

US EPA ARCHIVE DOCUMENT

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

## **Liquid Coatings – Generic Verification Protocol**

### **Revision No. 0**

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## TABLE OF CONTENTS

	Page
SI TO ENGLISH CONVERSIONS.....	iv
LIST OF ABBREVIATIONS AND ACRONYMS.....	v
1.0 INTRODUCTION .....	1
1.1 Purpose of the Liquid Coatings Generic Verification Protocol.....	1
1.2 Quality Assurance for the ETV CCEP.....	1
1.3 Organization of the Liquid Coatings GVP .....	1
1.4 Formatting.....	2
1.5 Approval Form.....	2
2.0 PROJECT DESCRIPTION.....	5
2.1 General Overview .....	5
2.1.1 Demonstration Factory Testing Site .....	5
2.1.2 Laboratory Facilities .....	7
2.1.3 Offsite Testing .....	9
2.2 Technical/Experimental Approach and Guidelines .....	9
2.2.1 Test Approach.....	10
2.2.2. Verification Test Objectives .....	10
2.2.3 Test Panels .....	11
2.2.4 Coating Specification.....	11
2.2.5 Standard Apparatus.....	11
2.2.6 Coating Baseline Test .....	13
2.2.7 Design of Experiment .....	14
2.2.8 Performance Testing.....	14
2.2.9 Quantitative Measurements .....	15
2.2.10 Participation .....	15
2.2.11 Critical and Noncritical Factors.....	15
2.2.12 Determination of VOCs and HAPs.....	20
2.3 Schedule.....	21
3.0 PROJECT ORGANIZATION AND RESPONSIBILITIES.....	22
4.0 QUALITY ASSURANCE OBJECTIVES.....	26
4.1 General Objectives.....	26
4.2 Quantitative Quality Assurance Objectives.....	26
4.2.1 Accuracy .....	29
4.2.2 Precision.....	29
4.2.3 Completeness .....	29
4.2.4 Impact and Statistical Significance Quality Objectives.....	29
4.3 Qualitative QA Objectives: Comparability and Representativeness .....	30
4.3.1 Comparability .....	30
4.3.2 Representativeness.....	30
4.4 Other QA Objectives .....	30

5.0	SITE SELECTION AND SAMPLING PROCEDURES .....	32
5.1	Site Selection .....	32
5.2	Site Description.....	32
5.3	Sampling Procedures and Handling.....	32
5.4	Sample Custody, Storage and Identification.....	33
6.0	ANALYTICAL PROCEDURES AND CALIBRATION .....	36
6.1	Facility and Laboratory Testing and Calibration.....	36
6.1.1	Facility Testing and Calibration .....	36
6.1.2	Laboratory Testing and Calibration Procedures .....	36
6.2	Product Quality Procedures .....	37
6.3	Standard Operating Procedures and Calibration.....	37
6.4	Nonstandard Methods .....	40
7.0	DATA REDUCTION, VALIDATION, AND REPORTING .....	42
7.1	Raw Data Handling.....	42
7.1.1	Error in Solids Content. ....	42
7.2	Preliminary Data Package Validation.....	42
7.3	Final Data Validation.....	43
7.4	Data Reporting and Archival .....	43
7.5	Verification Statement .....	44
8.0	INTERNAL QUALITY CONTROL CHECKS .....	46
8.1	Guide Used for Internal Quality Program.....	46
8.2	Types of QA Checks .....	46
8.3	Basic QA Checks .....	46
8.4	Specific Checks.....	47
9.0	PERFORMANCE AND SYSTEM AUDITS .....	48
10.0	CALCULATION OF DATA QUALITY INDICATORS .....	50
10.1	Precision.....	50
10.2	Accuracy .....	50
10.3	Completeness .....	50
10.4	Project Specific Indicators .....	50
11.0	CORRECTIVE ACTION .....	52
11.1	Routine Corrective Action .....	52
11.2	Nonroutine Corrective Action .....	52
12.0	QUALITY CONTROL REPORTS TO MANAGEMENT .....	54
13.0	REFERENCES .....	56

## LIST OF TABLES

Table 1.	Testing and Laboratories and Representative Laboratory Equipment Holdings.....	8
Table 2.	Overall Guidelines and Procedures Applied to this GVP.....	10
Table 3.	Critical Control Factors .....	16
Table 4.	Noncritical Control Factors .....	17
Table 5.	Critical Response Factors .....	18
Table 6.	Estimated Schedule as of 9/24/03 .....	21
Table 7.	Summary of Current ETV CCEP Experience and Responsibilities .....	23
Table 8.	Frequency and Mechanisms of Communications .....	24
Table 9.	Responsibilities During Testing.....	24
Table 10.	QA Objectives for Precision, Accuracy and Completeness for All Noncritical Control Factor Performance Analyses .....	27
Table 11.	QA Objectives for Precision, Accuracy and Completeness for All Critical Response Factor Performance Analyses.....	28
Table 12.	Process Responsibilities.....	33
Table 13.	Noncritical Control Factor Testing and Calibration Criteria.....	38
Table 14.	Critical Response Factor Testing and Calibration Criteria.....	39
Table 15.	CTC Laboratory QA/QC Format Sources.....	46

## LIST OF FIGURES

Figure 1.	Testing and Quality Assurance Project Plan Approval Form.....	3
Figure 2.	Demonstration Factory Layout .....	6
Figure 3.	Demonstration Factory Organic Finishing Line .....	7
Figure 4.	Project Organization Chart .....	22

