

US EPA ARCHIVE DOCUMENT

**Test/QA Plan for Testing of Dust Suppressant Products  
at Maricopa County, Arizona**

EPA Cooperative Agreement No. R 82943401 with RTI  
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MRI Project No. 101494

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Test/QA Plan for Testing of Dust Suppressant Products  
at Maricopa County, Arizona

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## LIST OF ACRONYMS/ABBREVIATIONS

ADQ	audit of data quality	in.	inch(es)
AED	MRI's Applied Engineering Division	kg	kilogram(s)
ANSI	American National Standards Institute	kph	kilometer(s) per hour
APCTVC	Air Pollution Control Technology Verification Center (ETV program at RTI)	L	liter(s)
		L/m <sup>2</sup>	liter(s) per square meter
		lb	pound(s)
		m	meter(s)
ASTM	American Society for Testing and Materials	m/s	meter(s) per second
		m <sup>2</sup>	square meter(s)
CAR	corrective action report	m <sup>3</sup> /s	cubic meter(s) per second
CE	control efficiency	mg	milligram(s)
cfm	cubic feet per minute	mg/ft	milligram(s) per foot
cm	centimeter(s)	mm	millimeter(s)
cm <sup>2</sup>	square centimeter(s)	mph	mile(s) per hour
cmh	cubic meter(s) per hour	MRI	Midwest Research Institute
cms	cubic meter(s) per second	NIST	National Institute of Standards and Technology
DQO	data quality objective	PEA	performance evaluation audit
EPA	U.S. Environmental Protection Agency	PM	particulate matter
ETV	Environmental Technology Verification (EPA program)	QA	quality assurance
		QC	quality control
FLW	Fort Leonard Wood	QMP	quality management plan
ft	feet	QSM	quality system manual
g	gram(s)	RSD	relative standard deviation
g/L	gram(s) per liter	RTI	Research Triangle Institute
gal	gallon(s)	SOP	standard operating procedure
gal/yd <sup>2</sup>	gallon(s) per square yard	TP	total particulate
GVP	generic verification protocol	TSA	technical systems audit
HAP	hazardous air pollutant	VOC	volatile organic compound
hi-vol	high-volume	µm	micrometer(s)
IFR	isokinetic flow rate		



**DISTRIBUTION LIST**

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Mary Ann Grelinger

## PREFACE

This test/QA plan was prepared by Midwest Research Institute (MRI) and Research Triangle Institute (RTI) for the Air Pollution Control Technology Verification Center (APCTVC). The test/QA plan provides a detailed plan for conducting and reporting results from a test of dust suppressant products in Maricopa County, Arizona. The plan was reviewed by Maricopa County, Midwest Industrial Supply, Inc., RTI, MRI, and EPA.

## SECTION A: PROJECT MANAGEMENT

### A1: Project/Task Organization

The U.S. Environmental Protection Agency (EPA) has overall responsibility for the Environmental Technology Verification (ETV) Program and the Air Pollution Control Technology Verification Center (APCTVC). Research Triangle Institute (RTI) is EPA's verification partner in this effort. For this work, Midwest Research Institute (MRI) is the testing organization for the APCTVC. The APCTVC has selected Maricopa County, Arizona as the site for this test of the following dust suppressant products.

1. Midwest Industrial Supply, Inc. – EK<sup>®</sup> 35 (dust suppressant)
2. Midwest Industrial Supply, Inc. – EnviroKleen<sup>®</sup> C (dust suppressant)

Management and testing of dust suppressants within the APCTVC are performed in accordance with procedures and protocols defined by a series of quality management documents. The primary source for the APCTVC quality system is EPA Order 5360.1 A2 (May 2000).<sup>1</sup> The quality system is in compliance with

1. EPA's *Requirements for Quality Management Plan Plans* (EPA QA/R-2),<sup>2</sup>
2. EPA's *Quality and Management Plan* for the overall ETV program (EPA ETV QMP),<sup>3</sup>
3. MRI's Applied Engineering Division (AED) Quality System Manuals,<sup>4</sup>
4. RTI's APCTVC QMP,<sup>5</sup>
5. The *Generic Verification Protocol (GVP) for Dust Suppression and Soil Stabilization Products*,<sup>6</sup> and
6. This test/QA Plan.

Table 1 summarizes these documents. This test/QA plan is in conformance with *EPA Requirements for Quality Assurance Project Plans* (EPA QA/R-5).<sup>7</sup>

MRI will, for RTI, conduct a field test of dust suppression products at Maricopa County, Arizona, analyze data, and prepare a report. The various quality assurance (QA) and management responsibilities are divided between EPA, RTI, and MRI key project personnel as defined below. The lines of authority between key personnel for this project are shown on the project organization chart in Figure 1.

#### A1.1 Management Responsibilities

Project management responsibilities are divided among the EPA, RTI, and MRI personnel as listed in Sections A.1.1.1 through A.1.1.6 below.

**Table 1. Quality Management Documents Applicable to This Test of Dust Suppressant Products at Maricopa County**

Document	Description
EPA Order 5360.1 A2 (May 2000) <sup>1</sup>	EPA Order 5360.1 A2 <sup>1</sup> includes quality specifications for EPA organizations that produce or use environmental data. The Agency-wide Quality System is a management system that provides the necessary elements to plan, implement, document, and assess the effectiveness of quality assurance (QA) and QA activities applied to environmental programs conducted by or for EPA. A consistent Agency-wide Quality System provides the needed management and technical practices to assure that environmental data used to support Agency decisions are of adequate quality and usability for their intended purpose.
<i>EPA Requirements for Quality Management Plan, EPA QA/R-2</i> <sup>2</sup>	This document provides the development and content requirements for Quality Management Plans for organizations that conduct environmental data operations for EPA through contracts, assistance agreements, and interagency agreements.
EPA ETV QMP <sup>3</sup>	EPA ETV QMP <sup>3</sup> lays out the definitions, procedures, processes, inter-organizational relationships, and outputs that will assure the quality of both the data and the programmatic elements of ETV. Part A of the ETV QMP contains the specifications and guidelines that are applicable to common or routine quality management functions and activities necessary to support the ETV program. Part B of the ETV QMP contains the specifications and guidelines that apply to test-specific environmental activities involving the generation, collection, analysis, evaluation, and reporting of test data. (EPA's <i>Quality and Management Plan for the Pilot Period (1995-2000)</i> , May 1998.)
MRI AED Quality System Manuals <sup>4</sup>	There are two Quality System Manuals for Environmental Systems including: <i>Quality Management Systems</i> , January 24, 2000, Revision 0 <sup>4</sup> and <i>Quality Systems for the Collection and Evaluation of Environmental Data</i> , August 1, 2000, Revision 0 <sup>4</sup> . These documents describe the quality systems in place for MRI's technical research unit participating in the APCT program. EED's quality manuals comply with American National Standards / American Society for Quality Control (ANSI/ASQC) Standard E4-1994. <sup>8</sup> The scope of these manuals encompasses performance criteria, requirements, and procedures for managing the quality of all work conducted by or on behalf of AED. Therefore, AED's quality manuals apply to all AED staff as well as people who perform work on behalf of AED, such as staff from other MRI research and administrative units, and others who contribute to projects managed by AED.
APCTVC QMP <sup>5</sup>	APCTVC QMP <sup>5</sup> describes the quality systems in place for the APCTVC. It was prepared by RTI and approved by EPA. Among other quality management items, it defines what must be covered in the GVPs and test/QA plans for technologies undergoing verification testing.
<i>GVP for Dust Suppression and Soil Stabilization Products</i> <sup>6</sup>	GVPs are prepared for each type of technology to be verified. These documents describe the overall procedures to be used for testing a specific technology and define the data quality objectives (DQO). With input from the Dust Suppressant Product Technical Panel, RTI and MRI prepared the <i>GVP for Dust Suppression and Soil Stabilization Products</i> <sup>6</sup> jointly with the Environmental Technology Evaluation Center and the Highway Innovative Technology Evaluation Center. The document was reviewed and approved by RTI and EPA.

**Table 1. (continued)**

Document	Description
This Test/QA Plan	This test/QA plan describes, in detail, how the testing organization will implement and meet the requirements of the <i>GVP for Dust Suppression and Soil Stabilization Products</i> <sup>6</sup> . The test/QA plan addresses issues such as the test organization's management structure, test schedule, test documentation, analytical methods, data collection requirements, and instrument calibration and traceability, and it specifies the QA and quality control (QC) requirements for obtaining verification data of sufficient quantity and quality to satisfy the DQO of the GVP.
<i>EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5</i> <sup>7</sup>	This document provides the Quality Assurance Project Plans requirements for organizations that conduct environmental data operations on behalf of EPA through contracts, financial assistance agreements, and interagency agreements. It provides suggestions on preparing, reviewing, and implementing QA Project Plans.

**A1.1.1 EPA Program Manager**

The EPA Program Manager, Theodore Brna, has overall coordination responsibility for the APCTVC. He is responsible for obtaining final EPA approval of project test/QA plans and reports.

**A1.1.2 RTI/APCTVC Director and RTI Task Leader**

The RTI/APCTVC Director is Jack Farmer. He has overall responsibility for the APCTVC and technology-specific verification tests. He will assign technology verification task leaders; oversee verifications; review technical panel makeup; and review GVP and test-specific documents. These responsibilities are described in greater detail in Section 2 of the APCTVC QMP.

