Spectrometer Spectral Light II	ASTM D 1729 ASTM D 2244	ΔE Values Visual	20% RPD N/A	± 0.2 ΔE N/A	90% 90%
Distinctness-of-Image (DOI)	ASTM D 5767 Method B	DOI units	20% RPD	± 3 DOI units	90%
Adhesion	ASTM D 3359	Pass/Fail and 0 to 5 rating	All pass or all fail	N/A	90%
Pencil Hardness	ASTM D 3363	H-B scale	N/A	N/A	90%
Corrosion Resistance (Salt Spray)	ASTM B 117	Pass/Fail	All pass or all fail	N/A	90%
Direct Impact	ASTM D 2794	Pass/Fail	All pass or all fail	Ranges listed in ASTM D 2794	90%
Mandrel Bend	ASTM D 522	Pass/Fail	All pass or all fail	± 15%	90%
Chemical Resistance [Methyl Ethyl Ketone (MEK) Rub]	ASTM D 5402	Visual	TBD by ASTM ^d	N/A	90%
Humidity Resistance	ASTM D 1735	Pass/Fail	All pass or all fail	N/A	90%
Weather Resistance	ASTM G 26	Pass/Fail	All pass or all fail	N/A	90%
Abrasion Resistance	ASTM D 4060	mg	46% RPD	Not reported in ASTM D 4060	90%

^a 1 mil = 0.001 in.

^b N/A – Not applicable

^c RPD – Relative Percent Difference

^d TBD – to be determined

4.2.2 Precision

The experimental approach of this GVP specifies guidelines for the number of test panels to be coated. The analysis of replicate test panels for each coating property at each of the experimental conditions will occur by design. The degree of precision will be assessed based on the agreement of all replicates within a property analysis group.

4.2.3 Completeness

The coating facility and laboratory strives for at least 90 percent completeness. Completeness is defined as the number of valid determinations expressed as a percentage of the total number of analyses conducted, by analysis type.

4.2.4 Impact and Statistical Significance Quality Objectives

All process/facility measurements and laboratory analyses will meet the accuracy and completeness requirements specified in Tables 10 and 11. The precision requirements also should be achieved; however, a nonconformance may result from the analysis of replicates due to limitations of the coating technology under evaluation, and not due to processing equipment or laboratory error. Regardless, if any nonconformance from TQAPP QA objectives occurs, the cause of the deviation will be determined by checking calculations, verifying the test and measurement equipment, and reanalysis. If an error in analysis is discovered, reanalysis of a new batch for a given run will be considered and the impact to overall project objectives will be determined. If the deviation persists despite all corrective action steps, the data will be flagged as not meeting the specific quality criteria and a written discussion will be generated.

If all analytical conditions are within control limits and instrument and measurement system accuracy checks are valid, the nature of any nonconformance may be beyond the control of the laboratory. If, given that laboratory quality control data are within specification, any nonconforming results occur, the results will be interpreted as the inability of the coating equipment undergoing testing to produce panels meeting the performance criteria at the given set of experimental conditions.

4.3 Qualitative QA Objectives: Comparability and Representativeness

4.3.1 Comparability

Participating technologies will be operated per the vendor's recommendations. The data obtained will be comparable from the standpoint that other testing programs could reproduce similar results using a specific TQAPP. Coating and environmental performance will be evaluated using EPA, ASTM, and other nationally or industry-wide accepted testing procedures as noted in previous sections of this GVP. Process performance factors will be generated and evaluated according to standard best engineering practices. In addition, vendors will be asked to provide performance data for their product and the results of preliminary or prior testing relevant to this GVP, if available.

The characteristics of test panels coated during these tests will be compared to the performance criteria and to other applicable end-user and industry specifications. The specifications will be used to verify the performance of the participating technology. Additional assurance of comparability comes from the routine use of precision and accuracy indicators as described above, the use of standardized and accepted methods and the traceability of reference materials.

4.3.2 *Representativeness*

The limiting factor to representativeness is the availability of a large sample population. An experimental design has been developed so that this project will either have sufficiently large sample populations or otherwise statistically significant fractional populations. The tests will be conducted at optimum conditions based on the manufacturers' and the coating vendors' literature and input and verified by setup testing. If the test data meet the quantitative QA criteria (precision, accuracy, and completeness) then the samples will be considered representative of the participating technology and will be used for interpreting the outcomes relative to the specific project objectives.

4.4 Other QA Objectives

There are no other QA objectives as part of this evaluation.

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5.0 SITE SELECTION AND SAMPLING PROCEDURES

5.1 Site Selection

Innovative coatings will be tested on large pilot-scale/small production-scale equipment, available at either the NDCEE facilities, at appropriate independent facilities, or the technology vendor's facilities. The following factors will be used to determine whether it is more beneficial to conduct a verification test at a non-NDCEE facility:

- (1) Lack of appropriate equipment at the NDCEE facilities, which also would not be cost-effective to acquire;
- (2) Ease of access to other facilities with proper equipment at reasonable cost;
- (3) Cooperative verifications [i.e., with the U.S. Army Environmental Center (USAEC)] with significant cost sharing; and
- (4) An expressed need from potential end users for testing conducted at an actual manufacturing site.

