

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

High Transfer Efficiency Spray Equipment – Generic Verification Protocol (Revision 0)

September 30, 2006

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13. ABSTRACT (<i>Maximum 200 words</i>) The Environmental Technology Verification (ETV) Program has been established by the U.S. Environmental Protection Agency (EPA) to verify the performance characteristics of innovative environmental technologies across all media and report this objective information to the states, buyers, and users of environmental technology; thus, accelerating the entrance of these new technologies into the marketplace. Verification organizations oversee and report verification activities based on testing and quality assurance protocols developed with input from major stakeholders and customer groups associated with the technology area. ETV consists of six technology centers. Information about each of these centers can be found on the Internet at http://www.epa.gov/etv/ . EPA's ETV Program, through the National Risk Management Research Laboratory (NRMRL), Air Pollution Prevention and Control Division (APPCD) has partnered with Concurrent Technologies Corporation (CTC), through the National Defense Center for Environmental Excellence (NDCEE), to verify innovative coatings and coating equipment technologies for reducing air emissions from coating operations. Pollutant releases to other media are considered in less detail. The following protocol outlines the basis for completing an ETV verification test of High-Transfer Efficiency Spray Guns.				
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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 High Transfer Efficiency Spray Equipment GVP	1
1.2 Quality Assurance for the ETV CCEP.....	1
1.3 Organization of the High-TE GVP	1
1.4 Formatting.....	2
1.5 Approval Form.....	2
2.0 PROJECT DESCRIPTION.....	4
2.1 General Overview	4
2.1.1 Demonstration Factory Testing Site	5
2.1.2 Laboratory Facilities	6
2.2 Technical/Experimental Approach and Guidelines	7
2.2.1 Test Approach.....	8
2.2.2. Verification Test Objectives	8
2.2.3 Large Target Description	8
2.2.4 Small Target Description	9
2.2.5 Coating Specification.....	11
2.2.6 Standard Apparatus.....	12
2.2.7 Process Standards	12
2.2.8 Design of Experiment	13
2.2.9 Performance Testing	13
2.2.10 Quantitative Measurements	14
2.2.11 Participation.....	14
2.2.12 Critical and Non-Critical Factors.....	14
2.3 Schedule.....	19
3.0 PROJECT ORGANIZATION AND RESPONSIBILITIES.....	20
4.0 QUALITY ASSURANCE OBJECTIVES.....	23
4.1 General Objectives.....	23
4.2 Quantitative Quality Assurance Objectives.....	23
4.2.1 Accuracy	25
4.2.2 Precision.....	26
4.2.3 Completeness	26
4.2.4 Impact and Statistical Significance Quality Objectives.....	26
4.3 Qualitative QA Objectives: Comparability and Representativeness	26
4.3.1 Comparability	26
4.3.2 Representativeness.....	27
4.4 Other QA Objectives	27
4.5 Impact of Quality	27
5.0 SITE SELECTION AND SAMPLING PROCEDURES	28

5.1	Site Selection	28
5.2	Site Description.....	28
5.3	Sampling Procedures and Handling.....	28
5.4	Sample Custody, Storage and Identification.....	29
6.0	ANALYTICAL PROCEDURES AND CALIBRATION	30
6.1	Facility and Laboratory Testing and Calibration.....	30
6.1.1	Facility Testing and Calibration	30
6.1.2	Laboratory Testing and Calibration Procedures	30
6.2	Product Quality Procedures	31
6.3	Standard Operating Procedures and Calibration.....	31
6.4	Non-Standard Methods	33
7.0	DATA REDUCTION, VALIDATION, AND REPORTING	34
7.1	Raw Data Handling.....	34
7.1.1	Variables Used In Analysis.....	34
7.1.2	Error in Mass of Coating Sprayed.	34
7.1.3	Error in Solids Content.	35
7.1.4	Error in Mass Deposited.	35
7.1.5	Calculation of Transfer Efficiency.	35
7.2	Preliminary Data Package Validation.....	36
7.3	Final Data Validation.....	36
7.4	Data Reporting and Archival	37
7.4.1	Calculation of DFT	37
7.4.2	Interpretation of the Numerical Results.....	37
7.4.3	Evaluation of the High-TE Spray Gun	37
7.5	Verification Statement	38
8.0	INTERNAL QUALITY CONTROL CHECKS	39
8.1	Guide Used for Internal Quality Program.....	39
8.2	Types of QA Checks.....	39
8.3	Basic QA Checks	39
8.4	Specific Checks.....	40
9.0	PERFORMANCE AND SYSTEM AUDITS	41
10.0	CALCULATION OF DATA QUALITY INDICATORS	42
10.1	Precision.....	42
10.2	Accuracy	42
10.3	Completeness	42
10.4	Project Specific Indicators	42
11.0	CORRECTIVE ACTION	43
11.1	Routine Corrective Action	43
11.2	Nonroutine Corrective Action	43
12.0	QUALITY CONTROL REPORTS TO MANAGEMENT	44

LIST OF TABLES

Table 1.	Testing and Laboratories and Representative Laboratory Equipment Holdings	7
Table 2.	Overall Guidelines and Procedures Applied to this Test/QA Plan	7
Table 3.	Critical Control Factors	16
Table 4.	Non-Critical Control Factors	17
Table 5.	Critical Response Factors	18
Table 6.	Estimated Schedule as of 9/27/06	19
Table 7.	Summary of ETV CCEP Experience and Responsibilities	21
Table 8.	Frequency and Mechanisms of Communications	22
Table 9.	Responsibilities During Testing	22
Table 10.	QA Objectives for Precision, Accuracy and Completeness for All Non-Critical Control Factor Performance Analyses	24
Table 11.	QA Objectives for Precision, Accuracy and Completeness for All Critical Response Factor Performance Analyses	25
Table 12.	Process Responsibilities	29
Table 13.	Non-Critical Control Factor Testing and Calibration Criteria	32
Table 14.	Critical Response Factor Testing and Calibration Criteria	33
Table 15.	CTC Laboratory QA/QC Format Sources	39

LIST OF FIGURES

Figure 1.	Test/QA Plan Approval Form	3
Figure 2.	Demonstration Factory Layout	5
Figure 3.	Demonstration Factory Organic Finishing Line	6
Figure 4.	Large Target Application Diagram	9
Figure 5.	Small Target Application Diagram	10
Figure 6.	Test Panel Measurement Locations	11
Figure 7.	Project Organization Chart	20

LIST OF APPENDICES

A ASTM International Methods

SI to English Conversions

SI Unit	English Unit	Multiply SI by factor to obtain English
°C	°F	(1.80 E + 00), then add 32
L	gal. (U.S.)	2.642 E - 01
m	ft	3.281 E + 00
kg	lbm	2.205 E + 00
kPa	psi	1.4504 E - 01
cm	in.	3.937 E - 01
mm	mil (1 mil = 1/1000 in.)	3.937 E + 01
m/s	ft/min	1.969 E + 02
kg/L	lbm/gal (U.S.)	8.345 E + 00

List of Abbreviations and Acronyms

%C	percent completeness
%R	percent recovered
%S	percent solids
ACGIH	American Conference of Governmental Industrial Hygienists
ACS	American Chemical Society
ANSI	American National Standards Institute
AOAC	Association of Official Analytical Chemists
ASQC	American Society for Quality Control
CCEP	Coatings and Coating Equipment Program
CTC	Concurrent Technologies Corporation
CS	coating sprayed
DFT	dry film thickness
DOI	distinctness-of-image
EP	empty pan
EPA	U.S. Environmental Protection Agency
ES	empty syringe
ETF	environmental technology facility
ETV	environmental technology verification
FS	full syringe
GVP	generic verification protocol
HVLP	high-volume, low-pressure
IR	infrared
ISO	International Standardization Organization
NDCEE	National Defense Center for Environmental Excellence
NIST	National Institute for Standards and Technology
OFL	organic finishing line
P2	pollution prevention
PLC	programmable logic controller
PS	pan solids
QA/QC	quality assurance/quality control
QMP	quality management plan
RPD	relative percent difference
RSD	relative standard deviation
SD	solids deposited
srm	standard reference material
SS	solids sprayed
TE	transfer efficiency
VOC	volatile organic compound
WBS	work breakdown structure

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1.0 INTRODUCTION

1.1 Purpose of the High Transfer Efficiency Spray Equipment GVP

The primary purpose of this document is to establish the generic verification protocol (GVP) for high transfer efficiency (TE) spray equipment, to which reference will be made frequently throughout this document as the High-TE GVP. The secondary purpose is to establish the generic format and guidelines for product specific Testing and Quality Assurance Plans (test/QA plans) that relate to this GVP.