## LIST OF APPENDICES

A	Default Standard Test Panels
B	ASTM Methods

## 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

<b>ACGIH</b>	American Conference of Governmental Industrial Hygienists
<b>ANSI</b>	American National Standards Institute
<b>AOAC</b>	Association of Official Analytical Chemists
<b>ASQC</b>	American Society for Quality Control
<b>ASTM</b>	American Society for Testing and Materials
<b>av</b>	average
<b>CCEP</b>	Coatings and Coating Equipment Program
<b>CTC</b>	Concurrent Technologies Corporation
<b>DFT</b>	dry film thickness
<b>DI</b>	deionized
<b>DOI</b>	distinctness-of-image
<b>EP</b>	empty pan
<b>EPA</b>	U.S. Environmental Protection Agency
<b>ES</b>	empty syringe
<b>ETF</b>	Environmental Technology Facility
<b>ETV</b>	Environmental Technology Verification
<b>FBO</b>	Fed Biz Ops
<b>FS</b>	full syringe
<b>GC/MS</b>	gas chromatography / mass spectrometry
<b>GVP</b>	Generic Verification Protocol
<b>HAP</b>	hazardous air pollutant
<b>ID</b>	identification
<b>IR</b>	infrared
<b>ISO</b>	International Standardization Organization
<b>MEK</b>	methyl ethyl ketone
<b>MSDS</b>	Material Safety Data Sheet
<b>NDCEE</b>	National Defense Center for Environmental Excellence
<b>NIST</b>	National Institute for Standards and Technology
<b>OFL</b>	Organic Finishing Line
<b>PEA</b>	Performance Evaluation Audit
<b>PLC</b>	programmable logic controller
<b>PS</b>	deposited solids
<b>QA/QC</b>	quality assurance/quality control
<b>QMP</b>	Quality Management Plan
<b>RFT</b>	Request for Technologies
<b>RPD</b>	relative percent difference
<b>SOP</b>	Standard Operating Procedure
<b>SRM</b>	standard reference material
<b>TBD</b>	to be determined
<b>TQAPP</b>	Testing and Quality Assurance Project Plan
<b>USAEC</b>	U.S. Army Environmental Center
<b>UV</b>	ultraviolet
<b>VOC</b>	volatile organic compound



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## 1.0 INTRODUCTION

### 1.1 Purpose of the Liquid Coatings Generic Verification Protocol

The primary purpose of this document is to establish the Generic Verification Protocol (GVP) for innovative liquid coatings, to which reference will be made frequently throughout this document as the Liquid 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) 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 liquid coatings TQAPPs to facilitate independent reviews of Project Plans and test results, and to provide a standard platform of understanding for 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<sup>1</sup>. This GVP will ensure that project results are compatible with and complementary to similar projects. All ETV CCEP liquid coatings TQAPPs are adapted from this standard, the ETV Program Quality Management Plan (QMP), and the ETV CCEP QMP<sup>2,3</sup>. These TQAPPs will contain sufficient detail to ensure that measurements are appropriate for achieving project objectives, that data quality is known, and that the data are legally defensible and reproducible.

### 1.3 Organization of the Liquid 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
- References
- 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 liquid coating tested. Acknowledgment by each key person indicates commitment toward implementation of the plan. Figure 1 shows the Approval Form format to be used.

## APPROVAL FORM

Date Submitted: \_\_\_\_\_ QTRAK No.: \_\_\_\_\_

Revision No.: \_\_\_\_\_ Project Category: \_\_\_\_\_

Title: \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

Project/Task Officer: \_\_\_\_\_

EPA/Address/Phone No.: \_\_\_\_\_

U.S. EPA -  
 U.S. DCC-W                                      U.S. AEC /  
 Interagency                                      NDCEE  
 Agreement No.: \_\_\_\_\_ Contract No.: \_\_\_\_\_ Task No.: \_\_\_\_\_

### APPROVALS

ETV CCEP Project Manager	Signature	Date
--------------------------	-----------	------

ETV CCEP QA Officer	Signature	Date
---------------------	-----------	------

EPA ETV CCEP Project Manager	Signature	Date
------------------------------	-----------	------

EPA ETV CCEP QA Manager	Signature	Date
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EPA – Environmental Protection Agency  
 DCC-W – Defense Contracts Command – Washington  
 AEC – Army Environmental Center

Figure 1. Testing and Quality Assurance Project Plan Approval Form

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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. Innovative liquid coatings are continually being developed or reformulated to reduce any detrimental effects to the environment. This is primarily accomplished by reducing the VOC or hazardous air pollutant (HAP) content of the coating. Often these coating technologies are slow to penetrate the market because potential users, especially an ever-growing number of small companies, do not have the resources to test the new coatings in their particular application and may be constructively skeptical of the technology provider's claims. If an unbiased, third party facility could provide pertinent test data, environmentally friendly coating technologies would penetrate the industry faster and accelerate environmental improvements.

The ETV CCEP, a joint venture of the U.S. Environmental Protection Agency (EPA) and CTC of Johnstown, Pennsylvania, in conjunction with the National Defense Center for Environmental Excellence (NDCEE) Program, has been established to provide unbiased, third party 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 the verification of the performance of innovative liquid 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 Testing Site

CTC has been tasked under the NDCEE Program to establish a demonstration factory capable of prototyping processes that will reduce or eliminate environmentally harmful materials used or produced in manufacturing. To accelerate the transition of environmentally friendly processes to the manufacturing base, CTC offers the ability to test processes and products on full-scale, commercial equipment. This demonstration factory is a major national asset. It includes a combination of organic finishing, cleaning, stripping, inorganic finishing, and

recycle/recovery equipment. The organic finishing equipment in the demonstration factory will be available for the pilot-scale testing performed in this project. A layout of the *CTC* demonstration factory is shown in Figure 2. A schematic of the Organic Finishing Line (OFL) is shown in Figure 3.