The necessary equipment for UV-curable coating is not currently available at the NDCEE. Therefore, an offsite location will be chosen that meets the requirements of this GVP, the ETV CCEP QMP, and the ETV Program's QMP. ETV CCEP staff will collect all relative test data during the coating application and curing operations at the offsite facility. Also, qualified ETV CCEP personnel will conduct any additional laboratory analyses that require the use of the curing equipment at the offsite facility. Test panels will be evaluated prior to application and after curing by ETV CCEP using the NDCEE facility.

5.2 Sampling Procedures and Handling

Test panels will be used in this project. These will be pre-labeled by marking their identification (ID) number with permanent marker on the untreated side of the test panels. The number of test panels processed during the testing depends on the experimental design, which in turn, depends on any equipment provider's claim(s) about performance characteristics and the respective confidence levels given in the responses to the RFT. If the UV-curable coating provider requests no specific performance characteristics for verification, the default experimental design will then be used. The default experimental design uses 40 panels for the test (8 panels per rack, 1 rack per run, and 5 runs per test).

A factory operations technician and laboratory analysts will process the test panels according to a preplanned sequence of stages identified in the product-specific TQAPP, which includes those identified in Table 12.

A laboratory analyst will record the date and time of each run and the time each measurement was taken. After curing, the test panels will be removed from the racks, separated by a layer of packing material, and stacked for transport to the laboratory. Sample custody documents will need to accompany the panels as they are transferred from the offsite processing

facility to the laboratory. The laboratory analyst will process the test panels through the laboratory login prior to performing the required analyses.

Table 12. Process Responsibilities

Procedure	Operations Technician	Laboratory Analyst
Visual Inspection of Test Panels		X
Numbering of Test Panels		X
Arrange Test Panels on the Racks		X
Prepare the Coating	X	
Setup the Application Equipment	X	
Take Coating Samples and Measurements		X
Load Coating	X	
Perform Setup Trials (before first run only)	X	
Apply Coating to Test Panels	X	
Take Process Measurements		X
Cure Test Panels	X	
Wrap/Stack/Transfer Test Panels to Lab	X	

Samples of the coating will be gathered prior to each run to determine the volatile content of the material. Samples that are to be transported back to the NDCEE ETF Laboratory will be packaged separately by run and analyzed as distinct batches. The coating samples will be packaged in a way that prevents exposure to ambient UV energy.

Panels that have been coated and cured will be packaged and transported to the NDCEE ETF Laboratory for analyses. All appropriate measures will be taken to assure that the applied coating is not damaged during transport. All custody changes will require that a custody log be completed and signed.

5.3 Sample Custody, Storage and Identification

The test panels will be given a unique laboratory ID number and logged into the laboratory record sheets. The analyst delivering the test panels will complete a custody log indicating the sampling point IDs, sample material IDs, quantity of samples, time, date, and analyst's initials. The test panels will remain in the custody of ETV CCEP, unless a change of custody form has been completed. The change of custody form should include a signature from ETV CCEP, the test product ID number, the date of custody transfer, and the signature of the individual to whom custody was transferred.

Laboratory analyses may only begin after each test product is logged into the laboratory record sheets. The laboratory's sample custodian will verify this information. Both personnel will sign the custody log to indicate transfer of the samples from the coating processing area or offsite location to the laboratory analysis area. The laboratory sample custodian will log the test panels into a bound record book; store the test panels under appropriate conditions (ambient room temperature and humidity); and create a work order for the various laboratory departments to initiate testing. The product evaluation tests also will be noted on the laboratory record sheet. Testing will begin within several days of coating application.

6.0 ANALYTICAL PROCEDURES AND CALIBRATION

6.1 Facility and Laboratory Testing and Calibration

ETV CCEP, in conjunction with the NDCEE, shall maintain a record of calibrations and certifications for all applicable equipment. Testing and measuring equipment shall be calibrated prior to the verification test and checked for accuracy after the verification test analyses are complete.

6.1.1 Facility Testing and Calibration

Calibration procedures for ETV CCEP within the NDCEE testing facility and laboratory shall be recorded. Certified solutions and reference materials traceable to NIST shall be obtained as appropriate to ensure the proper equipment calibration. Where a suitable source of material does not exist, a secondary standard is prepared and a true value obtained by measurement against a technical-grade NIST-traceable standard.

After the coating is mixed, the temperature and viscosity of the coating will be measured. In addition, coating samples will be taken to the lab for density and percent solids analyses. A listing of ASTM Methods can be found in Appendix B. All equipment used during facility testing is calibrated according to the appropriate criteria listed in Table 13.

Qualified ETV CCEP personnel will calibrate any equipment owned or operated by the offsite facility that will be used for these tests. The calibration results will be documented and incorporated into the laboratory report. An example of offsite equipment is a laboratory balance, which may be used in the determination of total volatile content of the UV-curable coatings.

6.1.2 Laboratory Testing and Calibration Procedures

The analytical methods performed for ETV CCEP at the NDCEE are adapted from standard ASTM, MIL-SPEC, EPA, Association of Official Analytical Chemists (AOAC) and industry protocols for similar manufacturing operations. Initial calibration and periodic calibration verification are performed to insure that an instrument is operating sufficiently to meet sensitivity and selectivity requirements. At a minimum, all equipment is calibrated before use and is verified during use or immediately after each sample batch. Standard solutions are purchased from reputable chemical supply houses in pure and diluted forms. Where certified and traceable to NIST reference materials and solutions are available, the laboratory purchases these for calibration and standardization. Data from all equipment calibrations and chemical standard certificates from vendors are stored in laboratory files and are readily retrievable. No samples are reported in which the full calibration curve, or the periodic calibration check standards, is outside method performance standards. As needed, equipment will be sent offsite for calibration or certification.