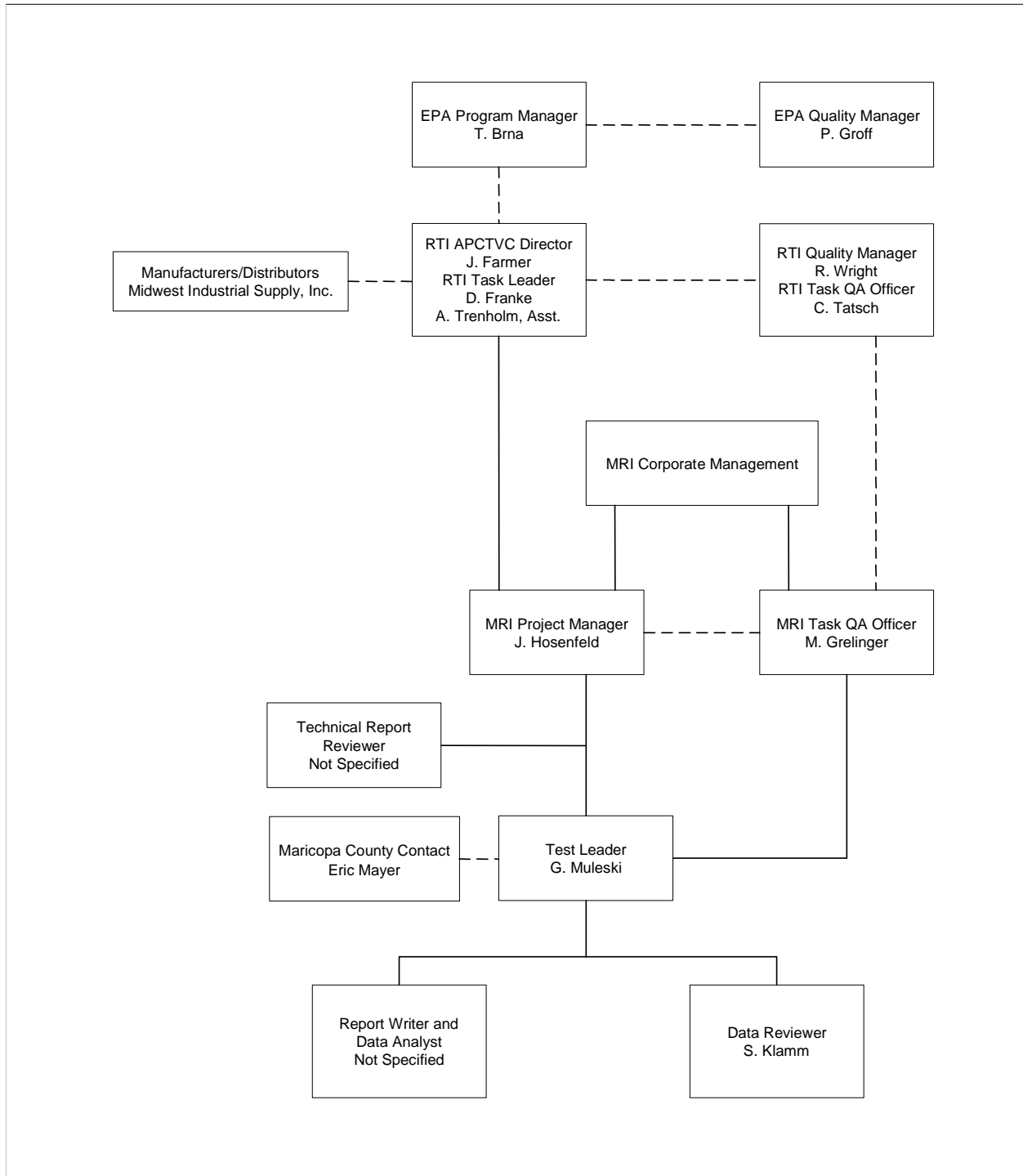
The RTI Task Leader, Deborah Franke, reports to the RTI/APCTVC Director. The Task Leader is responsible for any functions delegated to her by the RTI/APCTVC Director.

**A1.1.3 MRI Project Manager**

The MRI Project Manager for this verification test is John Hosenfeld. He will manage MRI's conduct of the dust suppressant test, select a test leader, develop staffing requirements, and propose a budget for the test. After a technical assessment, the MRI Project Manager is responsible for developing and implementing corrective actions within MRI. These responsibilities are described in greater detail in Section 1 of MRI's AED QSM. Mr. Hosenfeld has more than 30 years of experience in environmental regulation and measurements with research organizations and private industry.

**A1.1.4 MRI Test Leader**

The MRI Test Leader for this dust suppressant test is Greg Muleski. Dr. Muleski will manage the field testing and has responsibility for QC and on-site field activities. If test method QC



**Figure 1. Organizational chart.**  
**(Dashed lines indicate organizational independence)**

criteria are not met, he has the authority to halt testing until the sampling system is corrected and proven to meet the QC criteria. As the MRI Test Leader, he will oversee development of this test/QA plan and any standard operating procedures (SOPs) that are needed and prepare the draft

test report. Dr. Muleski is a principal scientist at MRI with more than 20 years of experience in the field of dust emission measurements.

#### **A1.1.5 MRI Data Reviewer**

The MRI Data Reviewer for the test is Scott Klamm. Mr. Klamm will, after the field test, be responsible for reviewing the field data package for completeness and general data quality. His function will be to serve as the first line, independent data quality reviewer of the field test data. Mr. Klamm has more than 10 years of direct experience in air pollutant measurements and related QA/QC procedures.

#### **A1.1.6 Facility Contact**

Eric Mayer will be the primary Maricopa County point of contact. Data provided by Maricopa County will be passed to the MRI Test Leader.

### **A1.2 Quality Assurance Responsibilities**

QA responsibilities are divided among the EPA, RTI, and MRI personnel as listed below.

#### **A1.2.1 EPA Quality Manager**

The EPA Quality Manager for the APCTVC is Paul W. Groff of EPA's Air Pollution Prevention and Control Division. In general, his responsibilities include:

1. Communicating quality systems requirements, quality procedures, and quality issues to the EPA Program Manager and the RTI APCTVC Director;
2. Reviewing and approving APCTVC quality systems documents to verify conformance with the quality provisions of the ETV Program's quality systems documents;
3. Performing technical systems audits (TSAs) and performance evaluation audits (PEAs) of APCTVC tests, as appropriate; and
4. Providing assistance to APCTVC personnel in resolving QA issues.

The EPA Quality Manager (or his designee) will perform the following specific activities associated with the tests of dust suppressants at Maricopa County:

1. Review and approve the GVP;
2. Review and approve the test/QA plan and the reports for dust suppressants verified at Maricopa County;
3. Conduct independent on-site technical and quality assessments of the tests of dust suppressants at Maricopa County; and
4. Determine whether the results of the tests of dust suppressants at Maricopa County conform to EPA quality requirements and whether test results attain the DQO.

### A1.2.2 RTI Quality Manager

The RTI Quality Manager for the APCTVC is Robert S. Wright of RTI's Center for Environmental Measurements and Quality Assurance. He is responsible for ensuring that all tests are performed in compliance with the QA requirements of the APCTVC QMP, GVPs, and test/QA plans. He has resources available to ensure conformance with the requirements and ensures that all personnel understand the requirements. Following are the general responsibilities of the RTI Quality Manager:

1. Preparing the APCTVC QMP and assisting the RTI APCTVC Director in the annual review and revision of this document, as needed;
2. Communicating with test-specific quality managers for specific tests;
3. Reviewing and approving the GVPs, test/QA plans, and any needed SOPs that will be developed by technology verification test leaders and test-specific quality managers;
4. Overseeing test-specific quality training;
5. Conducting independent technical and quality assessments in cooperation with the EPA Quality Manager and test-specific quality managers;
6. Reviewing and approving the test results and the QC results from tests;
7. Storing APCTVC documentation and data; and
8. Preparing the QA section of each test report.

The RTI Quality Manager will be assisted by the RTI Task QA Officer, C. E. Tatsch. They will perform the following specific activities associated with the tests of dust suppressants at Maricopa County:

1. Review the GVP;
2. Review the test/QA plan, test results, QC results, and the reports for dust suppression products;
3. Perform independent technical and quality assessments of the test of dust suppression products at Maricopa County; and
4. Determine whether the results of the tests of dust suppressants at Maricopa County conform to the APCTVC QMP and the test/QA plan and whether test results attain the DQO.

### A1.2.3 MRI Task QA Officer

The MRI Task QA Officer for this test is Mary Ann Grelinger. She will handle the QA activities directly associated with MRI's data collection and reporting for the dust suppressant test at Maricopa County. These activities will include:

1. Assist the Test Leader in preparing task-specific test/QA plans and SOPs to ensure that tests are implemented in conformance with these documents;
2. Conduct internal assessments of equipment calibration, equipment operation, sample handling, and data collection and reduction through oral communication with the testing team before the data packet has been prepared;



3. Perform internal on-site technical and quality assessments of the test of dust suppression products at Maricopa County to determine whether the tests of dust suppressants at Maricopa County are being implemented in accordance with the MRI quality system and the test/QA plan and prepare a written report of the assessment findings;.
4. Review test results within 30 days after each quarterly test campaign to make an independent determination whether QC criteria have been met and whether the project is on track to attain the DQO;
5. After all data has been analyzed, determine whether the tests of dust suppressants at Maricopa County conform with the MRI quality system and the test/QA plan and whether test results attain the DQO;
6. Upon completion of the testing and approval of the data packet by the MRI test leader, conduct an audit of data quality of a minimum of 10 percent of the quantitative data obtained in the field and laboratory to determine if they meet the specifications of the project and prepare a written report on the audit findings. Pseudo-random, systematic, and judgmental methods may be used to select the data to be reviewed;
7. Submit an assessment of test activities to MRI's program management and to RTI; and
8. Review the draft test report and participate in meetings with RTI's and MRI's program management.