Environmental Technology Verification Coatings and Coating Equipment Program (ETV CCEP) project level test/QA plans 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 High-TE test/QA plans 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. This GVP will ensure that project results are compatible with and complementary to similar projects. All ETV CCEP High-TE test/QA plans are adapted from this standard and the ETV Program Quality Management Plan (QMP). These test/QA plans 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 High-TE GVP

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

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

- Project Description
- Project Organization and Responsibilities
- Quality Assurance (QA) Objectives
- Site Selection and Sampling Procedures
- Analytical Procedures and Calibration
- Data Reduction, Validation and Reporting
- Internal Quality Control (QC) Checks
- Performance and System Audits
- Calculation of Data Quality Indicators
- Corrective Action
- Quality Control Reports to Management
- Appendices

1.4 Formatting

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

- Title Page
- Test/QA Plan 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 test/QA plan Approval Form for each piece of equipment tested. Acknowledgment by each key person indicates commitment toward implementation of the plan. Figure 1 shows the Approval Form format to be used.

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. Coating application equipment is continually being developed or redesigned to reduce any detrimental effects to the environment. This is primarily accomplished by increasing the TE of the coating operation and, therefore, reducing the amount of coating used, (i.e., less overspray) and VOCs released into the environment. Often these coating equipment 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 equipment in their particular application and may be constructively skeptical of the equipment provider's claims. If an unbiased, third-party facility could provide pertinent test data, environmentally friendly coating equipment 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 will verify the performance of high-TE spray equipment.

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 Pollution Prevention (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 Demonstration Factory 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. 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 ETV CCEP 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.

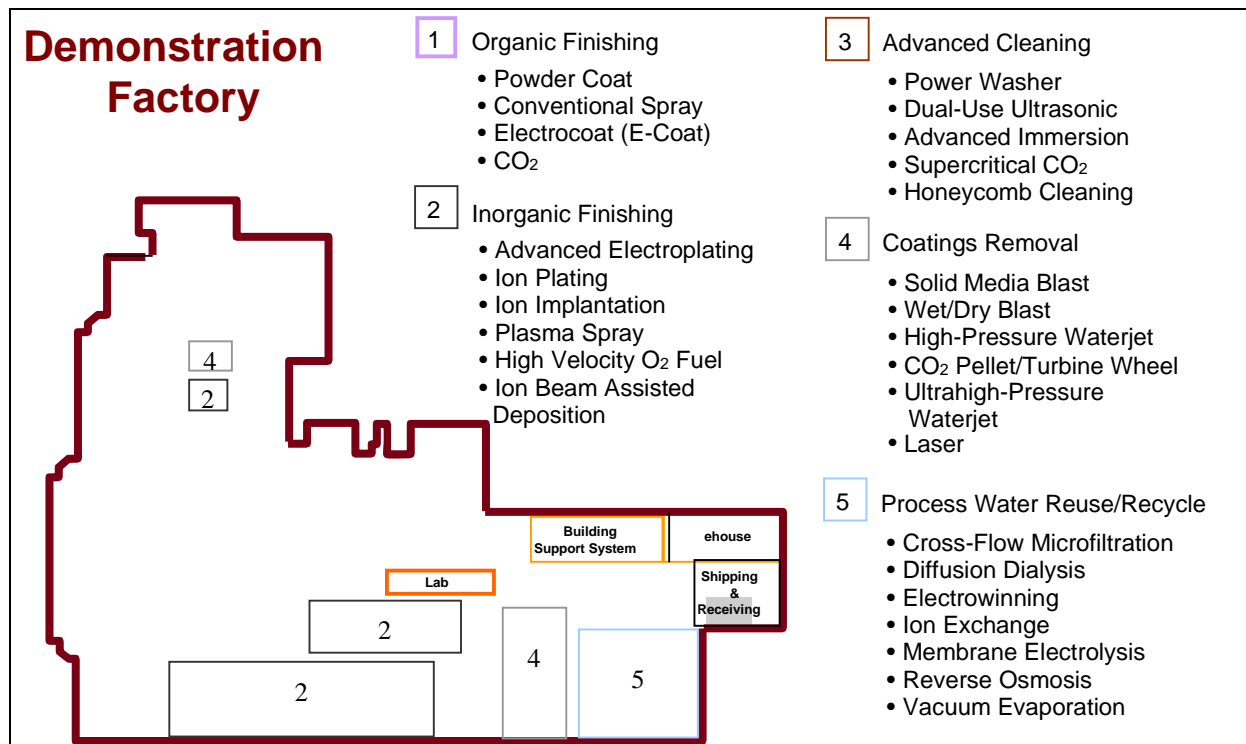


Figure 2. Demonstration Factory Layout

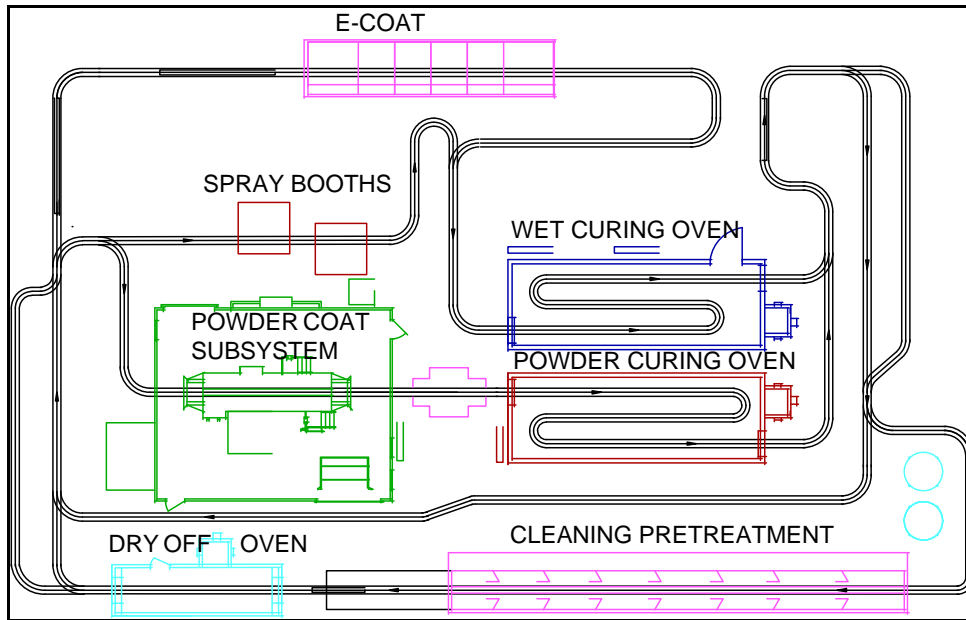


Figure 3. Demonstration Factory OFL

In the event that a particular technology demonstration or laboratory analysis cannot be performed at *CTC*, arrangements will be made to ensure the requirements of the test/QA plan and all associated QA procedures are completed.

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 equipment projects.