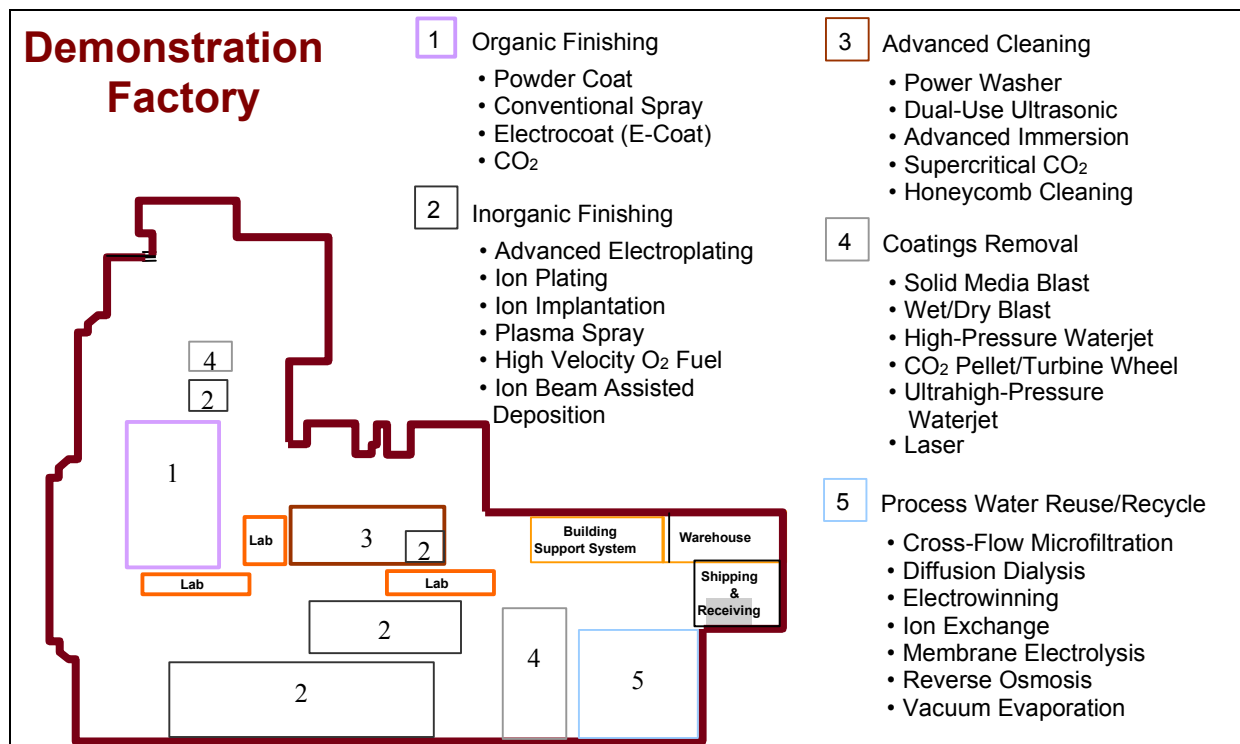


Figure 2. Demonstration Factory Layout

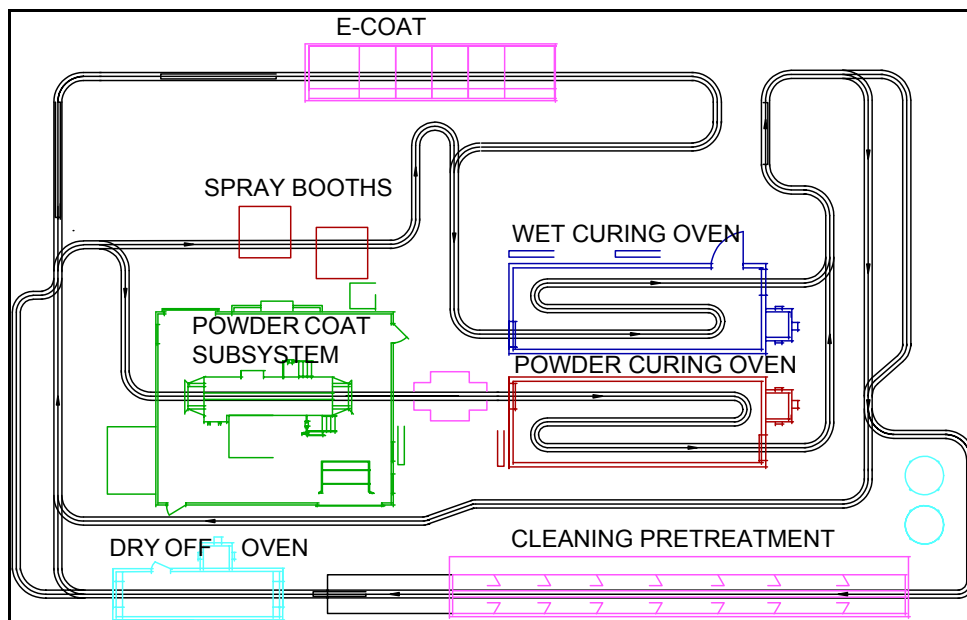


Figure 3. Demonstration Factory Organic Finishing Line

In the event that a particular technology verification or laboratory analysis cannot be performed at *CTC*, arrangements will be made to ensure the requirements of the TQAPP and all associated QA procedures are completed at an appropriate facility.

#### 2.1.2 Laboratory Facilities

In support of the demonstration factory coating processes, *CTC* maintains extensive, state-of-the-art laboratory testing facilities. These laboratory facilities are used for the measurement and characterization of processes and specimens, as well as for bench-scale coating technology evaluations. Table 1 lists the various testing and evaluation laboratories and the representative equipment holdings that are relevant to ETV CCEP coatings verifications.