A listing of relevant ASTM Methods can be found in Appendix B. All equipment, used for these analyses, is calibrated according to Tables 13 and 14.

The ambient temperature and relative humidity is measured both inside and outside the spray booth. Also, the temperature of one product per run is measured prior to starting each test run.

All equipment used for these analyses will be calibrated according to Tables 13 and 14.

6.2 Product Quality Procedures

Each apparatus that will be used to assess the quality of a coating on a test product is set up and maintained according to each manufacturer's, or the published instructions of the reference method. Actual sample analysis will take place only after setup is verified against the reference method and the equipment manufacturer's instructions. As available, samples of known materials with established product qualities are used to verify that a system is functioning properly. For example, traceable thickness standards are used to calibrate the DFT instrument. Applicable ASTM methods are listed in Appendix B.

6.3 Standard Operating Procedures and Calibration

Tables 13 and 14 summarize the methods and calibration criteria that will be used for the evaluation of the coatings. Each analysis shall be performed as adapted from published methods and references, such as ASTM and EPA, and from accepted protocols provided by industrial suppliers.

Table 13. Noncritical Control Factor Testing and Calibration Criteria

Noncritical Factor	Method	Method Type	Calibration Procedure	Calibration Frequency	Calibration Acceptance Criteria ^a
Input Air Pressure	Factory gauge	Pressure gauge	Comparison to NIST-traceable standard	Six months	± 5 psig
Products Involved in Testing	Test panels	N/A ^b	N/A	N/A	N/A
Coating Delivery Pressure	Pressure gauge	Pressure gauge	Comparison to NIST-traceable standard	Six months	± 5 psig
Pretreatment Analysis	ASTM B767	Chromate solution (50g/L CrO ₃)	Comparison to NIST-traceable standard	With each use	80—120%
Surface Area of Each Product	Ruler	Ruler	Inspect for damage, replace if necessary	With each use	Lack of damage
Ambient Factory Relative Humidity	Thermal hygrometer	Thermal hygrometer	Sent for calibration or certification	Annually	Calibration or certification documentation
Ambient Factory Temperature	Thermal hygrometer	Thermal hygrometer	Sent for calibration or certification	Annually	Calibration or certification documentation
Spray Booth Relative Humidity	Thermal hygrometer	Thermal hygrometer	Sent for calibration or certification	Annually	Calibration or certification documentation
Spray Booth Temperature	Thermal hygrometer	Thermal hygrometer	Sent for calibration or certification	Annually	Calibration or certification documentation
Spray Booth Airflow (Face Velocity)	Per ACGIH ^c	Anemometer	Sent for calibration or certification	Annually	Calibration or certification documentation
Temperature of Test Panels, as Coated	Infrared (IR) thermometer	IR thermometer	Sent for calibration or certification	Annually	Calibration or certification documentation
Distance From Gun to Test Panels	Ruler	Ruler	Inspect for damage, replace if necessary	With each use	Lack of damage
Horizontal Gun Traverse Speed	Stopwatch	Stopwatch	Sent for calibration or certification	Six months	N/A
Vertical Drop Between Passes	Ruler	Ruler	Inspect for damage, replace if necessary	With each use	Lack of damage
Volatile Content of Applied Coating	ASTM D 3960	Volatile content	Comparison to NIST-traceable standard	With each use	± 0.003 g
Density of Applied Coating	ASTM D 1475	Weight	Comparison to NIST-traceable standard	With each use	± 0.003 g
Weight % Solids of Applied Coating	ASTM D 2369	Weight	Comparison to NIST-traceable standard	With each batch of coating	± 0.003 g
Coating Temperature, as Applied	Thermometer	Thermometer	Comparison to NIST-traceable standard	Annually	± 1 °C
Coating Viscosity, as Applied	ASTM D 1200	#4 Ford Cup	Comparison to NIST-traceable standard	Prior to each test	± 10%
Cure Time	Stopwatch	Stopwatch	Comparison to NIST-traceable standard	Annually	± 10%

^a As a percent recovery of a standard

^b N/A – Not applicable

^c ACGIH – American Conference of Governmental Industrial Hygienists, Inc.