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 P-E Headspace GC/ECD/FID
Destructive and Nondestructive Evaluation	Evaluation of product and process performance, and surface cleanliness.	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 Nikon and Polaroid Light Optical Microscopes EDAX Energy Dispersive Spectrometer Impact Testers
Calibration Laboratory	Calibration of equipment, sensors, and components to nationally traceable standards.	Transmation Signal Calibrator (milliamps, millivolts) Thermal Dry Block Calibrator (Temperature) Druck Pressure Calibrator (Pressure) Fluke Digital Multimeter (Voltage)

2.2 Technical/Experimental Approach and Guidelines

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

- Develop product-specific test/QA plan
- 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 test/QA plan.

Table 2. Overall Guidelines and Procedures Applied to Test/QA Plans

- | |
|--|
| <ul style="list-style-type: none"> • A detailed description of each part of the test will be given. • Critical and non-critical factors will be listed. Non-critical 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 test/QA plans 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 select the performance parameters to be verified and recommend the optimum equipment settings for application and curing;
- The ETV CCEP will obtain enough test panels and foil for the verification and baseline tests;
- The ETV CCEP will obtain enough coating to complete the verification and baseline tests;
- The vendor will provide the high-TE spray gun and all necessary accessories to be verified;
- The ETV CCEP will obtain the baseline spray equipment;
- Data such as foil or panel weight (before coating and after curing), quantity of sprayed coating, quantity of supplied coating, and mil thickness of coating will be collected, following the ASTM International 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 test performed per this GVP are to verify the transfer efficiency and the finish quality achieved by the candidate technology and determine the technology's P2 benefits relative to a baseline. During the coating application phase, parameters such as: inlet air pressure, outlet air pressure, and airflow will be measured. During the laboratory analysis phase, coated test panels and foils will be used to measure TE. At a minimum, coated test panels will also be used to measure parameters such as: dry film thickness (DFT), gloss, distinctness-of-image (DOI), and visual appearance. The vendor may request additional performance tests to verify a specific claim.

2.2.3 Large Target Description

The large target will consist of an uncoated steel plate backboard measuring 91.4 cm by 91.4 cm (36 in. x 36 in.) attached to a stationary stand in the middle of the spray booth. The backboard will be covered with heavy gage (approximately 50 μm (0.002 in.)) aluminum foil by wrapping the excess foil around the edges of the backboard. Clean pre-weighed foil will be used to determine TE. In addition, cold-rolled steel panels will be coated to determine finish quality.

Each spray gun will utilize multiple passes per coat on the finish quality panels and foils using 50% overlap. The pattern for applying the coats

will typically follow passes 1, 2, 3, then 4 (see Figure 4). If a second coat is necessary, the pattern of the first coat will be repeated after a predetermined flash time. All passes will begin and end on the backboard (i.e., no lead or lag overspray). Also, there will be no overspray above or below the backboard. All guns will travel the same horizontal distance while spraying for each pass. All guns will be operated at the same distance from the target. The fan pattern heights will vary depending on the characteristics of the gun-coating interaction. The spray guns will typically be operated with the fluid and fan adjustments set at full open. However, the maximum variation between the fan patterns for each coating will be no greater than 2.5 cm (1 in.). In other words, assuming the smallest fan pattern is 25.4 cm (10 in.) for a particular coating, no spray gun shall have a fan pattern greater than 27.9 cm (11 in.) for that coating.

For each large target combination, a minimum of four (4) samples will be collected per gun, per coating. First, three TE (foil only) samples will be collected. Then, the backboard will be covered with a clean piece of aluminum foil. A cold-rolled steel finish quality panel, meeting SAE 1008 specifications, measuring 30.5 cm tall by 45.7 cm wide (12 in. by 18 in.), and treated with zinc phosphate at *CTC*, will be attached to the center of the foil-covered backboard. The spray guns will coat the finish quality panels using the same application pattern as the TE foils. A minimum of one finish quality panel will be collected for each test combination. The finish quality panels must be prepared under conditions representative of those used to obtain the TE data.

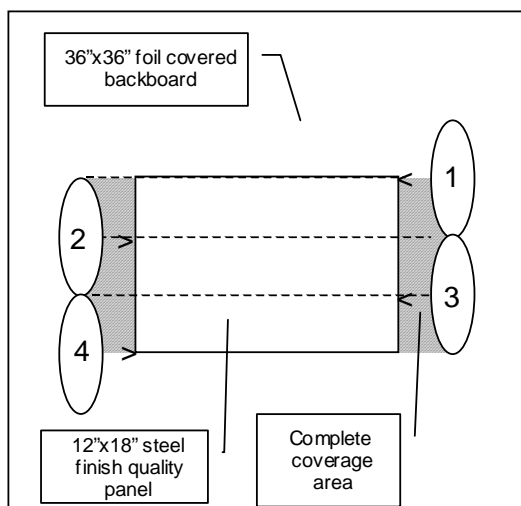


Figure 4. Large Target Application Diagram

2.2.4 Small Target Description

The small target will not use aluminum foil. The TE and finish quality analyses will be conducted on the same type of panel. Therefore, only three samples will be coated per gun per coating. The small panels will measure approximately 12.7 cm by 30.5 cm (5 in. x 12 in.) and will be made of 22-gauge cold-rolled steel meeting SAE 1008 specifications. The small panels will be obtained and will be treated with a zinc phosphate pretreatment at CTC.

Each spray gun will typically make 2 passes per coat on the small panels using 50% overlap. The pattern for applying the coats will be passes 1 then 2 (see Figure 5). If a second coat is necessary, the pattern of the first coat will be repeated after a predetermined flash time. Both the top and bottom passes will lose 50% of their fan pattern to overspray above and below the small panels. All passes will begin/end 6.4 cm (2.5 in.) from the leading/trailing edges of the fan pattern to the beginning/ending edges of the small panels (i.e., the spray guns will be triggered while in motion and when the center of the air cap is 6.4 cm plus half the horizontal width of the fan pattern away from the edge of the small panel). The guns will maintain a fan pattern height of 25.4 cm (10 in.) by varying the gun-to-target distance. The spray guns will be operated with the fluid and fan adjustments set at full open. Three small panels will be coated for each test combination and at least one of those panels will be randomly selected and evaluated for finish quality.

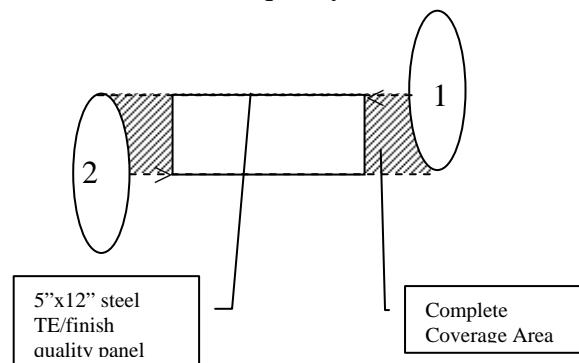


Figure 5. Small Target Application Diagram

The small panels will be manually transported into and out of the spray booth. A stand will be placed in the booth to hold the large backboard and the small panels. Figure 6 is a schematic of the small panels and the large finish quality panels showing the measurement locations for DFT and gloss.