For this project, Ms. Grelinger will report to MRI's QA Unit and will have no direct or indirect role in the data collection process. The QA Unit is a MRI corporate function that reports to senior corporate management and is independent of the section and division generating the data.

Ms. Grelinger is a Senior Environmental Scientist/Analyst with more than 20 years experience with emissions measurement and QA/QC activities. She has performed quality audits, directed quality reviews of emission inventories, and developed computer procedures to check emission inventory databases for completeness, consistency, and correctness.

## **A2: Problem Definition/Background**

The objective of the ETV APCTVC is to verify, with high data quality, the performance of air pollution control technologies. A subset of air pollution control technologies is products used to control dust emissions from unpaved roads. Control of dust emissions from unpaved roads is of increasing interest, particularly related to attainment of the ambient particulate matter (PM) standard. EPA recently issued a new ambient standard for particulate matter that specifies new air quality levels for particulate matter 2.5 micrometers ( $\mu\text{m}$ ) or less in aerodynamic diameter ( $\text{PM}_{2.5}$ ).

There are many products manufactured and sold to reduce unpaved road dust emissions. Two of these products, manufactured/distributed by one firm, are the subject of this test. The performance of these products will be assessed within a specified range of applicability as detailed in Section B1 of this test/QA plan, and reports will be produced. The goal of the test is to measure the performance of the products relative to uncontrolled sections of road.

### **A3: Project Description and Schedule**

#### **A3.1 Project Description**

Testing will be performed on two dust suppressant products on a rural, unpaved road in Maricopa County, Arizona. Test campaigns will be conducted at quarterly intervals over a 6-month period. Each test campaign will consist of five replicate dust emission measurements of controlled and uncontrolled road sections. Performance of the products will be determined in terms of dust control efficiency (CE) relative to uncontrolled roads. The CE will be determined relative to its decay over time and with traffic. The mobile dust sampler<sup>9</sup> will be used to obtain dust CE data for the products. The tests will gather information and data for evaluating the performance of the products as applied by the manufacturers/distributors. The critical measurement is the dust suppression CE. The specific conditions used during the testing will be documented. Table 2, in Section B2 of this test/QA plan, presents a summary of all measurements that will be made to either (1) evaluate the performance of the products or (2) document the test conditions.

#### **A3.2 Test Site Description**

The test will be conducted on rural, unpaved roads in Maricopa County, Arizona, approximately 50 miles west of Phoenix, near the towns of Buckeye and Wintersburg. The specific test locations are described in Section B2.1.

#### **A3.3 Product Descriptions**

The dust suppressant products to be evaluated during this test are described below.

Midwest Industrial Supply, Inc. – EK<sup>®</sup> 35: This product is a patent-pending dust control and soil stabilization agent formulated with continuous acting, long life synthetic fluids and naturally occurring rosos. It is uniquely developed with optimum environmental sensitivity especially for air, water, and stormwater criteria.

Midwest Industrial Supply, Inc. – EnviroKleen: This product is a patent-pending dust control and soil stabilization agent formulated with continuous acting, long life synthetic fluids and dust control modifiers. It is uniquely developed with optimum environmental sensitivity especially for air, water, and stormwater criteria.

<http://www.midwestind.com/envirokleen/envirobrochpg1.pdf> (for EnviroKleen)

#### **A3.4 Schedule**

The projected schedule for the dust suppressant test at Maricopa County is defined in Figure 2 and will start in February 2003.

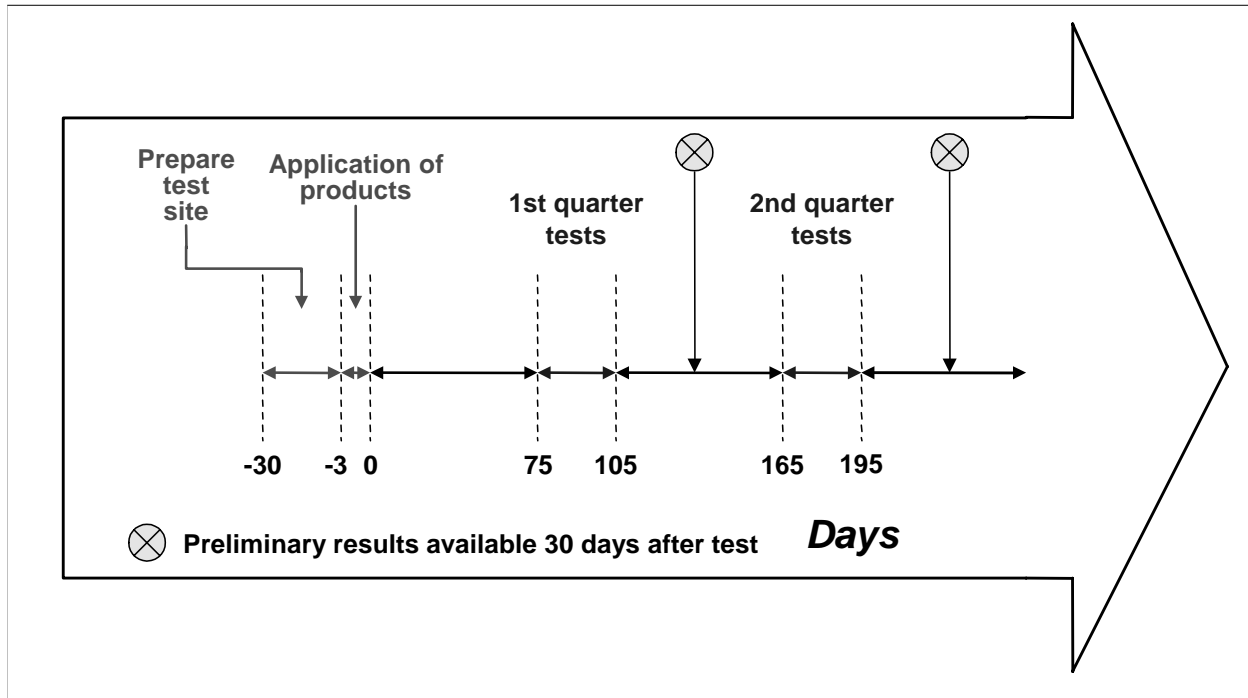


Figure 2. Projected schedule for the dust suppressant test.

#### A4: Quality Objectives and Criteria for Measurement Data

##### A4.1 Performance of the Products (DQO for Dust Suppression)

The performance of the dust suppressant products will be assessed using an experiment designed to achieve the DQO described below. The MRI Test Leader has the specific responsibility for QA of the on-site field testing and to run a mobile sampler quarterly criteria check as defined in the GVP. If method QC criteria are not met, he has the authority to halt testing until the sampling system is corrected and proven to meet the QC criteria. In addition, both the MRI Test Leader and the MRI Task QA Officer have responsibility to ensure that the tests conform to the MRI quality system and the test/QA plan. They both will determine independently within 30 days after each test campaign that the test results attain QC criteria and that the project is on track to meet the DQO. The critical measurement is the CE for the mobile dust sampler.

Product performance is the major determinant of the absolute magnitude of CE; however, CE is also influenced by climate and road characteristics. Climate may vary throughout the 6-month test, and both climate and road characteristics may vary with the location of the test site. Neither of these factors can be controlled to provide standardization of their effects on the measured product performance. The CE values will be provided; however, their primary value is to distinguish differences in product performance, e.g., at different times after application. Thus, the DQO focuses on the variability of the mobile dust sampler measurements, expressed in terms of CE. The DQO varies with CE and is set at  $(100-CE)/5$ , expressed in percent as the half-width interval for the 90 percent confidence limits. Use of a 90 percent confidence limit was judged

appropriate for open-source dust emission measurements that are subject to greater inherent variability than many environmental measurements. The DQO values are tabulated in Section D1.2. Compliance with the DQO calculation will be checked by the MRI Test Leader at the end of the last field test series. The derivation of this DQO is discussed in Section D1.2 and Appendix C.. There is also a mobile sampler quarterly criteria check defined in Section D1.1 which will help determine if the project is on track to meet the DQO.

#### **A4.2 Test Conditions**

While not critical, accurate measurement of test conditions such as road surface, traffic type and volume, and ambient conditions are important because the measurements define the conditions of the test. As specified in Section B2, Maricopa County personnel will obtain some of the measurements, while others will be supplied by MRI.

#### **A4.3 Associated Environmental Impacts for the Technology**

Associated environmental impacts will be measured by analysis of the products using composition and toxicity tests as specified in Table 2.

#### **A4.4 Associated Resources for the Technology**

Resources associated with use of the products are only the products and the equipment use and labor effort to apply them to the road. These measurements are specified in Table 2.