Table 14. Critical Response Factor Testing and Calibration Criteria

Critical Measurement	Method Number ^a	Method Type	Calibration Procedure	Calibration Frequency	Calibration Acceptance Criteria ^b
Total Volatile Content	ASTM D 5403, Method A	Volatile content	Comparison to NIST-traceable standard	Each use	± 0.003 g
Energy Usage of the UV Lamps	Calculated	Calculated	N/A	N/A	N/A
Dry Film Thickness (DFT)	ASTM B 499	Magnetic	Comparison to NIST-traceable standard	Verify calibration after each run	90—110%
Visual Appearance	N/A ^c	Visual	N/A	N/A	N/A
Gloss	ASTM D 523	Gloss meter	Comparison to NIST-traceable standard	Verify calibration after each run	90—110%
Color Spectrometer Spectral Light II	ASTM D 1729 ASTM D 2244	Spectrometer Visual	Zero w/ white tile N/A	Each use N/A	N/A N/A
Distinctness-of-Image (DOI)	ASTM D 5767 Method B	Image analyzer	Manufacturer's recommendation	Manufacturer's recommendation	Manufacturer's recommendation
Adhesion	ASTM D 3359	Tape test	Verify condition of scribes and freshness of adhesives	Each use	N/A
Pencil Hardness	ASTM D 3363	Pencil	Supplier-graded lead (use same supplier)	Each use	N/A
Corrosion Resistance (Salt Spray)	ASTM B 117	Salt fog, 5% NaCl, neutral pH	Verify collection rate, pH, salinity, and bare steel corrosion rate	Weekly chemical tests, monthly steel tests	RSD ^d ≤20% among steel panels, average of chemical tests within specific ranges
Direct Impact	ASTM D 2794	2-pound weight	Verify weight of indenter, verify ruler	Yearly	80—120%
Mandrel Bend	ASTM D 522	Conical mandrel	Verify conical diameter	Yearly	80—120%
Chemical Resistance [Methyl Ethyl Ketone (MEK) Rub]	ASTM D 5402	MEK-saturated cheesecloth	Reagent grade MEK	N/A	N/A
Humidity Resistance	ASTM D 1735	100% Humidity using fog apparatus	Collection rate, pH	Daily collection rate and pH	Within ASTM ranges
Weather Resistance	ASTM G 26	Xenon arc w/ and w/o humidity	Irradiance, temperature, black panel, wet and dry bulb, wattage, water quality	Weekly	Within ASTM ranges
Abrasion Resistance	ASTM D 4060	Taber Abraser	Verify load weights	Each use	95—105%

^a Listing of ASTM methods to be used is provided in Appendix B.

^b As a percent recovery of a standard

^c N/A – Not applicable

^d RSD – Relative Standard Deviation

6.4 Nonstandard Methods

ETV CCEP and the offsite test facility does not plan to use any nonstandard methods for this project. However, for methods that are nonstandard (i.e., no commonly accepted or specified method exists or no traceable calibration materials exist), procedures will be performed according to the manufacturer's instructions or to the best capabilities of the equipment and the laboratory. This information will be documented. The performance will be judged based on the manufacturer's specifications, or will be judged based on protocols developed by the testing organization. These protocols will be similar or representative in magnitude and scope to related methods performed in the laboratory, which do have reference performance criteria for precision and accuracy. For instance, if a nonstandard quantitative chemical procedure is being performed, it should produce replicate results of ± 25 relative percent difference (RPD) and should give values within ± 20 percent of true or expected values for calibration and percent recovery check samples. For qualitative procedures, replicate results should agree as to their final evaluations of quality or performance (i.e., both should either pass or both should fail if sampled together from a properly functioning process). The intended use and any limitations would be explained and documented for a nonstandard procedure.

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7.0 DATA REDUCTION, VALIDATION, AND REPORTING

7.1 Raw Data Handling

Raw data will be generated and collected by the analysts at the bench or process level. Process data are recorded into a process log during factory operations. Bench data will include original observations, printouts, and readouts from equipment for sample, standard, and reference QC analyses. Data will be collected both manually and electronically. At a minimum, the date, time, sample ID, instrument ID, analyst ID, raw or processed signal, and qualitative observations will be recorded. The sample ID will be traceable from the raw data sheets through the summary sheets reported in the Data Notebook. Comments documenting unusual or nonstandard observations will also be included on the forms as necessary. The analyst will process raw data manually, automatically by an electronic program, or electronically after being entered into a computer. The analyst will be responsible for scrutinizing the data according to specified precision, accuracy, and completeness policies. Raw data bench sheets, calculations, and data summary sheets will be maintained for each sample batch. From the written standard operating procedures (SOPs) and raw data bench files, the steps leading to a final result may be traced.

7.1.1 Error in Solids Content

The solids content is the difference between two masses, the wet mass and the dry mass of the coating. The procedure specifies four measurements to be made, mass of the empty pan (EP), mass of the full syringe (FS), the mass of the empty syringe (ES), and the mass of the pan with the deposited solids (PS).

$$\%S = (PS - EP) / (FS - ES) \cdot 100$$

Since two measurements are made in the numerator and the denominator, the total uncertainty in each of these values is the sum of the uncertainties, or $2 \cdot 0.0005$ g. Since between 200 and 300 mg of coating is used in the test, this uncertainty becomes negligible compared to the numerator uncertainty. Only about 50 to 100 mg of solids are expected to remain in the pan after drying, making the numerator value uncertain by a maximum of 2%. Therefore, the solids content reported can be safely reported as within 2% of the actual value.

7.2 Preliminary Data Package Validation

A laboratory analyst will assemble a preliminary data package consisting of the data generated by the laboratory analysis. This package will contain the QC and raw data results, calculations, electronic printouts, conclusions and laboratory sample tracking information. The ETV CCEP laboratory leader will review the entire package and may also check sample and storage logs, standard logs, calibration logs, and other files, as necessary, to insure that tracking, sample treatments and calculations are correct. After the package has been peer reviewed in this manner, a preliminary data report will be prepared. The entire package and final laboratory report will be submitted by the ETV CCEP laboratory leader to the ETV CCEP project leader for incorporation into the Data Notebook.