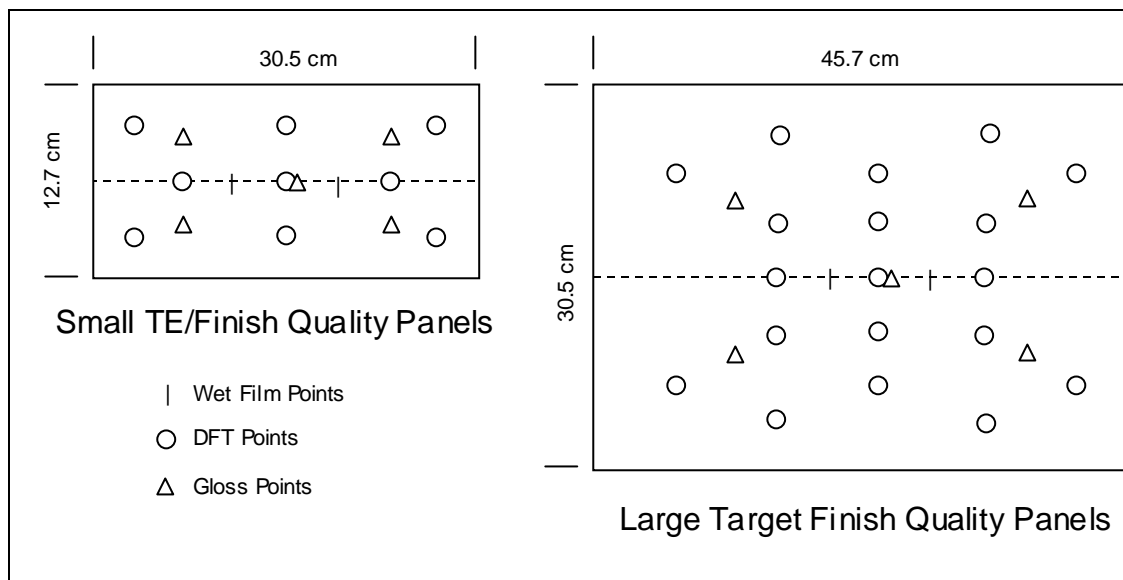


Figure 6. Test Panel Measurement Locations

The test will consist of a number of test combinations. Each test combination will consist of a spray gun (high-TE, high-volume, low-pressure (HVLP) #1, or HVLP #2), a coating (e.g., primer, basecoat, or topcoat), and a test panel (large combination foil, large combination finish quality panel, or small combination TE/finish quality panel). The large foils will not be used for finish quality and the large finish quality panels will not be used for TE analysis. The small panels will be used for both TE and finish quality analysis.

2.2.5 Coating Specification

The vendor will choose the test coating(s) based on its use in the target industry. The ETV CCEP will obtain a quantity of the test coating(s) to complete the verification and baseline tests. The test coating(s) will be prepared following the coating manufacturer's recommendations. The exact coating preparation procedures will be recorded. For comparison, the test coating(s) used during the verification test will be prepared the same as the coating batches prepared for the baseline test. Coating samples will be taken just prior to coating the test panels or foils to measure the coating temperature, viscosity, percent solids, volatile content and density. The coating measurements will be recorded on the coating batch worksheet.

2.2.6 Standard Apparatus

Figure 3 shows the testing location of the wet spray booth relative to the OFL. All testing will be performed in the same wet spray booth.

The test panels and foils will be attached to a stand in the spray booth. A programmable logic controller (PLC) will activate the motors that drive the linear motion translators. The translator can move the spray gun horizontally and vertically. The translator set-up could potentially cover an area approximately 1.37 m by 1.37 m (4.5 ft x 4.5 ft). The test panels and foils will be automatically sprayed using vertical overlap of the fan pattern. The spraying mechanism's PLC will control the triggering of the spray guns by way of pneumatically actuated clamp. During dwell time between passes, coating flow will be interrupted to minimize coating usage. Once the spray application is complete, the next rack or target will be moved into position.

The spray booth air filters will be changed prior to setting up the standard apparatus for the verification test. The pressure drop across the 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. As a comparison, the spray booth air filters will also be changed before the baseline spray guns are set up and tested as part of the TE baseline. This will minimize the difference in the initial air booth velocity between the guns. The air booth velocity will be measured in close proximity to the test panels or foils. Although the air velocity through the booth will exceed 0.5 m/s (100 ft/min), the velocity measured near the test panels or foils will be lower due to the disruption of the air currents by the test panel or foil.

After a target is coated, the next target in that test combination will be moved into position. After the test panels or foils have been cured, they will be transferred to the laboratory for analysis.

2.2.7 Process Standards

The cold-rolled steel panels will consist of two sizes (see Figure 6). The pretreatment method will be the same for all steel panels. The preparation of the test coatings used for the verification test will be the same as the HVLP tests. The TE analysis will follow Procedure A of ASTM D 5286. The environmental (ambient) conditions of the demonstration factory will be monitored, both inside the booth near the test panels or foils and near the outside of the curing oven. The curing process for the verification test will be similar to the baseline tests. Operating parameters during the verification test will be held relatively constant and will be comparable to the HVLP tests.

2.2.8 Design of Experiment

This GVP provides procedures used to determine the performance characteristics of high-TE spray equipment. A mean value and variance (or standard deviation) will be reported for each critical response factor. A confidence and specification limit of 95% will be applied to these tests.

Several test combinations will be used for each gun (multiple coating types, two target sizes). The order in which the combinations will take place will be randomized. This will enable both coating-to-coating and gun-to-gun variations to be determined for each response factor. The statistical analyses for all response factors will be performed using a statistical software package.

2.2.9 Performance Testing

The ETV CCEP will consult the manufacturers' recommendations for key operating factors to be used for testing, including the coating specifications: viscosity, weight % solids, etc. Recommended equipment settings for the coating will be obtained from the vendor. The ETV CCEP will test these conditions prior to starting the verification test. These conditions may be modified during the start-up phase to ensure proper gun performance. During the actual tests, no attempt will be made to optimize the equipment.

The high-TE spray equipment will be evaluated for both inlet and outlet air pressures and airflow. Test panels and foils will be used to measure equipment performance. The small panels and large finish quality panels will be used for DFT, gloss, DOI, and visual appearance. The small panels and large foils will be used for TE analysis. The coating characteristics may be affected by other parameters of the testing process, such as pretreatment, apparatus setup, and cleanup methods. The pretreatment process will be the same for all test panels; therefore, the variability of the pretreatment process should not be a significant factor. Non-critical control factors will be monitored or held relatively constant for the verification test. DFT measurements will be used to determine the variations in film thickness. Gloss, DOI, and visual appearance tests will be used to analyze the quality of the coating finish. TE measurements will be used to determine the quantitative difference between the high-TE spray equipment and a HVLP baseline. The TE test will follow Procedure A of ASTM D 5286.

The small panels and large foils will be weighed and the weights recorded prior to being placed in the spray booth. The weight of the gun, cup, coating, and coating container will be recorded on the worksheets immediately before applying the coating to each test panel. After each test panel or foil has been coated, the spray gun, cup, coating, and coating container will be re-weighed and the weights will be recorded. After the panels or foils are cured, they will be re-weighed.

2.2.10 Quantitative Measurements

In order to evaluate the TE and the finish quality obtained by using the high-TE and baseline spray guns, several measurements will be taken from the non-coated and coated test panels and foils. In the case of the non-coated panels or foils, the area in square feet and the weight of the TE foils or panels will be measured. For the coated panels or foils, weight of the TE foils and panels will be measured and DFT will be measured on the finish quality panels. This procedure will follow ASTM D 5286 whenever practical.

The uniformity of the coating applied can be determined by measuring DFT at several specified locations on the test panels. Measurements will be taken fifteen (15) locations on the large panels and at nine (9) locations on the small panels. Figure 6 displays the test panels with their respective locations of the film thickness and gloss measurements. Gloss measurements will be taken at five (5) locations on both the large and small panels. These sites will be numbered and measurements will be taken accordingly. The recorded measurements will be correlated to a specific site on each test panel for each test.

In addition to the performance analyses, the ETV CCEP will evaluate the potential environmental benefits associated with using the high-TE spray gun. Therefore, TE values will be quantitatively measured for each test combination using nearly identical test conditions as the HVLP baseline. A qualitative comparison will then be made to determine if the high-TE spray gun exhibits a comparable or higher TE than the HVLP baseline.

2.2.11 Participation

The vendor of the technology being verified is welcome to participate in the start-up phase and observe the verification and baseline testing. The ETV CCEP personnel will be responsible for performing all necessary test and demonstrations required for performance evaluation and full-scale validation.