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) Thermacal Dry Block Calibrator (Temperature) Druck Pressure Calibrator (Pressure) Fluke Digital Multimeter (Voltage)

### 2.1.3 Offsite Testing

While the NDCEE demonstration factory in *CTC*'s Johnstown, PA, facility is adequately equipped with a variety of organic finishing equipment, it may not possess certain application or laboratory equipment that is specified by a technology's vendor; therefore, if a vendor specifies the use of equipment not currently available for testing at *CTC*, the ETV CCEP may choose to procure the required equipment or conduct verification testing at an offsite facility. If testing is conducted off-site, the verification testing will be controlled and observed by ETV CCEP technical personnel. The facility will be chosen by the ETV CCEP and will be subject to a pretest visit to investigate the condition of equipment, instrumentation, and its working environment. A preparation checklist will be used for the pretest visit and will be updated to take into consideration what is learned during the visit. It must meet the standards of the individual TQAPP, the ETV CCEP QMP, and the ETV Program QMP. A complete description of the testing apparatus will be included in each TQAPP for offsite testing.

Each TQAPP will specify how the test parts are pretreated and cleaned, coated (liquid coating), cured, and analyzed for response factors. If the offsite facility is unable to pretreat the parts, pretreated parts will be purchased or pretreated at *CTC*'s facility. Otherwise, the parts will be cleaned, coated, and cured at the offsite facility, by ETV CCEP or qualified offsite personnel, and then shipped to *CTC* for analysis. *CTC* will perform the laboratory analyses of the critical response factors (see Tables 3, 4, and 5 and Section 2.2.9). The appropriate critical and noncritical control factors will be measured or monitored (see Table 3, 4, and 5 and Section 2.2.9) at the offsite testing facility, and the data will be recorded by ETV CCEP or qualified offsite personnel.

The qualified offsite personnel must be trained on the applicable measuring or monitoring equipment, be familiar with the QA requirements of the ETV, and be sufficiently independent as to assure the third party, unbiased objectives of the ETV Program.

## 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 Verification Report and Data Notebook
- Prepare 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/or baseline tests;
- The ETV CCEP will obtain the baseline coatings (as appropriate);
- The vendor will provide the liquid coating being verified;
- Data such as dry film build, gloss, and visual appearance will be collected, following the American Society for Testing and Materials (ASTM) methods, or equivalent;
- 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 VOC and HAP content and to verify the quality and durability of liquid coatings. In addition, the VOC/HAP emissions generated during the curing of the coating may be checked using an EPA approved of a method for determining those emissions. The coated test panels will be checked for dry film thickness (DFT), visual appearance, and at least three of the following analyses: gloss, color, distinctness-of-

image (DOI), adhesion, corrosion resistance, 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 liquid 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 liquid coating vendor's recommendations. Details concerning panel characteristics, pretreatment, and pretreatment analysis will be identified in each product-specific TQAPP. However, the standard test product, as is shown in Appendix A, *Default Standard Test Panel*, will be a panel 30.5 cm (12 in.) long and 10.2 cm (4 in.) wide with a 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 liquid coating vendor. (**Note:** CTC's OFL can accommodate parts with dimensions up to 1.2 m x 1.2 m x 0.9 m (4 ft x 4 ft x 3 ft), and parts weighing up to 113.5 kg (250 lb).)

### 2.2.4 Coating Specification

The liquid coatings submitted for verification testing should provide an environmental benefit over the existing coatings currently in use in each liquid coating's target industry. The stakeholders group will also review the liquid 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 Standard Apparatus

Figure 3 shows the testing location of the spray booth within the OFL. Most coating application processes will be performed in the same wet spray booth in the *CTC* Demonstration Factory.

Prior to testing, the coating vendors will be supplied with important information such as the average ambient temperature, ambient humidity, and substrate temperature. The coating vendors will then determine the operating parameters of the spray equipment (e.g., input air pressure, gun-to-target distance, horizontal gun speed, flash time, dwell time). Panel pretreatment is specific to the substrate material and will be specified by the liquid coatings vendor. If panels are not purchased in a pretreated condition, pretreatment will be performed at *CTC*. The pretreatment sequence typically conducted at *CTC* is as follows:

1. Panels receive an alkaline cleaning followed by a fresh water rinse.
2. Zinc phosphate or other recommended treatment is applied followed by a water rinse.
3. A nonchromate or other recommended sealer is applied followed by a deionized (DI) water rinse.
4. Panels are dried in a dry off oven.

One random test panel will be removed for pretreatment analysis for each verification run. Since typically each verification test will consist of five runs, each with one row of eight panels, eight additional panels will be pretreated for each verification test to be used for the pretreatment analysis and as setup panels for the test. Any pretreatment conducted by an offsite facility is expected to follow a similar sequence.

During testing using the default scenario, standard test panels will be suspended from racks containing a single row of up to eight panels per rack. The test panels will be fixtured on the rack to minimize movement during spraying. Fixturing consists of a flat bar that connects all eight hooks. The bar will minimize side-to-side rotation of the panels. A second bar is oriented near the bottom of the panels to prevent the bottom of the panels from moving away from the gun. The test panels will be transported to the spray booth by an overhead conveyor. A mechanical stop mechanism will align the racks of test panels in the proper position relative to the spraying mechanism. Once the racks are in position, the programmable logic controller (PLC) of the spraying mechanism will activate the motors that drive the linear motion translators. The translators will move both horizontally and vertically, enabling the application equipment to treat an area approximately 1.4 m by 1.4 m (4.5 ft x 4.5 ft). The panels will be automatically sprayed using vertical overlap of the spray pattern. The PLC will also trigger the pneumatic spray gun or a pneumatically actuated cylinder that compresses the trigger of a manual

spray gun. During dwell time between passes, paint flow will be interrupted to minimize paint usage. Once the spray application is complete, the PLC will release the mechanical stop that maintains the position of the rack, enabling the overhead conveyor to move the next rack into position.

Before each test, a set of dummy panels will be coated to ensure that the equipment parameters are set correctly. (The coating vendor may wish to assist in this step.) The input air pressure will be monitored throughout the test, and the air pressure at the cap and air horns (if applicable) will be measured using a verified test cap prior to each run. The paint usage will be determined through gravimetric means or by the use of an in-line flow meter, as appropriate.