7.3 Final Data Validation

The ETV CCEP laboratory leader shall be ultimately responsible for all final data released from this project. The ETV CCEP laboratory leader will review the final results for adequacy to project QA objectives. If the manager suspects an anomaly or nonconformance with expected or historical performance values, with project QA objectives, or with method specific QA requirements of the laboratory procedures, he will initiate a second review of the raw data and query the generating analyst about the nonconformance. Also, he will request specific corrective action. If suspicion about data validity still exists after internal review of laboratory records, the ETV CCEP laboratory leader may authorize a reanalysis. If sufficient sample is not available for retesting, a resampling will occur. If the sampling window has passed, or resampling is not possible, the ETV CCEP laboratory leader will flag the data as suspect and notify the ETV CCEP project leader. The ETV CCEP laboratory leader will sign and date the final data package and deliver it to the ETV CCEP project leader for review and incorporation into the Data Notebook.

7.4 Data Reporting and Archival

A report signed and dated by the ETV CCEP laboratory leader will be submitted to the ETV CCEP project manager, the ETV CCEP QA officer, the EPA ETV CCEP QA manager, and other technical principals involved in the project. The ETV CCEP project leader will incorporate any additional process information into the report prior to the ETV CCEP project manager's final review. The ETV CCEP project manager will decide on the validity of the data and will make any interpretations with respect to project QA objectives. The final laboratory report will contain the lab sample ID, date reported, date analyzed, the analyst, the procedures used for each parameter, the process or sampling point identification, the final result and the units. The NDCEE environmental laboratory will retain the data packages at least 10 years. The ETV CCEP project manager or the NDCEE program director will forward the results and conclusions to EPA in their regular reports for final EPA approval of the test data. This information will be used to prepare the Verification Report, which will be published by the ETV CCEP. The ETV CCEP, the vendor, the ETV CCEP Stakeholders, EPA technical peer reviewers, and the EPA Technical Editor will review the Verification Report. The EPA and the ETV CCEP will then approve the revised document prior to it being published.

7.5 Verification Statement

The ETV CCEP will also prepare a Verification Statement from the information contained in the Verification Report. After receiving the results and conclusions from the ETV CCEP project manager or the NDCEE program director, the EPA will approve the Verification Report and Verification Statement. Only after agreement by the vendor, will the Verification Statement be disseminated.

8.0 INTERNAL QUALITY CONTROL CHECKS

8.1 Guide Used for Internal Quality Program

ETV CCEP uses the NDCEE facility and its QA systems to verify coating technologies. The NDCEE has established an ISO 9001 operating program for its laboratories and the Demonstration Factory. The laboratory is currently establishing a formal quality control program for its specific operations. The format for laboratory QA/QC is being adapted from several sources as listed in Table 15. This QA system is consistent with the ETV QMP, the ETV CCEP QMP, and ANSI/ASQC guidelines.

Table 15. CTC Laboratory QA/QC Format Sources

Document	Reference Source
General Requirements for the Competence of Calibration and Testing Laboratories	ISO Guide 25, ISO Quality Programs
Critical Elements for Laboratories	Pennsylvania Department of Environmental Protection
Chapter One, Quality Control	SW-846, EPA Test Methods
Requirements of 100-300 series of methods	EPA Test Methods
Handbook of Quality Assurance for the Analytical Chemistry Laboratory, 2 nd Ed.	James P. Dux

8.2 Types of QA Checks

The NDCEE Environmental Technology Facility (ETF) Environmental Laboratory and Organic Finishing Line used by ETV CCEP follow published methodologies, wherever possible, for testing protocols. Laboratory and coating process methods are adapted from federal specifications, military specifications, ASTM Test Methods, and vendor instructions. The laboratory and finishing line adhere to the QA/QC requirements specified in these documents. In addition, where QA/QC criteria are not specified, or where the laboratory or finishing line perform additional QA/QC activities, these protocols are explained in the laboratory or finishing line's SOPs (Work Instructions). Each NDCEE facility that uses supplied products implements its own level of QA/QC. During ETV CCEP testing, the NDCEE laboratory and finishing line personnel will perform the testing and QA/QC verification outlined in Tables 10 and 11 (Precision, Accuracy, and Completeness) and Tables 13 and 14 (Calibration); therefore, these tables should be referred to for the method-specific QA/QC that will be performed.

8.3 Basic QA Checks

During each test, an internal Process QA Checklist will be completed by the laboratory and finishing line staff to ensure that the appropriate parts, panels, samples, and operating conditions are used. The laboratory also monitors its reagent DI water to ensure it meets purity levels consistent with analytical methodologies. The DI water filters are replaced quarterly before failures are encountered. The quality of the water is assessed with method reagent water blanks. Blank levels must not exceed minimum detection levels for a given parameter to be considered valid for use.

Thermometers are checked against NIST-certified thermometers at two temperatures. The laboratory checks and records the temperatures of sample storage areas, ovens, hot plate operations, and certain liquid baths that use thermometers.

Balances are calibrated by an outside organization using standards traceable to NIST. The ETF laboratory also performs in-house, periodic verifications with ASTM Class 1 weights. The ETF laboratory maintains records of the verification activities and calibration certificates. The laboratory analyst also checks the balances prior to use with ASTM Class 1 weights.