2.2.12 Critical and Non-Critical Factors

For the purpose of this GVP, the following definitions will be used for critical control factors, non-critical control factors, and critical response factors. A critical control factor is a factor that is varied in a controlled manner within the design of the experiment to determine its effect on a particular outcome of a system. Non-critical control factors are all the factors that are to be held relatively constant or randomized throughout the testing for each specific piece of equipment (some non-critical factors may vary from equipment to equipment). Critical response factors are the measured outcomes of each combination of critical and non-critical control factors given in the design of experiments.

In this context, the term “critical” does not convey the importance of a particular factor (that can only be determined through experimentation and characterization of the total process), but its relationship within the design of experiments. In the case of the verification testing of a particular piece of coating equipment, the only critical control factors are the pieces of coating equipment themselves. All other processing factors will be held relatively constant (or randomized) and are non-critical 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 this GVP, the critical control factors, non-critical factors, and critical response factors are identified in a table format along with acceptance criteria (where appropriate), data quality indicators, measurement locations, and measurement frequencies, broken down by each run. For example, parameters associated with the test panel pretreatment will remain constant and thus be non-critical control factors, while a parameter such as DFT is identified as a critical response factor.

The only critical control factors are the high-TE and HVLP spray guns themselves (see Table 3). Examples of the non-critical control factors are shown in Table 4, and examples of the critical response factors to be measured are shown in Table 5.

For finish quality targets, the pretreatment process provides a continuous surface on which the test coating can then be applied. To verify that these panels have been pretreated properly, coating weights will be determined on three (3) large panels and three (3) small panels prior to the coating application phase.

Where appropriate, the output air pressure will be measured using a pressure gauge obtained from the spray gun manufacturers. The ETV CCEP will check the accuracy of these gauges before and after testing.

The airflow requirements of the high-TE and baseline spray guns will be determined during this test. The airflow will be measured using a calibrated flow meter. Data will be recorded in m³/min.

The DFT measurements will follow ASTM B 499 (Magnetic), and will be taken on all coated test panels. The gloss analysis will follow ASTM D 523, and will be taken on all coated test panels. DOI analysis will follow ASTM D 5767 Test Method B (except that an eight-bladed rotating disc will be used instead of the sliding combed shutter). The visual appearance analysis will use normal lighting to examine the surface of the coated panel. The panels will be examined for fish-eyes in the finish, the presence of orange peel, the evenness of the coating, and the difference in the visual gloss caused by sandpaper finish, drips, runs, and inclusions (such as dirt, fuzz, and fibers).

The TE test will follow ASTM D 5286. An average TE value will be determined for each combination.

The values in the total number column reflect the experimental design of coating eighty test panels.

Table 3. Critical Control Factors

Critical Control Factor	Air Cap	Fluid Nozzle	Fan Adjustment	Fluid Adjustment	Fan Pattern at the Target
High-TE	TBE	TBE	TBE	TBE	TBE
HVLP #1	TBE	TBE	TBE	TBE	TBE
HVLP #2	TBE	TBE	TBE	TBE	TBE

TBE – To be established in each product specific test/QA plan

Table 4. Non-Critical Control Factors

Non-Critical Factor	Set Points/ Acceptance Criteria	Measurement Location	Frequency	Total Number for the Test
Product Involved in Testing	Two sizes of test panels	Factory floor	TBE based on the number of test coatings chosen	TBE
Pretreatment Analysis	Varies <1.2 g/m ²	Random panels removed prior to start-up	3 large and 3 small from initial lot of panels	6
Surface Area of Test Panels	Varies <10% within and between tests	Factory floor	Once per test combination	TBE
Ambient Factory Relative Humidity	Varies <10% during test	Factory floor	Once per test combination	TBE
Ambient Factory Temperature	Varies <5 °C during test	Factory floor	Once per test combination	TBE
Spray Booth Relative Humidity	Varies <10% during test	Factory floor	Once per test combination	TBE
Spray Booth Temperature	Varies <5 °C during test	Factory floor	Once per test combination	TBE
Spray Booth Air Flow	0.4-0.6 m/s (80-120 ft/min)	Factory floor	Once per test combination	TBE
Temperature of Panels as Coated	Varies <5 °C during test	Center of test panel	Once per test combination	TBE
Distance from Gun to Panels	Varies <1.3 cm (<0.5 in.) during test	Factory floor	Once per test combination	TBE
Horizontal Gun Traverse Speed	TBE	Factory floor	Once per test combination	TBE
Vertical Drop Between Passes	TBE	Factory floor	Once per test combination	TBE
Volatile Content of Applied Coating	Varies <5% for each coating	Sample from coating pot	1 sample per test combination	TBE
Density of Applied Coating	Varies <50 g/L for each coating	Sample from coating pot	1 sample per test combination	TBE
Wt.% Solids of Applied Coating	Varies <5% for each coating	Sample from coating pot	1 sample per test combination	TBE
Coating Temperature, as Applied	Varies <5 °C during test	Sample from coating	1 sample per test combination	TBE
Coating Viscosity as Applied (#4 Ford)	Varies <5 seconds for each coating	Sample from coating pot	1 sample per test combination	TBE
Cure Time	1 hour	Factory floor	Once per test combination	TBE
Cure Temperature	110 °C (230 °F)	Factory floor	Once per test combination	TBE

TBE – To be established in each product specific test/QA plan

Table 5. Critical Response Factors

Critical Response Factor ¹	Measurement Location	Frequency	Total Number for the Test
Dynamic Inlet Air Pressure	Factory Floor	Once per test combination	TBE
Dynamic Outlet Air Pressure (Air Cap)	Factory Floor	Once per test combination	TBE
Air Consumption	Factory Floor	Once per test combination	TBE
DFT (Magnetic method)	Figure 6 shows location of measurement points.	15 points on each large panel 9 points on each small panel	TBE
Gloss	From ASTM D 523	5 points on each panel	TBE
DOI	ASTM D 5767 Test Method B ²	1 point on one random panel per test combination	TBE
Visual Appearance	Entire test panel	1 per panel	TBE
Transfer Efficiency	From ASTM D 5286	One per test combination (average of all panels in combination)	TBE

TBE – To be established in each product specific test/QA plan

1 See Sections 2.1.3 and 2.2 for the environmental basis to which these factors relate.

2 Will follow the ASTM International method except that an eight-bladed rotating disc will be used instead of the combed shutter. This is an optional test, dependent on the types of coatings chosen.

Some target factors that may be used to test high-TE spray equipment include:

- Overlap 50%
- Number of passes Established in test/QA plan
- Number of coats Established in test/QA plan
- Target dry film thickness Established in test/QA plan

2.3 Schedule

ETV CCEP uses standard tools for project scheduling. Project schedules are prepared in Microsoft Project, which is an accepted industry standard for scheduling. Project schedules show the complete work breakdown structure of the project, including technical work, meetings and deliverables. Table 6 shows the estimated schedule for the testing of high-TE spray equipment.

Table 6. Estimated Schedule as of 9/27/06

ID	Name	Duration	Start Date	Finish Date
Task 1	Approval of Test/QA Plan	30d	TBE	TBE
Task 2	Verification Testing	20d	TBE	TBE
Task 3	Complete Data Analyses	20d	TBE	TBE
Task 4	Prepare Verification Report	30d	TBE	TBE
Task 5	Approval of Verification Report	60d	TBE	TBE
Task 6	Issue Verification Statement	15d	TBE	TBE

TBE – To be established in each product specific test/QA plan

3.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

CTC employs a matrix organization, with program and line management, to perform projects. The laboratory supports the ETV CCEP project manager 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 manager on testing schedules. The ETV CCEP project manager will be responsible for preparing the test/QA plans and Verification Report and Statement for each test.

The ETV CCEP QA manager, who is organizationally independent of both the laboratory and the program, is responsible for administering *CTC* policies developed by the 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 manager 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 7. A summary of the responsibilities of each ETV CCEP participant, his/her applicable experience, and his/her anticipated time dedication to the project during testing and reporting is given in Table 7.