#### **A5: Special Training Requirements/Certification**

The MRI Test Leader has extensive experience (20+ years) in field testing of dust emissions from roads and other fugitive dust sources. He is familiar with the requirements of all of the test methods that will be used in the test. The MRI Test Leader will ensure that all persons assigned to the field crew have appropriate training and are fully capable of performing the tasks assigned to them. Each field crew member is thoroughly familiar with this test/QA plan, the measurement equipment, procedures, and methods for their assigned jobs. All field test personnel will receive the required and appropriate safety training, and a safety briefing will be given to all test team members by the MRI Test Leader.

#### **A6: Documentation and Records**

Requirements for recordkeeping and data management for the overall APCTVC program are found in Section 3.6 of the APCTVC QMP. All test data, calibration data, certificates of calibration, assessment reports, and test reports will be retained by MRI's APCTVC project files for a period of not less than 7 years after the final payment of the assistance agreement as per Part A, Section 5.3 of the EPA ETV QMP.

### **A6.1 Field Test Documentation**

The MRI Test Leader will oversee the recording of all field activities. The MRI Test Leader reviews all data sheets and maintains them in an organized file. The required test information is described in Section B. The MRI Test Leader or his designee also maintains a field notebook that documents the activities of the field team each day and any deviations from the schedule, test plan, or any other significant event.

Following the completion of a test run, the test technician will review the data recorded on the test run data sheets for completeness and accuracy. At the end of the test day, the MRI Test Leader will collect all data sheets completed during the day and will perform his own review of the sheets for completeness and accuracy. Of particular interest in this review is the notation of any significant deviation from planned test operations. The reviewed data will include field test data sheets, filter log sheets, and traffic logs. The electronic data logger used to record on-site wind data will be downloaded with the relevant files saved to two separate diskettes. Completed data forms associated with the tests will be removed from the site at the end of the day for safekeeping.

At the completion of individual field test campaigns (i.e., upon return to MRI's main laboratories), the MRI Test Leader will have copied two sets of data sheets and the electronic files containing the meteorological wind data. The MRI Test Leader will submit one copy of the data sheets and electronic files to the MRI Data Reviewer. The MRI Data Reviewer will ensure that all necessary information is available for input to the data analysis computer templates and review the field data package for completeness and general data quality. Following this review and confirmation that the appropriate data were collected, the MRI Data Reviewer will pass the data back to the MRI Test Leader.

The MRI Test Leader will independently oversee input of information to the same computer templates. The resulting files will be directly compared in a spreadsheet program and discrepancies noted and resolved. A final data analysis template will be created by the MRI Test Leader. He will run a mobile sampler quarterly criteria check as defined in the GVP, and with the MRI Task QA Officer, will determine if the project is on track to meet the DQO. If not, corrective action will be taken to ensure that the quarterly QC criterion is attained in subsequent test series. After completing all test campaigns, the data will be analyzed to determine if the DQO was met. The DQO analysis will be done using a statistical analysis technique, as discussed in Section 3 of the GVP. The reconciliation of the measurement data with the DQO will be done as discussed in Section D3 of this test/QA plan.

### **A6.2 Quality Control Records**

After the completion of tests, control test data, sample inventory logs, calibration records, and certificates of calibration will be stored with the test data in MRI's APCTVC project files. Calibration records will include such information as equipment being calibrated, date, person performing the calibration, standards used in the calibration, and raw data related to the calibration. To the extent practical, calibration records will be kept with the same data records

used with the calibrated equipment. For example, balance checks associated with filter weighing will be recorded in the filter weight book. Air sampler calibration records generated in the field will be kept with the field data sheets used to record the operation of those samplers. For equipment that has been calibrated prior to arriving at the field site (e.g., rotameters calibrated by MRI's instrument services, high-volume transfer standards, or miscellaneous field equipment such as thermometers and altimeters), the original data form or an exact copy of the original data form will be maintained in the MRI Test Leader's project file during the testing and then transferred to MRI's APCTVC project files. Final reports of self-assessments and independent assessments (i.e., TSA and audits of data quality (ADQ)) will be retained in the MRI's APCTVC project files, and copies of these reports will be included in the data packets that are sent to the APCTVC for review and retained by the APCTVC. Each report will contain a QA section, which will describe the extent that test data comply with the DQO.

### **A6.3 Reports**

The content and format for the reports are specified in Section 5 of the GVP. An outline of the Test Report is shown below in Section B10.1.3. Test reports will be prepared by the MRI Test Leader, will be reviewed by the MRI Project Manager and Task QA Officer, and will be submitted to the RTI Task Leader for review and approval by the APCTVC.



## SECTION B: MEASUREMENT/DATA ACQUISITION

### B1: Test Design

This test program is designed to determine the control performance of dust suppressants applied to unpaved roads. The test approach for dust suppressants is to measure the source emission strength of both the treated and untreated unpaved road surface. However, there are several features inherent to open dust sources (as opposed to more traditional stack sources) that must be addressed in the test design:

1. Unlike stack sources with “end of the pipe” controls, one cannot test simultaneously at the front and back ends to determine controlled and uncontrolled emission levels. In contrast, one must either (a) perform uncontrolled testing followed by a separate set of controlled tests after the suppressant is applied to the same section of road or (b) perform uncontrolled and controlled tests on separate sections of the test road. In other words, one must always separate the controlled and uncontrolled tests either spatially or temporally.
2. Next, all unpaved road dust suppression is time-dependent, decaying from roughly complete control at the time of application to essentially no control after some period of time (ranging from hours in the case of watering to months for chemical dust suppressants). Thus, no set of measurements during a single time period can characterize the long-term, average control performance. The extended period of time necessary to complete the test program as well as the method used to present the emissions control as a meaningful long-term average must be considered.
3. The extended period of time in Item 2 is further complicated by the open nature of the emission source. Unlike stacks, roads are exposed for a long period to the ambient conditions of precipitation and water erosion from neighboring areas, etc. Furthermore, the test program may be affected because of human intervention (such as damage to the treated surface from very heavy or tracked vehicles or vandalism).

The test program described in this plan is designed to address the above issues. Controlled and uncontrolled tests will be conducted on physically separated road sections. To guard against variability between different road sections, the test sections are located at nearby sites along the same road. This approach provides uniformity of both road construction and traffic. Control efficiency results will be plotted versus time (or cumulative number of vehicle passes). Ambient conditions and visible effects of human intervention will be monitored and any potential effect on the results will be assessed.

#### B1.1 Product Application

The manufacturers/distributors are responsible for applying their products to the assigned test sections. Manufacturers/distributors have been asked to supply written descriptions of their applications that discuss items such as: any preparation (grading) of the surface, any dilution of the product with water, equipment used to apply the product, target application intensity (i.e.,

volume of product per unit road surface area), number of application passes, and desired curing time without vehicle traffic. Manufacturers/distributors must make arrangements directly with Maricopa County to schedule application of their products. MRI will independently observe and record all application activities that occur on-site. The manufacturers/distributors and the county will keep MRI informed of all arrangements and scheduling. Scheduling should minimize any manufacturer/distributor needing to travel over another's freshly treated surface. The application method must comply with any Maricopa County requirements. The county will provide road traffic control during the application.

All steps at the test site in the application of each dust suppressant by the manufacturer/distributor will be observed. This includes surface preparation, mixing of the suppressant with water, and final application onto the road surface. A field notebook will be used to record these activities. Samples will be collected to quantify the volume of material applied to the road surface and to characterize the spatial distribution of material over the roadway.

## **B1.2 Data Design**

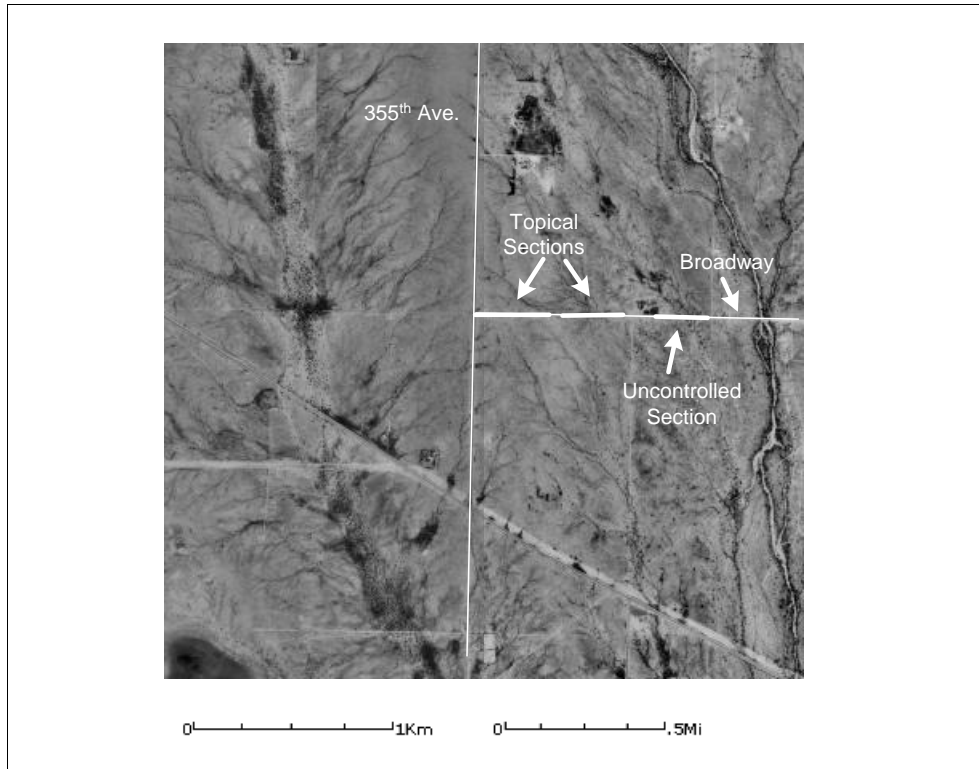
This test is designed to determine the performance of the subject dust suppressant products in terms of dust CE relative to uncontrolled roads. The CE will be determined relative to its decay over time and with traffic. Figure 2 shows the test schedule that will be conducted during the 6-month test. During each quarterly test, five replicate measurements will be made at the uncontrolled test section and at each product's test section. The mobile dust sampling method will be used at all of the test locations to measure dust emissions.