Reagents purchased directly by the laboratory are American Chemical Society (ACS) grade or better. Reagents are not used beyond their certified expiration dates. Reagents are dated on receipt and when first opened.

Laboratory waste is segregated according to chemical classifications in labeled containers to avoid cross-contamination of samples.

8.4 Specific Checks

The NDCEE Environmental Laboratory will analyze uncoated panels for DFT to verify that the instrument has not drifted from zero, perform duplicate analyses on the same samples, and perform calibration checks of the laboratory equipment during ETV CCEP testing. Laboratory personnel will also check any referenced materials and equipment as available and specified by the referenced methodology and the project-specific QA/QC objectives. Laboratory records are maintained with the sample data packages or in centralized files as appropriate. To ensure comparability, laboratory and finishing line personnel will carefully control process conditions and perform product evaluation tests consistently for each specimen. The specific QA checks listed in Tables 10, 11, 13, and 14 provide the necessary data to determine whether process control and product testing objectives are being met. ASTM, federal, and military methods that are accepted in industry for product evaluations and vendor-endorsed methods for process control, will be used for all critical measurements, thus satisfying the QA objective. A listing of the published methods that will be used for this GVP is included in Appendix B.

8.5 Offsite QA Checks

Several QA activities will be conducted at the offsite facility, including: a pre-test site visit, completion of a QA and calibration checklist, collection of calibration certificates, and performance audits on equipment to be used during the test. This information will be included in the laboratory report and Data Notebook. Equipment owned by the offsite facility that may be used during these tests consists of the UV lamps (energy usage), the conveyor system, and laboratory balances (total volatile analysis).

9.0 PERFORMANCE AND SYSTEM AUDITS

ETV CCEP uses the NDCEE facility and its QA systems to verify coating technologies. The NDCEE has developed a system of internal and external audits to monitor both program and project performance which are consistent with the audit requirements specified in the ETV and ETV CCEP QMPs. These include monthly managers meetings and reports, financial statements, EPA reviews and stakeholders meetings, and In-Process Reviews. The ETV laboratory also analyzes performance evaluation samples in order to maintain Pennsylvania Department of Environmental Protection Certification.

ISO Internal Audits

The NDCEE has established its quality system based on ISO 9000 and 14000 and has implemented a system of ISO internal audits. This information will be used for internal purposes.

Onsite Visits

The EPA ETV CCEP project manager may visit the NDCEE or the offsite test facility for an onsite visit during the execution of this project. All project, process, quality assurance, and laboratory testing information will be available for review.

EPA Audits

The EPA will periodically audit the ETV CCEP during this project. All project, process, quality assurance, and laboratory testing information will be made available per the EPA's auditing procedures.

Technical Systems Audits

A list of all coating equipment, laboratory measuring and testing devices, and procedures, coating procedures, and a copy of the approved ETV QMP and the approved ETV CCEP QMP will be given to the ETV CCEP QA officer. The ETV CCEP QA officer will conduct an initial audit, and additional audits thereafter according to the ETV CCEP QMP, of verification and testing activities. The NDCEE program director or the ETV CCEP project manager will forward a summary of the results of this activity to EPA.

Performance Evaluation Audits (PEAs)

The precision and accuracy of the measurement equipment will be examined to determine compliance with the product-specific TQAPPs. The auditor will evaluate measurements such as DFT and total volatile content. The ETV CCEP QA officer will conduct a PEA for each verification test. The NDCEE program director or the ETV CCEP project manager will forward a summary of the results of this activity to the EPA.

Audits of Data Quality

Peer review in the laboratory constitutes a process whereby two analysts review raw data generated at the bench level. After data are reduced, they undergo review by laboratory management. For this GVP, laboratory management will spot check 10 percent of the project data by performing a total review from raw to final results. This activity will occur in addition to the routine management review of all data. Records will be kept to show which data have been reviewed in this manner.

10.0 CALCULATION OF DATA QUALITY INDICATORS

10.1 Precision

Duplicates will be performed on separate samples as well as on the same sample source, depending on the method being employed. In addition, the final result for a given test may be the arithmetic mean of several determinations on the part or matrix. In this case, duplicate precision calculations will be performed on the means. The following calculations will be used to assess the precision between duplicate measurements.

$$\text{Relative Percent Difference (RPD)} = [(C1 - C2) \cdot 100\%] / [(C1 + C2) / 2]$$

where: C1 = larger of the two observations
C2 = smaller of the two observations

$$\text{Relative Standard Deviation (RSD)} = (s/y) \cdot 100\%$$

where: s = standard deviation
y = mean of replicates.

10.2 Accuracy

Accuracy will be determined as percent recovery of a check standard, check sample, or matrix spike. For matrix spikes and synthetic check samples:

$$\text{Percent Recovery (\% R)} = 100\% \cdot [(S - U)/T]$$

where: S = observed concentration in spiked sample
U = observed concentration in unspiked sample
T = true value of spike added to sample.

For standard reference materials (srm) used as calibration checks:

$$\% R = 100\% \cdot (C_m / C_{srm})$$

where: C_m = observed concentration of reference material
C_{srm} = theoretical value of srm.