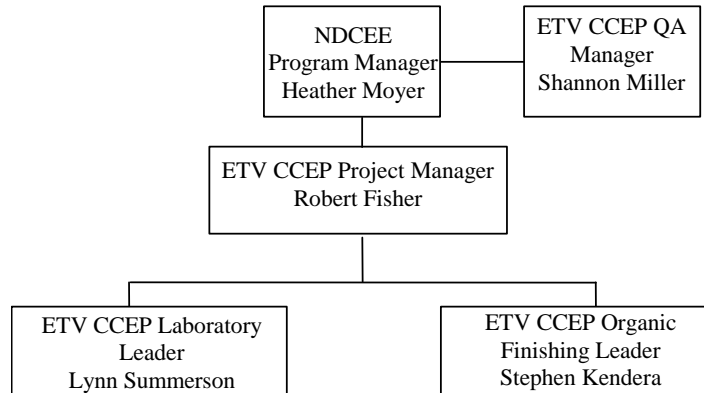


Figure 7. Project Organization Chart

Table 7. Summary of ETV CCEP Experience and Responsibilities

Key CTC Personnel and Roles	Responsibilities	Applicable Experience	Education	Time Dedication
Heather Moyer – NDCEE Program Manager	Manages NDCEE Program Accountable to CTC Technical Services Manager and CTC Corporate Management	Project Manager (10 years)	B.S., Chemical Engineering	1%
Shannon Miller – ETV CCEP QA Manager	Responsible for overall project QA Accountable to NDCEE Program Manager	Quality Mgmt. /ISO 9000 (6 years) Environmental Compliance and ISO 14000 Management Systems (6 years) ISO Internal Auditor (5 years)	B.A., Communications	5%
Rob Fisher – Staff Process Engineer/ ETV CCEP Project Manager	Technical project support Process design and development Accountable to NDCEE Program Manager	Organic Finishing Regulations (10 years) Organic Finishing Operations (10 years) Registered Professional Engineer	M.S., Manufacturing Systems Engineering B.S., Chemical Engineering	60%
Lynn Summerson – ETV CCEP Laboratory Leader/ Statistical Support Staff	Laboratory analysis Accountable to ETV CCEP Project Manager	Industrial and Environmental Laboratory Testing (22 years)	M.S., Chemistry B.S., Chemistry	15%
Stephen Kendera – ETV CCEP Organic Finishing Leader	QC Analysis Accountable to ETV CCEP Project Manager	Organic Finishing Operations (25 years)	N/A	5%

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 Manager, ETV CCEP Project Manager	EPA ETV CCEP Project Officer	Written Report Verbal Status Report	Monthly Weekly
ETV CCEP Project Manager	NDCEE Program Manager	Written or Verbal Status Report	Weekly
ETV CCEP Laboratory Leader	ETV CCEP Project Manager	Data Reports	As Generated
ETV CCEP QA Manager	NDCEE Program Manager	Quality Review Report	As Required
EPA ETV CCEP Project Officer	<i>CTC</i>	Onsite Visit	At Least Once per Year
Special Occurrence	Initiator	Recipient	Mechanism/ Frequency
Schedule or Financial Variances	NDCEE Program Manager or ETV CCEP Project Manager	EPA ETV CCEP Project Officer	Telephone Call, Written Follow-up Report as Necessary
Major (will prevent accomplishment of verification cycle testing) Quality Objective Deviation	NDCEE Program Manager or ETV CCEP Project Manager	EPA ETV CCEP Project Officer	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 Manager	Audits of verification testing operations and laboratory analyses
Statistical Support	Coordinates interpretation of test results

4.0 QUALITY ASSURANCE OBJECTIVES

4.1 General Objectives

The overall objectives of this ETV CCEP GVP are to verify the performance of high-TE spray equipment spray by establishing the TE improvement and by documenting finish quality. These objectives will be met by controlling and monitoring the critical and non-critical factors, which are QA objectives for each technology-specific test/QA plan based on this GVP. Tables 3 and 4 list the critical and non-critical control factors, respectively.

The analytical methods that will be used for coating evaluations are adapted from ASTM International 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 A (ASTM International 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 non-critical control factors. 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 evaluate test results and QA objectives.

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

Measurement	Method	Units	Precision	Accuracy	Completeness
Product Involved in Testing	Test panels	N/A	N/A	N/A	100%
Pretreatment Analysis	ASTM B 767	g/m ²	±0.005	±0.01	90%
Surface Area of Test Panels	Ruler	cm ² (ft ²)	±0.025 (±0.0036)	±0.025 (±0.0036)	90%
Ambient Factory Relative Humidity	Thermal Hygrometer	RH	±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	RH	±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 Air Flow	per ACGIH	m/s (ft/min)	±0.03* (±5)	±0.03* (±5)	90%
Temperature of Panels as Coated	Infrared (IR) Thermometer	°C	±0.5%	±1.0%	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%
Wt.% 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 (Ford #4 Cup)	ASTM D 1200	seconds	±10%	±10%	90%
Cure Time	Stopwatch	minutes	±5%	±5%	90%
Cure Temperature	Thermocouple	°C	±0.5 °C	±0.2 °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
Dynamic Inlet Air Pressure	Pressure Gauge	psig	±0.5 psig	±0.5%	90%
Dynamic Outlet Air Pressure (Air Cap)	Pressure Gauge	psig	±0.5 psig	±0.5%	90%
Air Consumption	Flow Meter	m ³ /min	±0.5% RPD	±0.5%	90%
DFT – Magnetic	ASTM B 499	mils ⁽¹⁾	20%	10% true thickness	90%
Gloss	ASTM D 523	gloss units	20%	±0.3	90%
DOI	ASTM D 5767 Method B	DOI units	20%	±3 DOI units	90%
Visual Appearance	N/A	N/A	N/A	N/A	N/A
Transfer Efficiency	ASTM D 5286	%	25%	RSD ≤ 20% ⁽²⁾	90%

(1) 1 mil = 0.001 in.

(2) Unknown according to ASTM D 5286

RPD = relative percent difference

RSD = relative standard deviation

N/A = Not Applicable

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, 80-120 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 per the specified test method. The degree of precision will be assessed based on the agreement of all replicates within a property analysis group.

4.2.3 Completeness

The OFL and laboratory strive for at least 90% completeness. Completeness is 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 OFL 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 non-conformance 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 non-conformance from test/QA plan QA objectives occurs, the cause of the deviation will be determined by checking calculations, verifying the test and measurement equipment, and re-analysis. If an error in analysis is discovered, re-analysis 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 non-conformance may be beyond the control of the laboratory. If, given that laboratory quality control data are within specification, any non-conforming results occur, the results will be interpreted as the inability of the coating equipment undergoing testing to produce panels meeting the performance criteria at the given set of experimental conditions.

4.3 Qualitative QA Objectives: Comparability and Representativeness

4.3.1 Comparability

Participating technologies will be operated per the vendor's recommendations. The data obtained will be comparable from the standpoint that other testing programs could reproduce similar results using a specific test/QA plan. Coating and environmental performance

will be evaluated using EPA, ASTM International, 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 characteristics of the HVLP baseline guns 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 suppliers' literature and verified by setup testing. If the test data meets the quantitative QA criteria (precision, accuracy, and completeness) then the samples will be considered representative of the technology under evaluation 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.

4.5 Impact of Quality

Due to the highly controllable nature of the test panel evaluation methods and predictability of factors affecting the quality of the laboratory testing of panels, the quality control of test panel performance characteristics is expected to fall within acceptable levels. Comparison of response factors will be checked for run-to-run process variations.

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. Application of the coating involves transporting test panels in and out of the spray booth. 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 coatings 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 and foils will be used in this project. These will be pre-labeled by marking their ID (identification) number with permanent marker on the untreated side of the test panels or foils. The number of test panels and foils processed during the testing depends on the experimental design, which in turn, depends on any equipment provider's claim(s) about performance and the respective confidence levels given in the responses to the Request for Technology. If no specific performance characteristics are requested for verification by the high-TE equipment providers, the default experimental design of three TE targets and one finish quality target per test combination will be used.