## **B2: Sampling Methods**

### **B2.1 Sampling Locations**

Figure 3 shows the test site and test sections. All test sections are located on Broadway Road (a county road) near the towns of Buckeye and Wintersburg in Maricopa County, Arizona. The sections used for dust suppressant testing will be on portions of the road constructed of shale. Based on information received from the Maricopa County Department of Transportation, the road experiences approximately 150 vehicle passes per day, with the majority of passes by light-duty cars and trucks. Much of the traffic appears to be associated with local residents commuting to their workplaces and thus occurs during the early morning and late afternoon hours. Traffic between 9 a.m. and 4 p.m. is infrequent. To accommodate the needs of different products, test sections have been established on Broadway Road near 355th Avenue. The uncontrolled measurements will be conducted on a separate section of Broadway Road as shown on Figure 3.





**Figure 3. Location of test sites to be used during the field program.**

## B2.2 Measurement Methods

Table 2 lists the measurement methods and they are discussed below.

Mobile dust sampling, an airborne dust sampling method, will be used during the test program to develop CE performance data. Testing of the road surface without product application (uncontrolled) and also after treatment will be conducted. The performance of road dust controls will be delineated by particle size: total particulate (TP or  $PM_{30}$ ),  $PM_{10}$ , and  $PM_{2.5}$ .

In addition to the airborne dust sampling, a number of additional samples/records that will provide supplementary information are also discussed below. These include:

1. Samples of the treated and untreated road surface material,
2. Visual evaluation of emissions from controlled and uncontrolled road surfaces,
3. Record of traffic over the treated road surface,
4. General meteorological records for the period from application to the end of testing, and
5. Documentation of the amount of water mixed with the product and product application.

**Table 2. Measurement Methods**

Factor to be Verified	Parameter to be Measured	Measurement Method	Frequency	Comment
<b>Performance Factor Parameters</b>				
Dust suppressant control efficiency	Uncontrolled dust emissions	Mobile dust sampler <sup>9</sup>	Quarterly (5 replicate test runs)	MRI to conduct tests for two dust suppressant products.
	Controlled dust emissions			
<b>Associated Parameters</b>				
Qualitative effectiveness of dust suppression	Dust emission	Maricopa County sampling device similar to the Colorado State University - Dustometer <sup>10</sup>	Quarterly (3 test runs)	Maricopa Co. to conduct tests for two dust suppressant products.*
<b>Associated Impacts of Using the Products</b>				
Whole effluent toxicity 40 CFR Part 136	Acute toxicity of product	EPA/600/4-90/027F <sup>11</sup> • Water fleas LC50 • Fathead minnow LC50 • Mysid shrimp LC50	Once for each product at start of test	MRI to conduct sampling. Analysis by ABC Labs.
	Chronic toxicity of product	EPA/600/4-91/002 <sup>12</sup> • Water fleas LC50 • Fathead minnow LC50 • Mysid shrimp LC50		
Biochemical oxygen demand (BOD) of product	5-day BOD	EPA Method 405.1 <sup>13</sup>	Once for each product at start of test	MRI to conduct sampling. Analysis by Tri-State Labs.
Chemical oxygen demand (COD)	COD	EPA Method 410.4 <sup>14</sup>	Once for each product at start of test	MRI to conduct sampling. Analysis by Tri-State Labs.
Evaporative VOC or HAP emissions from use of product	Composition of product	Manufacturer's/ distributor's MSDS sheet	Once for each product at start of test	Supplied by vendor before testing.
	VOC content of product	EPA Method 24 <sup>15</sup>		MRI to conduct sampling. Analysis by RTI.
Hazardous waste impacts	Toxicity of product	Toxicity Characteristics Leaching Procedure (TCLP) (EPA Method 1311) <sup>16</sup> • Inorganics/metals, EPA Method 6010B • Semivolatile organics, EPA Method 8270D • Volatile organics, EPA Method 8260B • Pesticides & herbicides, EPA Method 8270D	Once for each product at start of test	MRI to conduct sampling. Analysis by Tri-State Labs.

**Table 2. Measurement Methods (continued)**

Factor to be Verified	Parameter to be Measured	Measurement Method	Frequency	Comment
Total product testing	Chemical composition of product	TCLP (EPA Method 1311) <sup>16</sup> <ul style="list-style-type: none"> <li>• Semivolatile organics, EPA Method 8270</li> <li>• Volatile organics, EPA Method 8260B</li> <li>• Title 22 Metals, EPA Method 6010B</li> </ul>	Once for each product at start of test	MRI to conduct sampling. Analysis by Tri-State Labs.
Polyaromatic hydrocarbons using tentatively identified compounds (TIC)	Chemical composition of product	Semivolatile organics, EPA Method 8270 <sup>16</sup>	Once for each product at start of test	MRI to conduct sampling. Analysis by Tri-State Labs.
<b>Associated Resource Usage Parameters</b>				
Product application intensity	Number of test pans	Recordkeeping	During each application	MRI to conduct recordkeeping, measurements and calculations.
	Test pan tare mass/final mass	Balance		
	Test pan area	Measuring tape		
	Product density	Graduated cylinder and balance		
Product application resources	Description of equipment	Recordkeeping	During each application	MRI to conduct.
	Labor			
<b>Test Conditions Documentation Measurements</b>				
Method of application of product	Amount of water added to amount of product	Recordkeeping	During each application	MRI to conduct recordkeeping.
	How each product was applied			
Untreated soil properties	Type of soil	USGS <sup>17</sup>	Once at start of field testing program	MRI to collect samples and Maricopa Co. to analyze.*
Road surface samples	Silt loading	Dry sieving <sup>18</sup>	Once at start of field testing program	Maricopa Co. to collect samples. Analysis by MRI.
	Moisture content	Weight loss test <sup>18,19</sup>		
General observation of road conditions	Visual observation	Not applicable	Monthly or when on site	Maricopa Co. to conduct.*

**Table 2. Measurement Methods (continued)**

Factor to be Verified	Parameter to be Measured	Measurement Method	Frequency	Comment
Traffic	Vehicle type Vehicle weight Number of axles Vehicle passes	Periodic visual observation coupled with use of pneumatic traffic counter	Continuously	Maricopa Co. to conduct.*
Size of uncontrolled and controlled test sections	Length and width	Measuring device	Once	Maricopa Co. to conduct.*
Area climatic conditions	Wind speed and direction, rainfall, and ambient temperature	Local records of climatic conditions	Continuously	Information obtained from Arizona DEQ, see Section B2.2.4.

\* Maricopa County will provide all information to MRI for completion of the test reports.

### B2.2.1 Mobile Dust Sampling

The objective of the mobile dust sampling system is to produce relative (i.e., control efficiency) rather than absolute (i.e., mass emitted per vehicle-mile-traveled) emissions information. Also the emissions source, dust emissions from unpaved roads, has considerable variability at any time both when controlled and when uncontrolled. Thus, the mobile sampler and its operation were developed with the idea that precision is more important than accuracy<sup>9</sup>. Also its operation should avoid or "even out" potential systematic biases to the extent practical. This objective led to the physical placement of the sampler with its intake aligned along the truck centerline to avoid any possibility of bias related to crossroad winds. Other operating procedures were established to address wind-related issues as follows.

1. The truck travel speed is set well above ambient wind speeds (at the sampler height) so that plume flow dynamics at the sampler intake are dominated by the vehicle wake rather than ambient winds.
2. A nozzle is used that matches the sampling intake velocity to the truck travel speed.
3. A test consists of an equal number of multiple trips in both directions along the test road to "average out" the effect of wind direction and speed.

The mobile system consists of a high-volume (hi-vol) PM<sub>10</sub> cyclone combined with a PM<sub>2.5</sub> cyclone, as shown in Figure 4. The hi-vol sampler inlet is located approximately 1 meter (m) [3.3 feet (ft)] above the road surface and 2.5 m (8.2 ft) behind the pickup truck's (closed) tailgate. The sampler is located above the heaviest portion of the dust plume immediately behind the vehicle where it samples material that is truly airborne. The same truck, tires, and driver are used during all sampling runs at a location.