10.3 Completeness

$$\text{Percent Completeness (\% C)} = 100\% \cdot (V/T)$$

where: V = number of determinations judged valid
T = total number of determinations for a given method type.

10.4 Project Specific Indicators

Process control limit: range specified by vendor for a given process parameter.

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11.0 CORRECTIVE ACTION

11.1 Routine Corrective Action

Routine corrective action will be undertaken in the event that a parameter in Tables 10, 11, 13, and 14 is outside the prescribed limits specified in these tables, or when a process parameter is beyond specified control limits. Examples of nonconformances include, but are not limited to, invalid calibration data, inadvertent failure to perform method-specific QA tests, process control data outside specified control limits, and failed precision or accuracy indicators. Such nonconformances will be documented on a standard laboratory or process/facility testing form. Corrective action will involve taking all necessary steps to restore a measuring system to proper working order and summarizing the corrective action and results of subsequent system verifications on a standard form. Some nonconformances will be detected while analysis or sample processing is in progress, and can be rectified in real time at the bench level. Other nonconformances may be detected only after a processing trial or sample analyses are completed. These types of nonconformances are typically detected at the ETV CCEP laboratory leader level of data review. In all cases of nonconformance, the laboratory leader will consider repeating the sample analysis as one method of corrective action. If a sufficient sample is not available, or the holding time has been exceeded, complete reprocessing may be ordered to generate new samples if a determination is made by the ETV CCEP project manager that the nonconformance jeopardizes the integrity of the conclusions to be drawn from the data. In all cases, a nonconformance will be rectified before sample processing and analysis continues. If corrective action does not restore the production or analytical system, causing a deviation from the ETV CCEP QMP, the ETV CCEP will contact the EPA ETV CCEP project manager. In cases of routine nonconformance, EPA will be notified in the NDCEE program director or ETV CCEP project manager's regular reports to the EPA ETV CCEP project manager. A complete discussion will accompany each nonconformance.

11.2 Nonroutine Corrective Action

While not anticipated, activities such as internal audits by the ETV CCEP QA officer, and onsite visits by the EPA ETV CCEP project manager, may result in findings that contradict deliverables in the ETV CCEP QMP. In the event that nonconformances are detected by bodies outside the laboratory organizational unit, as for routine nonconformances, these problems will be rectified and documented prior to processing or analyzing further samples or specimens.

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12.0 QUALITY CONTROL REPORTS TO MANAGEMENT

As shown on the Project Organization Chart in Figure 4, the ETV CCEP QA officer is independent from the project management team. It is the responsibility of the ETV CCEP QA Officer to monitor ETV CCEP verifications for adherence to the ETV CCEP QMP. The ETV CCEP laboratory leader monitors the operation of the laboratory on a daily basis and provides comments to the ETV CCEP QA officer to facilitate his activities. The ETV CCEP QA officer will audit the operation records, laboratory records, and laboratory data reports and provide a written report of the findings to the ETV CCEP project manager and laboratory leader. The ETV CCEP project manager will ensure these reports are included in the report to the EPA. The laboratory leader will be responsible for achieving closure on items addressed in the report. Specific items to be addressed and discussed in the QA report include the following:

- General assessment of data quality in terms of general QA objectives in Section 4.1
- Specific assessment of data quality in terms of quantitative and qualitative indicators listed in Sections 4.2 and 4.3
- Results of the site surveys and pretest audits of offsite testing locations and equipment listed in Section 2.1.1
- Listing and summary of all nonconformances and deviations from the ETV CCEP QMP
- Impact of nonconformances on data quality
- Listing and summary of corrective actions
- Results of internal QA audits
- Closure of open items from last report or communications with EPA in current reporting period
- Deviations or changes in the ETV CCEP QMP
- Progress of the NDCEE QA Programs used by ETV CCEP in relation to current project
- Limitations on conclusions, use of the data
- Planned QA activities, open items for next reporting period