To help ensure proper equipment setup and operation, the liquid coatings vendors will be invited to participate in the startup phase of the testing and to observe the testing of their coatings.

The pressure drop across the paint spray booth filters will be checked prior to each run and at the end of the test. The pressure drop is monitored in the event that the filter bank system malfunctions. A pressure drop across the filter bank greater than 1 cm (0.4 in.) of water shall indicate that the system requires service and/or changing of the filters.

#### 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 liquid 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 liquid coating'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 liquid coating verification test. Other parameters will be developed from the application equipment's or coating manufacturer's recommendations and/or experimental trials performed by the ETV CCEP.

### 2.2.7 Design of Experiment

This test protocol will verify the performance of liquid coatings submitted in response to the associated Fed Biz Ops (FBO) or Request for Technologies (RFT). A mean value and variance (or standard deviation) will be reported for each critical response factor. If a liquid coating vendor makes a claim about a particular coating characteristic, he 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, 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 coating equipment parameters are correct. Also, one panel for each test run will be used for pretreatment analysis. Forty panels will be coated during the verification test to determine the pollution prevention 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 to the part size and weight restrictions identified in Section 2.2.3.

### 2.2.8 Performance Testing

Liquid 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 liquid coating vendors with a list of key noncritical test factors that may affect the critical response factors (i.e., 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 critical response factors.

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 liquid coating, several measurements will be taken on the coating, and noncoated and coated test panels. Coating samples will be analyzed for VOC and HAP content. 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 test and verifications required for performance evaluation.

#### 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 experiments matrix 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
Liquid 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	Once per test	1
Input Air Pressure to Gun or Pot	From coating provider	Factory floor	Once per test	1
Air Pressure at Air Cap and Horns (HVLP only)	≤ 10 psig	Factory floor	Once per run	5
Product Involved in Testing	Standard Test Plan (material TBD)	Factory floor	See default scenario in Section 5.3	40 panels
Pretreatment Analysis	Varies <1.2 g/m <sup>2</sup>	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
Spray Booth Filter Pressure Drop	<1 cm H <sub>2</sub> O (<0.4 in. H <sub>2</sub> O)	Across the filter bank	Once per run	5
Temperature of Panels, as Coated	Varies <5 °C during test	Center of test panel in coatings laboratory	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
Cure Temperature	TBD	Factory floor	Once per run	5

TBD – To be determined

Table 5. Critical Response Factors

Critical Response Factor	Measurement Location or Method	Frequency	Total Number for the Test
Environmental			
VOC Content	See Section 2.2.12	5 samples from coating batch used during test	5
HAP Content	See Section 2.2.12	5 samples from coating batch used during test	5
Quality/Durability (mandatory for all coatings)			
Dry Film Thickness (DFT) (Magnetic method)	ASTM B 499	TBD <sup>a</sup>	TBD
Visual Appearance	Entire test panel	1 per panel	40
Quality/Durability (optional)			
Gloss	ASTM D 523	One random panel per run	5
Color	ASTM D 1729 and/or ASTM D 2244 <sup>b</sup>	One random panel per run	5
Distinctness-of-image (DOI)	ASTM D 5767 Test Method B <sup>c</sup>	One random panel per run	5
Adhesion <sup>d</sup>	ASTM D 3359	One random panel per run	5
Pencil Hardness <sup>d</sup>	ASTM D 3363	One random panel per run	5
Salt Spray	ASTM B 117	One random panel per run	5
Direct Impact	ASTM D 2794	One random panel per run	5
Mandrel Bend	ASTM D 522	One random panel per run	5
Chemical Resistance (MEK Rub)	ASTM D 5402	One random panel per run	5
Humidity Resistance	ASTM D 1735	One random panel per run	5
Weather Resistance	ASTM G 26	One random panel per run	5
Abrasion Resistance	ASTM D 4060	One random panel per run	5

<sup>a</sup> TBD – To be determined

<sup>b</sup> Both color analyses will use the same panel if both are selected.

<sup>c</sup> Except that the sliding combed shutter is replaced by a rotating eight-bladed disc

<sup>d</sup> The adhesion and pencil hardness tests will all be performed on the same panel as the DFT test.

Some target factors that may be used to test liquid coatings include:

- Equipment preparation                      TBD
- Spray pattern                                    TBD
- Number of passes                              TBD
- Dwell time between passes                TBD
- Number of coats                                TBD
- Flash time between coats                    TBD

- Target dry film thickness (DFT)      TBD

## 2.2.12 Determination of VOCs and HAPs

The VOC and HAP contents of the liquid coatings will be determined by EPA Methods 24 and 311 (or 8260B), respectively. To assist in VOC/HAP determinations and assessments, the vendor will be required to submit material safety data sheets (MSDSs) and coating composition information, including the following:

- Total volatile matter
- Coating density
- Solids content
- Water content (as appropriate)
- EPA-exempt solvents content
- Total VOC content
- Specific VOC/HAP identification
- Density of cured coating.

Bulk formulation tests will be performed to assess the quantity of VOCs/HAPs contained in the paint per EPA Method 24. EPA Method 24 requires three separate measurements to determine the total VOC content in the coating. First, the total volatile matter contained within the liquid coating will be determined using gravimetric analysis (ASTM D2369). Next, a Karl Fischer Titration (ASTM D4017 or E1064) will determine water content of the coating, and gas chromatography (ASTM D4457) will determine the amount of exempt solvents. Finally, the total VOC content is calculated by subtracting the mass of water and mass of exempt solvents from the total volatile matter. Identification of the specific VOCs/HAPs contained in the paint will be performed using gas chromatography/mass spectrometry (GC/MS) per EPA Method 311 or EPA Method 8260B. These analyses may be conducted at an offsite laboratory.