The primary air sampling device is a standard hi-vol air sampler fitted with a cyclone preseparator. The cyclone preseparator is shown in Figure 5. The cyclone exhibits an effective 50 percent cutoff diameter (D50) of approximately 10 micrometers ( $\mu\text{m}$ ) in aerodynamic diameter when operated at a flow rate of 68 cubic meters per hour ( $\text{cmh}$ ) [40 cubic feet per minute ( $\text{cfm}$ )]. Thus, mass collected on the 20- by 25-centimeter ( $\text{cm}$ ) [8- by 10-inch ( $\text{in}$ )] backup filter represents a  $\text{PM}_{10}$  sample.

Three PM size fractions will be sampled:  $\text{PM}_{10}$  on the 20- by 25-cm (8- by 10-in) filter,  $\text{PM}_{2.5}$  on the 47-millimeter ( $\text{mm}$ ) (1.9-in) glass fiber filter (URG-2000-30EH cyclones, fitted with filter holders), and coarse TP greater than  $\text{PM}_{10}$  within the main body of the cyclone. To avoid interference by large particles, intakes to the  $\text{PM}_{2.5}$  devices sample a small portion of the total flow through the hi-vol unit (Figure 4). To determine the sample weight of material that collects on the interior of the cyclone preseparator, the cyclone is washed with distilled water, and the wash water is collected in clean sample jars, which are capped and taped shut. The entire wash solution is passed through a Buchner-type funnel holding a tared glass fiber filter under suction. This ensures the collection of all suspended material on the filter.

Determination of the number of passes (or equivalently, the total distance over which the mobile sampler is operated) is an iterative process. The objective is to determine how to collect adequate sample mass within each of the three PM size ranges and avoid overloading the sampler. Secondly, the range of travel distances needs to accommodate a range of source conditions (i.e., from uncontrolled [0 percent CE] to very high levels of control [greater than 90 percent CE]). Furthermore, to maintain the mobile sampler's principal advantages over other sampling methods, the travel distance should not be so great as to require cycle times greater than 1 hour between back-to-back tests.

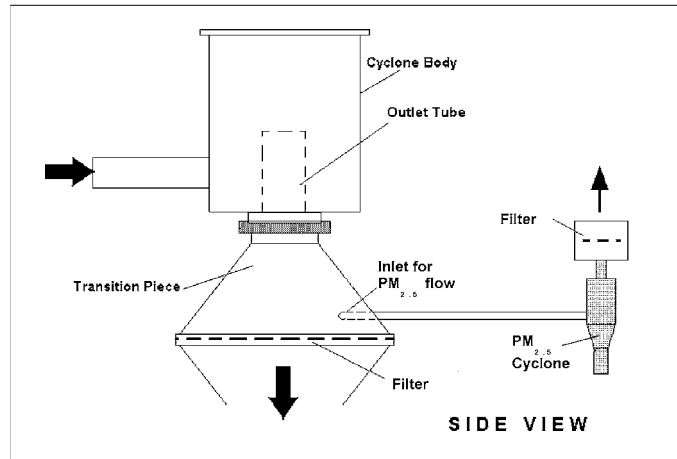


Figure 4. Hi-vol unit (fitted with  $\text{PM}_{2.5}$  cyclone).

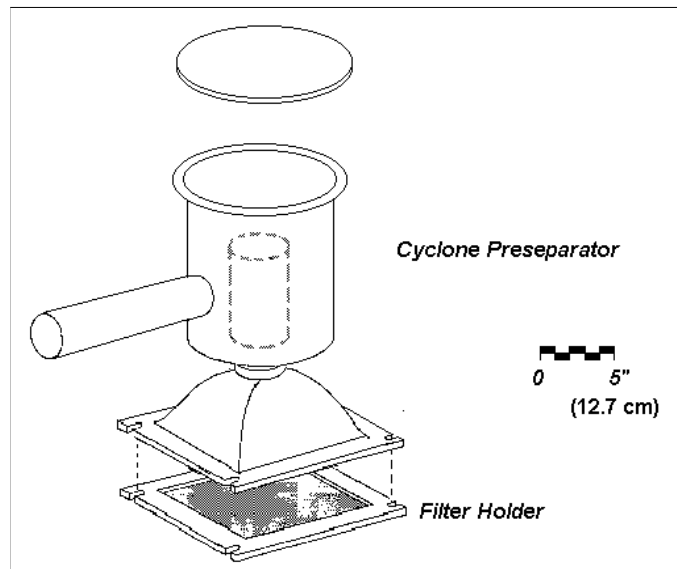


Figure 5. Cyclone preseparator.

Based on experience, the default number of passes for uncontrolled and controlled surfaces is 6 and 12, respectively. These defaults can be modified in several instances. During the initial uncontrolled tests, the exposed 20- by 25-cm (8- by 10-in.) filter and the cyclone wash can be visually examined to determine (a) if adequate or possibly excessive mass has been captured and (b) whether the number of vehicle passes should be increased or reduced, respectively.

Upon return to the main laboratories, the gravimetric analysis of the exposed and blank filters provides a more quantitative basis for judgment. It can be computed based on Equation 1:

$$R = \frac{M(\text{exposed filter}) - M(\text{blank filter})}{\text{STD of } M(\text{blank filters})} \quad \text{Eq. 1}$$

where:

R	=	measure of the level of quantifiable mass needed to achieve a reliable test
M (exposed filter)	=	mass collected (exposed filter), milligrams (mg),
M (blank filter)	=	mass collected (blank filter), mg, and
STD of M (blank filters)	=	standard deviation of M for blank filters determined from previous experience.

In general, one desires the ratio R to be 2 or more. This goal is more easily achieved for uncontrolled rather than controlled surfaces. In addition, it is easier to meet the goal for the coarser PM size ranges (i.e., TP and PM<sub>10</sub>) than for PM<sub>2.5</sub>.

Although one can collect more PM<sub>2.5</sub> mass by sampling for more passes (i.e., over a longer cumulative distance), two factors limit this approach. First, extended sampling will also produce additional mass in the PM<sub>10</sub> and TP size ranges and one must guard against overloading either the cyclone body or the 20- by 25-cm (8- by 10-in.) filter. Overloading in the first case would overstate the “true” amount of PM<sub>10</sub> mass attributable to the road. In the second case, sample mass could be easily lost from the overloaded filter and thus lead to erroneous results.

Calibration, maintenance, and operation of the hi-vol samplers incorporates the essential features of EPA’s guidance on PM<sub>10</sub> ambient air monitoring<sup>20</sup> with modifications allowing for the differences between ambient monitoring and mobile sampling. For example, individual sampler operating times are much less than the standard 24-hour period. Furthermore, because of the much higher than ambient concentration levels that will be encountered, the PM<sub>10</sub> and PM<sub>2.5</sub> sampler inlets are cleaned and the entire sampler inspected between each sampling event rather than at manufacturer-specified intervals. In addition, because calibration is performed more frequently, there is no need to incorporate anticipated seasonal variations in calibration of the device. For that reason, different formats are used to calibrate the transfer standard and the hi-vol devices for source-testing purposes. Additional details on sampler calibration, maintenance, and operation are provided in Section B7. Operating procedures for the mobile sampler are described in Appendix A.



The mass of dust collected during a sampling or blank run is calculated using Equation 2:

$$M = (W_F - W_T) \quad \text{Eq. 2}$$

where:

- M = mass collected, mg,
- W<sub>F</sub> = final mass of the filter, mg, and
- W<sub>T</sub> = tare mass of the filter, mg.

An emissions value is determined by dividing the sample mass by the cumulative length of road traveled by the mobile sampler using Equation 3:

$$e_m = \frac{M}{D} \quad \text{Eq. 3}$$

where:

- e<sub>m</sub> = emission value expressed as milligrams per meter of road traveled by the operating sampler, milligrams per meter (mg/m),
- M = mass, mg, and
- D = length of road traveled by the operating sampler, m.

The isokinetic flow ratio (IFR) is the ratio of a directional sampler's intake air speed to the mean wind speed approaching the sampler. It is given by Equation 4:

$$IFR = \frac{Q}{aU} \quad \text{Eq. 4}$$

where:

- Q = volumetric flow rate of the sampler, cubic meters per second (m<sup>3</sup>/s),
- a = sampler intake area, square meters (m<sup>2</sup>), and
- U = vehicle speed, meters per second (m/s).

This ratio is of interest in the sampling of TP, since isokinetic sampling ensures that particles of all sizes are sampled without bias. Specially designed nozzles are available for the hi-vol cyclone preseparator to maintain isokinetic (within 20 percent) sampling for wind speeds in the range of approximately 4.5 to 18 m/s [10 to 40 miles per hour (mph)]. Because the primary interest in this program is directed to PM<sub>10</sub> and PM<sub>2.5</sub> emissions, sampling under moderately nonisokinetic conditions should cause little bias. It is readily recognized that 10 μm (aerodynamic diameter) and smaller particles have weak inertial characteristics at normal wind speeds and therefore are relatively unaffected by anisokinesis.

On highly controlled surfaces, background PM concentrations may constitute a significant fraction of the total mass sampled. For that reason, background PM data will be collected from the nearest available ambient PM monitor. PM<sub>10</sub> and PM<sub>2.5</sub> are monitored on a 1-day-in-6 schedule at the Palo Verde station in Arlington, Arizona. The site, located at 36248 W. Elliott

Road, is within 10 miles of the ETV test sites. Sampling employs reference method dichotomous samplers. The site is maintained by the Arizona Department of Environmental Quality (DEQ). Continuous meteorological monitoring for the area is performed at the Allegheny site. This, too, is approximately 10 miles from the ETV test areas. Data will be obtained on a quarterly basis through the Arizona DEQ.