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

Table 12. Process Responsibilities

Procedure	Operations Technician	Laboratory Analyst
Visual inspection of test panels or foils		X
Numbering of test panels or foils		X
Initial weight of test panels or foils		X
Arrange test panels or foils in the spray booth		X
Prepare the coating	X	
Setup the high-TE gun or baseline guns	X	
Take coating samples and measurements		X
Load coating & prime gun	X	
Perform setup trials (before first run only)	X	
Initial weight of gun, pump, and coating container	X	X
Apply coating to test panels or foils	X	
Take process measurements		X
Cure test panels or foils	X	
Final weight of gun, pump, and coating container	X	X
Wrap/stack/transfer test panels or foils 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.

6.0 ANALYTICAL PROCEDURES AND CALIBRATION

6.1 Facility and Laboratory Testing and Calibration

The NDCEE shall maintain a record of calibrations and certifications for all applicable equipment used during ETV CCEP testing. 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 the ETV CCEP within the OFL and laboratory shall be recorded. Certified solutions and reference materials traceable to NIST shall be obtained as appropriate to ensure the proper equipment calibration. Where a suitable source of material does not exist, a secondary standard is prepared and a true value obtained by measurement against a technical-grade NIST-traceable standard.

After the coating is mixed, the temperature and viscosity of the coating will be measured. In addition, coating samples will be taken to the lab for density and percent solids analyses. All equipment used within the OFL during ETV CCEP testing will be calibrated according to relevant portion of Tables 13 and 14.

6.1.2 Laboratory Testing and Calibration Procedures

The analytical methods performed at *CTC* are adapted from standard ASTM International, MIL-SPEC, EPA, Association of Official Analytical Chemists 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 are calibrated before use and are 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, are outside method performance standards. As needed, equipment will be sent off-site for calibration or certification.

The listing of ASTM International Methods for dry film thickness, gloss, DOI, and transfer efficiency can be found in Appendix A. All equipment, used for these analyses, is calibrated according to Tables 13 and 14.

Like the test panels and foils, the solids pans will be prepared as specified by the ASTM International 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 is recorded on the coating batch worksheet.

The percent of solids is calculated as:

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

where: W1 = the 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 equipment manufacturer's instructions. As available, samples of known materials with established product qualities are used to verify that a system is functioning properly. For example, traceable thickness standards are used to calibrate the dry film thickness instrument. Applicable ASTM International methods are listed in Appendix A.

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 International and EPA, and from accepted protocols provided by industrial suppliers.

Table 13. Non-Critical Control Factor Testing and Calibration Criteria

Non-Critical Factor	Method	Method Type	Calibration Procedure	Calibration Frequency	Calibration Accept. Criteria(1)
Products Involved in Testing	Test panels	N/A	N/A	N/A	N/A
Pretreatment Analysis	ASTM B767	Chromate solution 50g/L CrO ₃	Comparison to NIST-traceable standard	With each use	80-120%
Surface Area of Test Panels	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 Air Velocity	per ACGIH	Anemometer	Sent for calibration or certification	Annually	Calibration or certification documentation
Temperature of Test Panels, as Coated	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
Wt. % Solids of Applied Coating	ASTM D 2369	Weight	Comparison to NIST-traceable standard	With each batch of coating	±0.003 g
Coating Temperature, as Applied	Thermometer	Thermometer	Comparison to NIST-traceable standard	Annually	±1 °C
Coating Viscosity, as Applied	ASTM D 1200	#4 Ford Cup	Comparison to NIST-traceable standard	Prior to each test	±10%
Cure Time	Stopwatch	Stopwatch	Sent for calibration or certification	Six months	N/A
Cure Temperature	Thermocouple	Thermocouple/ (controllers)	Comparison to NIST-traceable standard	Semi-annually	±1 °C

(1) As a percent recovery of a standard
N/A = Not Applicable

Table 14. Critical Response Factor Testing and Calibration Criteria

Critical Measurement	Method Number ⁽¹⁾	Method Type	Calibration Procedure	Calibration Frequency	Calibration Accept. Criteria ⁽²⁾
Dynamic Inlet Air Pressure	Manufacturer's recommendation	Pressure gauge	Comparison to NIST-traceable standard	Annually	±5 psig
Dynamic Outlet Air Pressure (Air Cap)	Manufacturer's recommendation	Test cap	Manufacturer's recommendation	Manufacturer's recommendation	Manufacturer's recommendation
Air Consumption	Manufacturer's recommendation	Flow Meter	Comparison to NIST-traceable standard	Six months	±1% of full scale
DFT	ASTM B 499	Magnetic	Comparison to NIST-traceable standard	Verify calibration after each run	90-110%
Gloss	ASTM D 523	Glossmeter	Comparison to NIST-traceable standard	Verify calibration after each run	90-110%
DOI	ASTM D 5767 Method B	Image analyzer	Manufacturer's recommendation	Manufacturer's recommendation	Manufacturer's recommendation
Visual Appearance	N/A	Visual	N/A	N/A	N/A
Transfer Efficiency (product and coating weights)	ASTM D 5286	Weight	Comparison to NIST-traceable standard	Verify calibration prior to each use	±3.0 g

(1) Listing of ASTM International methods to be used is provided in Appendix A.

(2) As a percent recovery of a standard

N/A = Not Applicable

6.4 Non-Standard Methods

CTC will not use any non-standard methods for this project. However, for methods that are non-standard (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 +/- 25 relative percent difference and should give values within +/- 20 percent of true or expected values for calibration and percent recovery check samples. For qualitative procedures, replicate results should agree as to their final evaluations of quality or performance (i.e., both should either pass or both should fail if sampled together from a properly functioning process). The intended use and any limitations would be explained and documented for a non-standard procedure.

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 is 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 signal or processed signal, and/or qualitative observations will be recorded. Comments to document unusual or non-standard observations also will be included on the forms as necessary. Raw data will be processed manually by the analyst, automatically by an electronic program, or electronically after being entered into a computer. The analyst will be responsible for scrutinizing the data according to specified precision, accuracy, and completeness policies. Raw data bench sheets, calculations and data summary sheets will be kept together for each sample batch. From the documented procedures and the raw data bench files, the steps leading to a final result may be traced.

7.1.1 Variables Used In Analysis

- CS - The mass of (wet) coating sprayed
- %S - The percent of the coating which is non-volatile (solids)
- SS - The mass of coating solids sprayed is equal to $(CS \times \%S) / 100\%$
- SD - The mass of solids deposited
- TE - Transfer efficiency is equal to $(SD / SS) \times 100\%$, expressed as a percentage

The accuracy of the TE values can be calculated based on the accuracy of each of the measurements involved. Random errors propagate as follows.

7.1.2 Error in Mass of Coating Sprayed.

The coating sprayed (CS) is the difference between two masses, the mass of the coating pot prior to and after applying the coating to the foils. The scale has an accuracy of ± 0.01 g. The mass of coating sprayed on each foil should be on the order of 50 g. Since two weight measurements must be made, and each contains an uncertainty of 0.01 g, the total uncertainty in a worst-case scenario is 0.02 g. The uncertainty in the mass sprayed, is $\pm 0.04\%$.

7.1.3 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 pan 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 2×0.0005 g. Since between 0.2 and 0.3 g of coating is used in the test, this uncertainty becomes negligible compared to the numerator uncertainty. Only about 0.05-0.1 g of solids 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.

7.1.4 Error in Mass Deposited.

The mass of the solids deposited on the foils is measured by weighing the foils before and after spraying. The scale used has an accuracy of ± 0.001 g. The mass of solids typically deposited on each foil is on the order of 20 g. A control foil is also weighed to determine whether the foils gain or lose weight during the curing process, which results in two additional weight measurements. Since four weight measurements must be made, and each contains an uncertainty of 0.001 g, the total uncertainty in a worst-case scenario is 0.004 g. The uncertainty in the mass deposited, is $\pm 0.02\%$.