The release of VOCs and HAPs during curing is a potential environmental concern. However, no standard test method has been accepted to measure such emissions. If a standard test method for cure emissions is developed, the ETV CCEP will incorporate the analysis of cure emissions as one of the critical response factors to provide data concerning this issue.

## 2.3 Schedule

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

Table 6. Estimated Schedule as of 9/24/03

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

TBD – To be determined

### 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 link 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 participant, his/her applicable experience, and his/her anticipated time dedication to the project during testing and reporting is given in Table 7.

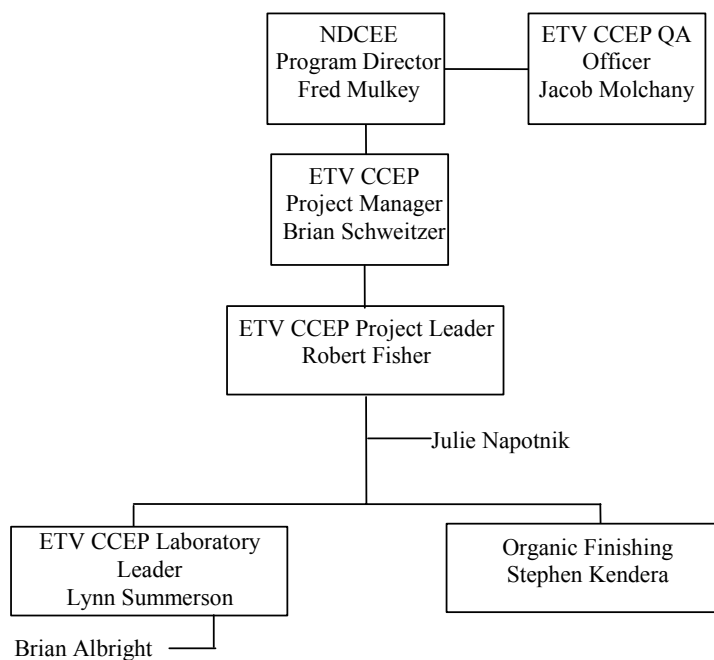


Figure 4. Project Organization Chart

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 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	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 (OFL) Accountable to Finishing Engineer	Industrial Paint and Coatings Experience (28 years)		10%
Lynn Summerson – ETV CCEP Laboratory Leader/Statistical Support Staff	Laboratory analysis 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.



Table 8. Frequency and Mechanisms of Communications

Initiator	Recipient	Mechanism	Frequency
NDCEE Program Director, ETV CCEP Project Manager, and/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	<i>CTC</i>	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 (will prevent accomplishment of verification cycle testing) Quality Objective Deviation	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

If non-*CTC* support personnel are used, their roles and responsibilities will be addressed in the appropriate product-specific TQAPP.

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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 innovative liquid coatings by establishing their environmental benefit and by documenting the applied coating's finish quality. Controlling and monitoring the critical and noncritical factors, which are QA objectives for each technology-specific TQAPP based on this GVP, will meet these objectives. 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. Values and/or equipment will be updated in product-specific TQAPPs. 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 and/or spikes 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.

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%
Air Pressure at Air Cap and Horns (HVLP only)	Pressure gauge	psig	±0.5 psig	±0.5%	90%
Product Involved in Testing	Test panels	N/A	N/A	N/A	100%
Pretreatment Analysis	ASTM B 767	g/m <sup>2</sup>	±0.005	±0.01	90%
Surface Area of Test Panels	Ruler	cm <sup>2</sup> (ft <sup>2</sup> )	±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%
Spray Booth Pressure Drop	Water manometer	cm H <sub>2</sub> O (in. H <sub>2</sub> O)	±0.07 (±0.03)	±0.07 (±0.03)	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	Calendar	d	N/A	N/A	90%
Cure Temperature	Thermocouple	°C	±2.2 °C	±2.2 °C	90%

ACGIH - American Conference of Governmental Industrial Hygienists, Inc.

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

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

Measurement	Method	Units	Precision	Accuracy	Completeness
VOC Content	EPA Method 24	g/L (lb/gal)	±0.6% RPD <sup>a</sup>	±1.8%	90%
HAP Content	EPA Method 311 or 8260B	g/L (lb/gal)	±0.6% RPD	±1.8%	90%
Dry Film Thickness (DFT) (Magnetic method)	ASTM B 499	mils <sup>b</sup>	20%	10% true thickness	90%
Visual Appearance	N/A <sup>c</sup>	N/A	N/A	N/A	100%
Gloss	ASTM D 523	Gloss units	20% RPD	±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%
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 (MEK Rub)	ASTM D 5402	Visual	TBD by ASTM <sup>d</sup>	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%

<sup>a</sup> RPD – Relative Percent Difference

<sup>b</sup> 1 mil = 0.001 in.

<sup>c</sup> N/A – Not applicable

<sup>d</sup> TBD – To be determined

#### 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.

#### 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 strive 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/or 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 technology 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.

Test panels used in 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.



## 5.0 SITE SELECTION AND SAMPLING PROCEDURES

### 5.1 Site Selection

Where possible, this project will be conducted at *CTC*, in Johnstown, PA, and ETV CCEP personnel will perform all processing and testing, when possible. The site for application and evaluation will be at the NDCEE demonstration factory in the Environmental Technology Facility (ETF) under the direct control of the Engineering, Statistical Support, and OFL groups.

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.

Application of the coating involves transporting test panels via an automatic conveyor through the OFL. The test panel will be coated in the first of the two wet spray booths. Test panels will be evaluated prior to application and after curing. The experimental design involves applying a coating according to the manufacturers' recommended optimum conditions. The test panels will be sampled and analyzed to generate performance data.