### **B2.2.2 Maricopa County Dust Collector Test**

A dust collecting unit mounted on a truck provides qualitative dust rating information on the relative effectiveness of the products. The dust collector device collects dust generated by the moving vehicle. The dust collector device consists of a filter box (with coffee filter) and a vacuum unit located in a truck bed; the sampler intake is located below the vehicle's rear bumper and samples dust thrown up from one of the rear tires. The vehicle makes one pass at 35 mph over a 0.5-mile test section. The filter is bagged, identified, and weighed. Three paired test runs are conducted to provide sufficient data to obtain a statistically significant average dust rating for the product. Note, that MRI will not provide QA on these data.

### **B2.2.3 Surface Sampling**

Surface samples will be collected from each test section (uncontrolled or controlled) evaluated. Duplicate samples will be collected. The samples will be analyzed for moisture and silt (i.e., fraction passing 200 mesh upon dry sieving). Sample collection and analysis will conform to EPA guidance in Appendices C.1 and C.2, respectively, to AP-42.<sup>18</sup> All sampling should be completed on the same day, with as short a time as practical between the sampling of the first and last sections.

MRI will coordinate Maricopa County's collection of samples at the time of the first quarterly test campaign. Roads must be dry to be sampled. If the road is visibly wet in the morning, sampling should wait until traffic and the sun have dried the surface. The road surface will be sampled and analyzed by the following procedure:

1. Ensure that the site offers an unobstructed view of traffic and that sampling personnel are visible to drivers. If the road is heavily traveled, use one person to "spot" and route traffic safely around another person collecting the surface sample (increment).
2. Using string or other suitable markers, mark a 0.3 m (1 ft) width across the road. (See the sample specifications given in Item 5 below.) Do not mark the collection area with a chalk line or in any other method likely to introduce fine material into the sample.
3. With a whisk broom and dustpan, remove the loose surface material from the hard road base. Do not abrade the base during sweeping. Sweeping should be performed slowly so that fine surface material is not injected into the air. Collect material only from the portion of the road over which the wheels and carriages routinely travel (i.e., not from berms or any "mounds" along the road centerline).



4. Periodically deposit the swept material into a clean, labeled container of suitable size (such as a metal or plastic 19-liter (L) [5-gallon (gal)] bucket) with a sealable polyethylene liner. Increments may be mixed within this container.
5. For uncontrolled unpaved road surfaces, a gross sample of 5 to 20 kilograms (kg) is desired. For surfaces treated with chemical dust suppressant, the above goal may not be achieved unless a very large area is swept. Continue taking additional increments from the controlled unpaved surface until the minimum sample mass of 200 grams (g) is achieved.
6. Measure and record the area that was sampled. Record necessary information on a data form.
7. Prepare the sample for storage. In general, a minimum of 400 g is required for silt and moisture analysis. Heavy samples may be split in the field with a riffle-type splitter to approximately 1000 g prior to shipment. The split sample should be placed in a clean (glass or plastic) sample jar with a screw-on lid. (If a splitter is not available, store the sample in multiple jars). Seal the jar lid using electrical tape for storage. If two jars are necessary, there should be notations “1 of 2 jars” and “2 of 2 jars” placed on the appropriate containers. Jars should be identified either by directly writing on the jar with a permanent marker or using an adhesive label. The label should contain sample identification (including test number if the sample is associated with a particular emission test), date of collection, initials of person collecting the sample, pertinent dimensions of the area sampled, and the number of sample splits (if any). Groups of approximately 12 samples are then placed in a container that also contains a sample inventory (tracking) sheet.

#### **B2.2.4 Ambient and Service Environment Records**

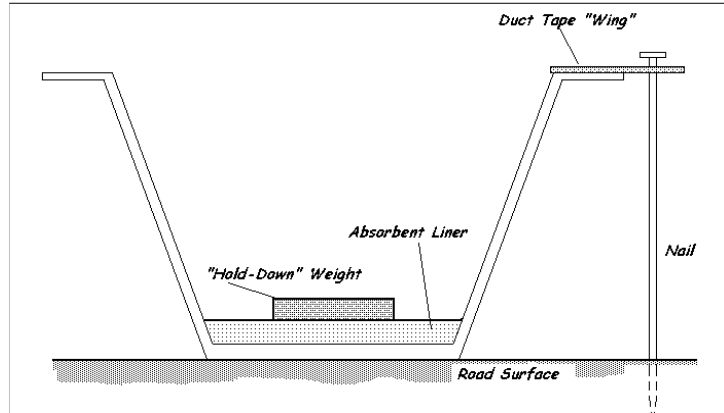
The degree of control achieved by an unpaved road dust suppressant depends on many types of factors. It is important that the test plan make provisions to quantify how the suppressant was applied and what service environment was experienced during the testing program.

The host facility will supply records on traffic over the test roads from the time that suppressants are first applied through the end of the test program. In addition, the nearest meteorological stations will supply ambient meteorological data for the period from product application until completion of the test program. At a minimum, the records will include daily precipitation, minimum and maximum temperatures, and wind speed and direction. Locations of the stations are defined in Section B2.2.1.

#### **B2.2.5 Product Application Rates**

Prior to application if water is added to or mixed with the product, the amount of water added will be documented. Product application rates will be measured by the following procedure:

1. Approximately 12 prepared suppressant spray sampling pans will be used for each test section being treated. The bottom of the pans (Figure 6) will be lined with an absorbent material (such as several layers of paper towels attached with duct tape, glue, etc.). In addition, the pans should have duct tape “wings” for nailing to the surface or should have a “hold-down” weight (such as a large bolt or washer). Each pan will be identified by a unique number or letter. Pans are tare weighed after being labeled and with wings or hold-down weights attached.



**Figure 6. Suppressant sampling pan.**

2. Distribute the sampling pans near the midpoint of the road section to be treated, with more pans toward the center of the test section than near the ends. Attempt to place pans so that the spray truck will straddle them. Record the location of each pan on a sketch of the test section in the field notebook.
3. Instruct the spray truck driver to (a) apply the suppressant to the test section in as normal a fashion as possible and (b) not attempt to “dodge” the sampling pans. Record spraying start and stop times. Photograph the application.
4. Once the test section has been treated, retrieve and reweigh the intact sampling pans. Record weights in the field notebook. Indicate which pans were crushed.
5. Collect a liquid sample in a tared, disposable graduated container. Record the mass of the container as well as the volume of liquid contained. Pour the liquid onto bare spots left by the pans on the road. The density of the recovered liquid is determined from a composite of the product caught in all of the pans, a portion of which is decanted into a graduated cylinder, using Equation 5:

$$\gamma = \frac{C_f - C_t}{V_\ell} \quad \text{Eq. 5}$$

where:

- $\gamma$  = density of recovered liquid, g/L,
- $C_f$  = mass, g, of the graduated cylinder containing recovered liquid,
- $C_t$  = tare mass, g, of the graduated cylinder, and
- $V_\ell$  = volume of liquid, L, in the graduated cylinder.

6. Using the density calculated above, determine the application intensity using Equation 6:

$$I = 10^4 * \frac{P_f - P_t}{a_p * \gamma} \quad \text{Eq. 6}$$

where:

- I = application intensity for each pan, liters per square meter (L/m<sup>2</sup>),  
 P<sub>f</sub> = full pan mass, g,  
 P<sub>t</sub> = pan tare mass, g,  
 a<sub>p</sub> = top surface area of pan, cm<sup>2</sup>, and  
 γ = density of recovered liquid, grams per liter (g/L).

7. Convert the results to units of gallons per square yard (gal/yd<sup>2</sup>) or L/m<sup>2</sup>. Calculate a mean and standard deviation over all intact pans. Record each value on a sketch of the test site. Examine if there is any discernible difference between one side of the road to another.
8. Record the application in the field notebook. Include data forms, photos, etc.

### B3: Sample Handling

The majority of environmental samples collected during the test program consists of PM captured on a filter medium. Analysis of these samples will be gravimetric, as described in Section B4.

To maintain sample integrity, the following procedure will be used. Each hi-vol filter will be stamped with a unique seven-digit identification number. A file folder will also be stamped with the identification number and the filter will be placed in the corresponding folder. Other filters also will be associated with a unique seven-digit identification number, although the number will be placed on the filter container rather than stamped on the filter itself.

Particulate samples are collected on glass fiber filters (20- by 25-cm [8- by 10-in.]) or on 47-mm (1.9-in.) glass fiber/quartz filters. Prior to the initial (tare) weighing, the filter media are equilibrated for 24 hours at constant temperature and humidity in a special weighing room. During weighing, the balance is checked at frequent intervals with standard American Society for Testing and Materials (ASTM) Class 1 weights to ensure accuracy. The filters remain in the same controlled environment for at least 24 hours after which a second analyst reweighs them as a precision check. A minimum of 10 percent of the filters and collection media used in the field will serve as blanks to account for the effects of handling. (Wash blanks are obtained by washing “clean” (unexposed) cyclone preseparators in the field.) The QC guidelines pertaining to preparation of sample collection media are presented in Section B5.