7.1.5 Calculation of Transfer Efficiency.

SD is the weight of the product after spraying and curing, minus the weight of the bare product. SS is the product of CS and %S divided by 100. The transfer efficiency is calculated as below:

$$TE \% = (SD / SS) \times 100\%$$

The method for calculating %TE has been redefined (per ASTM D 5286) to consider the TE per run. By this method, the formula is as follows:

$$TE (\%) = \frac{\text{(average weight gain of test panels in a run)} \times 100\%}{\text{(weight of paint solids sprayed)} / \text{(number of panels per run)}}$$

An example calculation is included below:

$$\text{TE (\%)} = \frac{0.8 \text{ g} \times 100\%}{(4.8 \text{ g}) / 3}$$

$$\text{TE (\%)} = \frac{80\%}{1.6}$$

$$\text{TE (\%)} = 50\%$$

The relative TE improvement is determined using the equation below:

$$\text{TE}_{\text{RI}} (\%) = \frac{(\text{TE}_{\text{High-TE}} - \text{TE}_{\text{HVLP Ave}}) \times 100\%}{\text{TE}_{\text{HVLP Ave}}}$$

TE_{RI} – the relative improvement over the HVLP baseline average

$\text{TE}_{\text{High-TE}}$ – the average TE for the High-TE system

$\text{TE}_{\text{HVLP Ave}}$ – the average TE for all three HVLP guns

For example, for the TE data (High-TE = 60%, HVLP average = 50%):

$$\text{TE}_{\text{RI}} (\%) = \frac{(60\% - 50\%) \times 100\%}{50\%} = 20\%$$

7.2 Preliminary Data Package Validation

The generating operation technician and analyst will assemble a preliminary data package. 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 to the ETV CCEP laboratory manager.

7.3 Final Data Validation

The ETV CCEP laboratory manager shall be ultimately responsible for all final data released from this project. The ETV CCEP laboratory manager will review the final results for adequacy to project QA objectives. If the manager suspects an anomaly or non-concurrence with expected or historical performance values, with project QA objectives, or with method specific QA requirements of the laboratory procedures, he will initiate a second review of the raw data and query the generating analyst and the ETV CCEP laboratory leader about the non-conformance. Also, he will request specific corrective action. If suspicion about data validity still exists after internal review of laboratory records, the ETV CCEP

Laboratory manager may authorize a re-analysis. If sufficient sample is not available for re-testing, a re-sampling will occur. If the sampling window has passed, or re-sampling is not possible, the ETV CCEP laboratory manager will flag the data as suspect and notify the ETV CCEP project manager. The ETV CCEP laboratory manager will sign and date the final data package.

7.4 Data Reporting and Archival

7.4.1 Calculation of DFT

The DFT gauge has a stated accuracy of 0.1 mil. NIST traceable thickness standards will be used to calibrate the DFT gauge. DFT measurements will be made at several locations on each product. The location of each measurement is indicated in Figure 6.

7.4.2 Interpretation of the Numerical Results

The overall accuracy of the test data will allow calculation of TE to within a few percent. The largest uncertainty lies in the mass-used values, which contain a random error of about 2%, due to the solids calculation. The mass-deposited values are estimated to be within 1% and an overall accuracy of 3% leaves a reasonable margin.

7.4.3 Evaluation of the High-TE Spray Gun

A report signed and dated by the ETV CCEP laboratory manager will be submitted to the ETV CCEP project manager, the ETV CCEP QA manager, the EPA QA manager, and other technical principals involved in the project. 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 manager 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 staff, the vendor, 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 3-7 page Verification Statement summarizing the information contained in the Verification Report. After receiving the results and conclusions from the ETV CCEP project manager or the NDCEE program manager, the EPA will approve the Verification Report and Verification Statement. Only after agreement by the vendor, will the Verification Statement be disseminated.

8.0 INTERNAL QUALITY CONTROL CHECKS

8.1 Guide Used for Internal Quality Program

The ETV CCEP uses the NDCEE facility and its QA system to verify coating technologies. The NDCEE has established an International Organization for Standardization (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. The ETV CCEP verifications adhere to the ETV Program QMP, the ETV CCEP QMP, and the ANSI/ASQC standards.

Table 15. NDCEE Environmental Laboratory QA/QC Format Sources

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

8.2 Types of QA Checks

The NDCEE ETF Environmental Laboratory and OFL used by ETV CCEP follow published methodologies, wherever possible, for testing protocols. Laboratory methods are adapted from Federal Specifications, Military Specifications, ASTM International Test Methods, and supplier instructions. The laboratory adheres to the QA/QC requirements specified in these documents. In addition, where QA/QC criteria are not specified, or where the laboratory performs additional QA/QC activities, these protocols are explained in the laboratory's work instructions. Each NDCEE facility that uses supplied products implements its own level of QA/QC. During ETV CCEP testing, the NDCEE laboratory 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 OFL staff to ensure the appropriate parts, panels, samples, and operating conditions are used. The laboratory also monitors its reagent deionized water to ensure it meets purity levels consistent with analytical methodologies.

The filters are replaced quarterly before failures are encountered. Samples are not processed until the filters are replaced when failures do occur. 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 International 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 International 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 meet hazardous waste handling requirements.

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 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 International, Federal, and Military methods that are accepted in industry for product evaluations and supplier-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 A.

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 specified in the ETV Program and ETV CCEP QMPs. These include monthly managers meetings and reports, financial statements, EPA reviews and stakeholders meetings, and In-Process Reviews. The ETF laboratory also analyzes performance evaluation samples in order to maintain Pennsylvania Department of Environmental Protection Certification.

ISO Internal Audits

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

On-Site Visits

The EPA ETV CCEP project officer and EPA ETV CCEP QA manager may visit the ETV CCEP for an on-site visit during the execution of this project. All project, process, quality assurance, and laboratory testing information will be available for review.

Performance Evaluation Audits

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

Technical Systems Audits

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

Audits of Data Quality

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

10.0 CALCULATION OF DATA QUALITY INDICATORS

10.1 Precision

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

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

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_m = observed concentration of reference material

C_{srm} = 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 supplier for a given process parameter.

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 process or laboratory 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 laboratory manager level of data review. In all cases of nonconformance, the laboratory manager 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 NDCEE will contact the EPA ETV CCEP project officer. In cases of routine nonconformance, EPA will be notified in the NDCEE program manager's or ETV CCEP project manager's regular reports to the EPA ETV CCEP project officer. 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 manager, and on-site visits by the EPA ETV CCEP project officer, 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.

12.0 QUALITY CONTROL REPORTS TO MANAGEMENT

As shown on the Project Organization Chart in Figure 7, the ETV CCEP QA manager is independent from the project management team. It is the responsibility of the ETV CCEP QA manager to monitor ETV CCEP verifications for adherence to the ETV CCEP QMP. The laboratory manager monitors the operation of the laboratory on a daily basis and provides comments to the ETV CCEP QA manager to facilitate their activities. The ETV CCEP QA manager will audit the operation records, laboratory records, and laboratory data reports and provide a written report of the findings to the ETV CCEP project manager and laboratory manager. The ETV CCEP project manager will ensure these reports are included in the report to the EPA. The laboratory manager 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 the ETV CCEP in relation to current projects
- Limitations on conclusions, use of the data
- Planned QA activities, open items for next reporting period

APPENDIX A
ASTM International Methods

ASTM International Methods

- 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 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 2369 -- Standard Test Method for Volatile Content of Coatings
- ASTM D 3960 -- Standard Practice for Determining Volatile Organic Compound (VOC) Content of Paints and Related Coatings
- ASTM D 5286 -- Standard Test Method for Determination of Transfer Efficiency Under General Production Conditions for Spray Application of Paint.
- ASTM D 5767 -- Standard Test Methods for Instrumental Measurement of Distinctness-of-Image Gloss of Coating Surfaces