### 5.2 Site Description

Figure 2 illustrates the overall layout of the NDCEE demonstration factory and the location of the process equipment that will be used for this project. This project may involve the use of the pretreatment line, the wet spray booths, and the wet cure oven. Other equipment or testing sites may be used, as necessary.

### 5.3 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 technology provider's claim(s) about performance characteristics and the respective confidence levels given in the responses to the RFT. If the liquid coatings providers request 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, which includes those identified in Table 12.

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	

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

#### 5.4 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 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.

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## 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 after the verification test analyses are complete.

#### 6.1.1 Facility Testing and Calibration

Calibration procedures for ETV CCEP within the OFL test 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. 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 Tables 13 and 14.

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.

#### 6.1.2 Laboratory Testing and Calibration Procedures

The analytical methods performed by ETV CCEP at NDCEE are adapted from standard ASTM, MIL-SPEC, EPA, Association of Official Analytical Chemists (AOAC) and/or 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 and/or immediately after each sample batch. Standard solutions are purchased from reputable chemical supply houses in neat 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.

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

Solids pans will be prepared as specified by the ASTM standard for determining volatile content of coatings (ASTM D 2369). The solids pans will be labeled with an identification number and letter. Two separate solids pans will be used for each batch of coating and the values obtained will be averaged. The data required for the solids test are recorded on the coating batch worksheet.

The percent of solids is calculated as:

$$N = ((W2 - W1) / S) \times 100$$

where:

- W1 = Weight of the dish
- W2 = Weight of dish plus specimen after heating
- S = Specimen weight (Sy1 - Sy2)
- Sy1 = Syringe before dispensing coating
- Sy2 = Syringe after dispensing coating

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, and/or the published reference method's, instructions. Actual sample analysis will take place only after setup is verified per the reference method and the technology 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 industry.

Table 13. Noncritical Control Factor Testing and Calibration Criteria

Noncritical Factor	Method	Method Type	Calibration Procedure	Calibration Frequency	Calibration Acceptance Criteria <sup>a</sup>
Input Air Pressure	Factory gauge	Pressure gauge	Comparison to NIST-traceable standard	Six months	±5 psig
Air Pressure at Air Cap and Horns (HVLV only)	Factory gauge	Pressure gauge	Comparison to NIST-traceable standard	Six months	±5 psig
Products Involved in Testing	Test panels	N/A <sup>b</sup>	N/A	N/A	N/A
Pretreatment Analysis	ASTM B767	Chromate solution (50g/L CrO <sub>3</sub> )	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	Anemometer	Sent for calibration or certification	Annually	Calibration or certification documentation
Spray Booth Filter Pressure Drop	Manometer	Water manometer	Comparison to NIST-traceable standard	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	Calendar Day	Calendar Day	N/A	N/A	N/A
Cure Temperature	Thermo-couple	Thermocouple/ (controllers)	Comparison to NIST-traceable standard	Annually / (six months)	N/A

<sup>a</sup> As a percent recovery of a standard

<sup>b</sup> N/A – Not applicable

Table 14. Critical Response Factor Testing and Calibration Criteria

Critical Measurement	Method Number <sup>a</sup>	Method Type	Calibration Procedure	Calibration Frequency	Calibration Acceptance Criteria <sup>b</sup>
VOC Content	EPA Method 24	Volatile content	Comparison to NIST-traceable standard	Each use	±0.003 g
HAP Content	EPA Method 311 or 8260B	GC/MS	Comparison to NIST-traceable standard	Each use	±0.003 g
Dry Film Thickness (DFT)	ASTM B 499	Magnetic	Comparison to NIST-traceable standard	Verify calibration after each run	90–110%
Visual Appearance	N/A <sup>c</sup>	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
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 <sup>d</sup> ≤20% among steel panels, av of chemical tests within specific ranges
Direct Impact	ASTM D 2794	2 lb 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 (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%

<sup>a</sup> A list of ASTM methods to be used is provided in Appendix B.

<sup>b</sup> As a percent recovery of a standard

<sup>c</sup> N/A – Not applicable

<sup>d</sup> RSD – relative standard deviation



## 6.4 Nonstandard Methods

ETV CCEP will not 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 in-house developed protocols. 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 non-standard quantitative chemical procedure is being performed, it should produce replicate results of  $\pm 25$  relative percent difference (RPD) and should give values within  $\pm 20$  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 and/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/or qualitative observations will be recorded. 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 process data sheets, bench data 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).

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

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  $2 \times 0.0005$  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 analyses. 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 an anomaly or nonconformance with expected or historical performance values is suspected, 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 and the ETV CCEP laboratory 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.

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## 8.0 INTERNAL QUALITY CONTROL CHECKS

### 8.1 Guide Used for Internal Quality Program

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. ETV CCEP uses the NDCEE facility and its QA systems to verify coating technologies. 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 <sup>nd</sup> Ed.	James P. Dux

### 8.2 Types of QA Checks

The NDCEE ETF Environmental Laboratory and OFL 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 process conditions and the laboratory analyses will adhere to the QA/QC requirements specified in these documents. In addition, where QA/QC criteria are not specified, or where the laboratory or process line performs additional QA/QC activities, these protocols are explained in the laboratory or process 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 OFL process personnel at ETF 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 process 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 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 dry film thickness 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/or the project-specific QA/QC objectives. Laboratory records are maintained with the sample data packages and/or in centralized files, as appropriate. To ensure comparability, the laboratory and process 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.



## 9.0 PERFORMANCE AND SYSTEM AUDITS

The 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 is consistent with the audit requirements of 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 established its quality system based on ISO 9000 / 14000 and 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 ETV CCEP 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 (TSAs)

A listing of all coating equipment, laboratory measuring and testing devices, and procedures, coating methods, and a copy of the approved ETV Program and ETV CCEP QMPs 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 the 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.

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## 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.