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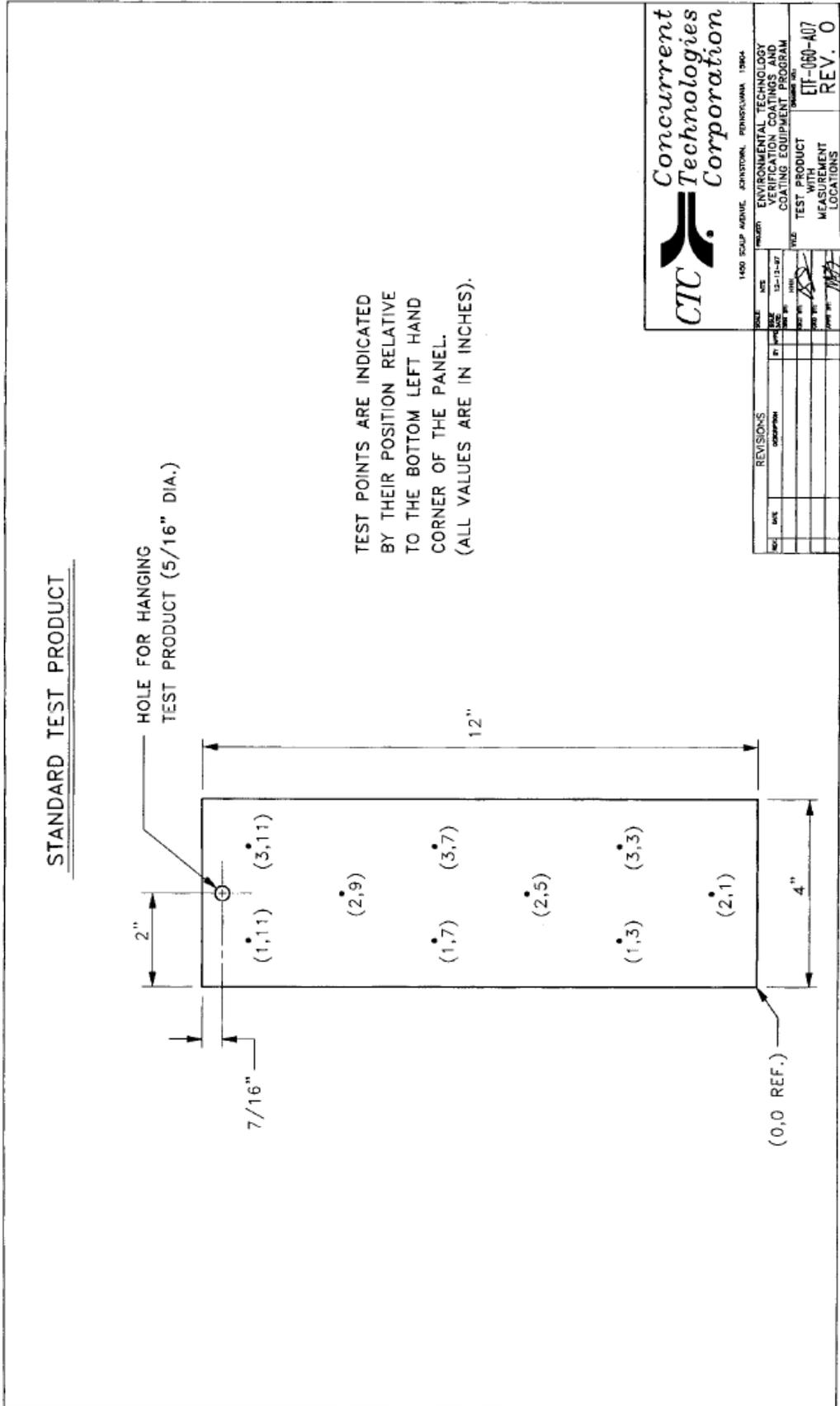
13.0 REFERENCES

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2. U.S. Environmental Protection Agency. Environmental Technology Verification Program: Quality Management Plan. EPA/600/R-03/021. December 2002. http://www.epa.gov/etv/pdfs/qmp/00_qmp_etv.html.
3. Concurrent Technologies Corporation. Environmental Technology Verification Coatings and Coating Equipment Program (ETV CCEP) Pilot: Quality Management Plan. December 21, 1998. http://www.epa.gov/etv/pdfs/qmp/06_qmp_p2.pdf.

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APPENDIX A

Default Standard Test Panel



APPENDIX B

ASTM Methods

ASTM Methods

- ASTM B 117 -- Standard Practice for Operating Salt Spray (Fog) Apparatus
- ASTM B 499 -- Standard Test Method for Measurement of Coating Thickness by the Magnetic Method: Nonmagnetic Coatings on Magnetic Basis Metals
- ASTM B 767 -- Standard Guide for Determining Mass per Unit Area of Electrodeposited and Related Coatings by Gravimetric and other Chemical Analysis Procedures
- ASTM D 522 -- Standard Test Methods for Mandrel Bend Test of Attached Organic Coatings
- ASTM D 523 -- Standard Test Method for Specular Gloss
- ASTM D 1200 -- Standard Test Method for Viscosity by Ford Viscosity Cup
- ASTM D 1475 -- Standard Test Method for Density of Liquid Coatings, Inks, and Related Products
- ASTM D 1729 -- Standard Practice for Visual Evaluation of Color Differences of Opaque Materials
- ASTM D 1735 -- Standard Practice for Testing Water Resistance of Coatings Using Water Fog Apparatus
- ASTM D 2244 -- Standard Test Method for Calculation of Color Differences from Instrumentally Measured Color Coordinates
- ASTM D 2369 -- Standard Test Method for Volatile Content of Coatings
- ASTM D 2794 -- Standard Test Method for Resistance of Organic Coatings to the Effects of Rapid Deformation (Impact)
- ASTM D 3359 -- Standard Test Method for Measuring Adhesion by Tape Test
- ASTM D 3363 -- Standard Test Method for Film Hardness by Pencil Test
- ASTM D 3960 -- Standard Practice for Determining Volatile Organic Compound (VOC) Content of Paints and Related Coatings
- ASTM D 4060 -- Standard Test Methods for Abrasion Resistance of Organic Coatings by the Taber Abraser
- ASTM D 5402 -- Assessing the Solvent Resistance of Organic Coatings Using Solvent Rubs
- ASTM D 5403 -- Standard Test Methods for Volatile Content of Radiation Curable Materials
- ASTM D 5767 -- Standard Test Methods for Instrumental Measurement of Distinctness-of-Image Gloss of Coating Surfaces
- ASTM G 26 -- Practice for Operating Light Exposure Apparatus (Xenon-Arc Type) With and Without Water for Exposure of Nonmetallic Materials