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

where: C1 = larger of the two observations

C2 = smaller of the two observations

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

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:

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

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:

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

where: C<sub>m</sub> = observed concentration of reference material

C<sub>srm</sub> = theoretical value of srm.

### 10.3 Completeness

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

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 and/or accuracy indicators. Such nonconformances will be documented on a standard laboratory or facility/process 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 and/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 ETV CCEP 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 project manager will contact the EPA ETV CCEP project manager. In cases of routine nonconformance, EPA will be notified in the 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 the ETV CCEP laboratory leader. The ETV CCEP project manager will ensure these reports are included in the report to the EPA. The ETV CCEP 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
- Listing and summary of all nonconformances and/or 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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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](http://www.epa.gov/etv/pdfs/qmp/06_qmp_p2.pdf).

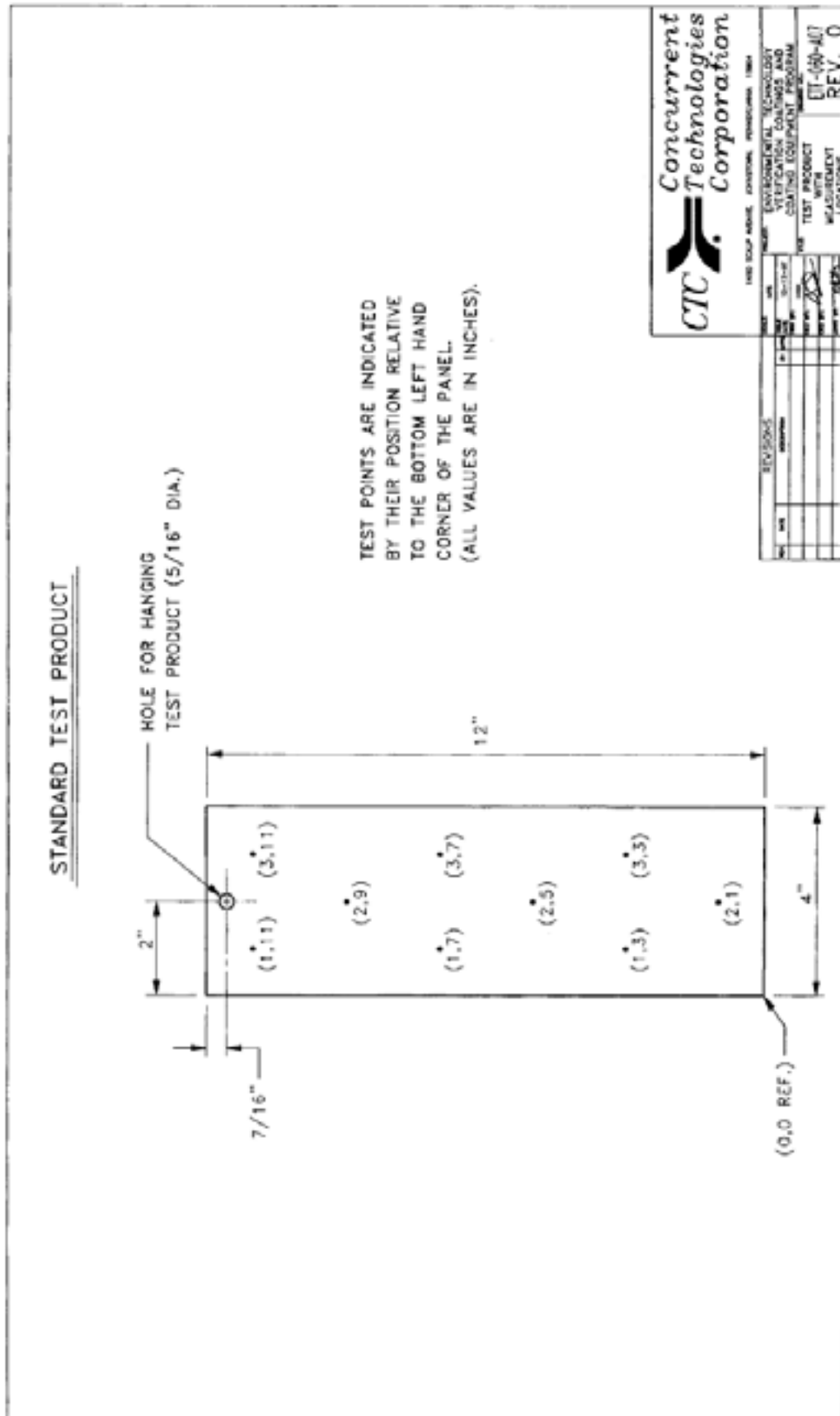
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## APPENDIX A

### 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 Methods 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 4017 -- Standard Test Method for Water in Paints and Paint Materials by Karl Fischer Method
- ASTM D 4060 -- Standard Test Methods for Abrasion Resistance of Organic Coatings by the Taber Abraser
- ASTM D 4457 -- Standard Test Methods for Determination of Dichloromethane and 1,1,1-Trichloroethane in Paints and Coatings by Direct Injection into a Gas Chromatograph
- ASTM D 5402 -- Assessing the Solvent Resistance of Organic Coatings Using Solvent Rubs
- ASTM D 5767 -- Standard Test Methods for Instrumental Measurement of Distinctness-of-Image Gloss of Coating Surfaces
- ASTM E 1064 -- Standard Test Method for Water in Organic Liquids by Coulometric Karl Fischer Titration
- ASTM G 26 -- Practice for Operating Light Exposure Apparatus (Xenon-Arc Type) With and Without Water for Exposure of Nonmetallic Materials
- EPA Method 24 -- Determination of Volatile Matter Content, Density, Volume Solids, and Weight Solids of Surface Coatings
- EPA Method 311 -- Analysis of Hazardous Air Pollutant Compounds in Paints and Coatings by Direct Injection into a Gas Chromatograph
- EPA Method 8260B -- Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)