The hi-vol filters are placed in their folders. Groups of approximately 50 are sealed in heavy-duty plastic bags and stored in a heavy corrugated cardboard or plastic filing box equipped with a tight-fitting lid. Unexposed filters are transported to the field in the same truck as the sampling

equipment and are then kept in the field laboratory. The 47-mm (1.9-in.) filters are kept in separate holders, “face up” in groups of approximately 20.

Once they have been used, exposed filters are placed in individual glassine envelopes and then into numbered file folders. Groups of up to 50 file folders are sealed within heavy-duty plastic bags and then placed into a heavy-duty cardboard or plastic filing box fitted with a tight-fitting lid. Exposed 47-mm (1.9-in) filters are returned to their individual holders. All exposed and unexposed filters are always kept separate to avoid any cross-contamination. When exposed filters and the associated blanks are returned to the laboratory, they are equilibrated under the same conditions as the initial weighing. After reweighing, a minimum of 10 percent of each type are audited to check weighing accuracy.

#### **B4: Analytical Methods**

All analytical methods required to determine dust CE for this testing program are gravimetric methods. The final and tare weights are used to determine the net mass of particulate captured on filters and other collection media. The tare and final weights of blank filters are used to account for the systematic effects of filter handling. The determination of surface moisture and silt contents are also gravimetric in nature and are described in Appendix C.2 of AP-42.<sup>18</sup> The following procedures are followed whenever a sample-related weighing is performed:

1. An accuracy check at the minimum of one level, equal to approximately the tare weight and actual weight of the sample or standard. Standard weights should be ASTM Class 4 or better.
2. The acceptance criterion for the balance mass QC will be three times the balance’s repeatability.
3. If the balance calibration does not pass this test at the beginning of the weighing, the balance should be repaired or another properly calibrated balance should be used. If the balance calibration does not pass this test at the end of the weighing, the samples or standards should be reweighed using a balance that can meet these requirements.
4. Prior to weighing filters, the balance will be checked with ASTM Class 1 weights and will be checked at least once during every 4 hours of the weighing period. The balance checks should encompass the range of filter weights encountered.
5. ASTM Class 1 weights will be verified on an annual basis in accordance with ANSI/ASTM E617 requirements.<sup>21</sup>

Other analytical methods for this testing program are specified in Table 2.

**B5: Quality Control Requirements**

A quantitative QC criterion for the five replicate measures that comprise a test run during the quarterly tests was set. The estimated criterion is to achieve a relative standard deviation (RSD) for a test run of 0.334 or less. The RSD is calculated as:

$$RSD = \sqrt{\frac{\sum_{i=1}^5 X_i^2 - 5\bar{X}^2}{4}} / \bar{X} \quad \text{Eq. 7}$$

where:

$X_i$  = ith measurement, and  
 $\bar{X}$  = mean of 5 measurements.

This value is calculated by the MRI Test Leader after each quarterly series of tests. The quarterly criterion is described in more detail in Section D1.1

Tables 3, 4, and 5 list the QC procedures for sampling media, sampling equipment, and miscellaneous instrumentation, respectively, for gravimetric methods used for dust CE. For the analytical methods used in Table 2, all QC specified by the referenced methods will be followed.

**B6: Instrument/Equipment Testing, Inspection, and Maintenance Requirements**

This is covered in the calibration and maintenance of sampling and analytical equipment.

**B7: Instrument Calibration and Frequency**

Calibration and frequency requirements for the balances used in the filter gravimetric analyses are given in Table 3.

Requirements for hi-vol sampler flow rates rely on the use of secondary and primary flow standards. The Roots meter is the primary volumetric standard and the BGI orifice is the secondary standard for calibration of hi-vol sampler flow rates. The Roots meter is calibrated and traceable to a National Institute of Standards and Technology (NIST) standard by the manufacturer. As noted in EPA's *Quality Assurance Guidance Document 2.11*, periodic recertification is not normally required under clean service conditions unless the meter has been damaged and must be repaired.<sup>20</sup> The BGI orifice is calibrated by MRI against the primary standard on an annual basis. Before going to the field, the BGI orifice is first checked to assure that it has not been damaged. In the field, the orifice is used to calibrate the flow rate of each hi-vol sampler. Table 4 specifies the frequency of calibration and other QC checks regarding air samplers.

Table 5 outlines the QC checks employed for the miscellaneous instrumentation needed.

**Table 3. Quality Control Procedures for Sampling Media**

Activity	QC Check/Requirement
Preparation	Inspect and imprint hi-vol glass fiber or quartz media with identification numbers. Inspect 47-mm filters and place in appropriate container (such as a polycarbonate petri dish). Place unique identification numbers on the filter container.
Conditioning	Equilibrate media for 24 hours in clean controlled room with relative humidity (RH) of 35% (variation of less than $\pm 5\%$ RH) and with temperature of 21 degrees Celsius (°C)[(70 degrees Fahrenheit (°F)] [variation of less than $\pm 3^\circ\text{C}$ ( $\pm 5.4^\circ\text{F}$ )].
Weighing	Weigh hi-vol filters to nearest 0.1 mg. Weigh 47-mm (1.9-in.) filters to nearest 0.01 mg.
Auditing of filter mass	Independently verify the mass of at least 10% of filters and substrates. Reweigh entire batch if the mass of any hi-vol filters deviate by more than $\pm 2.0$ mg. For tare mass, conduct a 100% audit. Reweigh any hi-vol filter whose mass deviates by more than $\pm 1.0$ mg.
Collection of field blanks	Conduct at least one complete field blank test for every 1 to 9 emission tests. Field filter blanks are loaded into sampling devices (which are then uncovered but never activated) and then retrieved. In all other respects, these blanks are handled in exactly the same manner as all other filters. Field wash blanks are collected by cleanly washing cyclone preseparators. These samples are then handled in exactly the same manner as all other wash samples.
Calibration of balance	Balance to be calibrated once per year by manufacturer's certified representative. Check prior to each use with ASTM Class 1 weights.

**Table 4. Quality Control Procedures for Sampling Equipment**

Activity	QC Check/Requirement <sup>a</sup>
Maintenance <ul style="list-style-type: none"> <li>All samplers</li> </ul>	Check motors, brushes, gaskets, timers, and flow measuring devices prior to loading onto the truck and upon arrival at each site prior to testing. Repair/replace as necessary. Recalibrate before use. Clean sampler interior surfaces between individual tests.
Calibration <ul style="list-style-type: none"> <li>Transfer Standard</li> <li>Mobile dust sampler</li> <li>Rotameters</li> </ul>	Orifice calibrated against displaced volume test meter annually. For 68 cmh (40 cfm) devices, calibrate sampler back plate pressure drop against orifice prior to use at each site. Recalibrate every 2 weeks. Flow rate should be within $\pm 10\%$ . Calibrate through MRI Instrument Services annually.
Operation <ul style="list-style-type: none"> <li>General</li> </ul>	Cover sampler inlets prior to and immediately after sampling to prevent static deposition from active sources.
<ul style="list-style-type: none"> <li>PM<sub>10</sub> cyclone (mobile dust sampler)</li> </ul>	Match nozzle to captive vehicle travel speed between 40 to 56 kph (25 to 35 mph). Set sampler flow rate to 68 cmh (40 cfm) at start of individual test. Activate sampler only during passage over 150-m (500-ft) test section.
<ul style="list-style-type: none"> <li>PM<sub>2.5</sub> cyclone (mobile dust sampler)</li> </ul>	Sampling rates set manually at start of individual test. Activate PM <sub>2.5</sub> samplers before PM <sub>10</sub> sampler and leave on during entire test period. Deactivate hi-vol device before the PM <sub>2.5</sub> sampler.

<sup>a</sup> "Mean" denotes a 5-minute average.



**Table 5. Quality Control for Miscellaneous Instrumentation**

Instrumentation	QC Check/Requirement <sup>a</sup>
Watches/stopwatches	The MRI Test Leader will compare an elapsed time (> 4 hours) recorded by his watch against the U.S. Naval Observatory master clock. Do not use if more than 3 minutes difference. All crew members will synchronize watches (to the nearest minute) at the start of each test day.
Field balances (used for application intensity determination)	Units calibrated by MRI Instrument Services on annual basis. Check prior to each day’s use in the field with a calibration weight.

<sup>a</sup> Activities performed prior to going to the field, except as noted.

**B8: Inspection/Acceptance Requirements for Supplies and Consumables**

The primary supplies and consumables for this field exercise consist of the air filters and collection media. Prior to stamping and initial weighing (Table 3), each filter is visually inspected and is discarded for use if any pin-holes, tears, or other damage is found.

**B9: Data Acquisition Requirements**

No indirect measurements will be made.

**B10: Data Management**

**B10.1 Data Flow**

**B10.1.1 Data Origination from Test Site**

Data and collection activities for dust emissions are shown in Figure 7. This flow chart includes all data activities from the initial pretest QA steps to the passing of the data to the MRI Test Leader.

The data activities include activities and assessments performed by the MRI Task QA Officer immediately preceding, during, and immediately after the field tests. These will include:

Before tests:

1. Discuss program requirements and data acquisition activities with test team members to verify that the personnel are aware of test requirements and are trained in proper QA procedures.
2. Review data acquisition formats (forms, computer file formats) to be used in the test program and make any recommendations for needed changes.

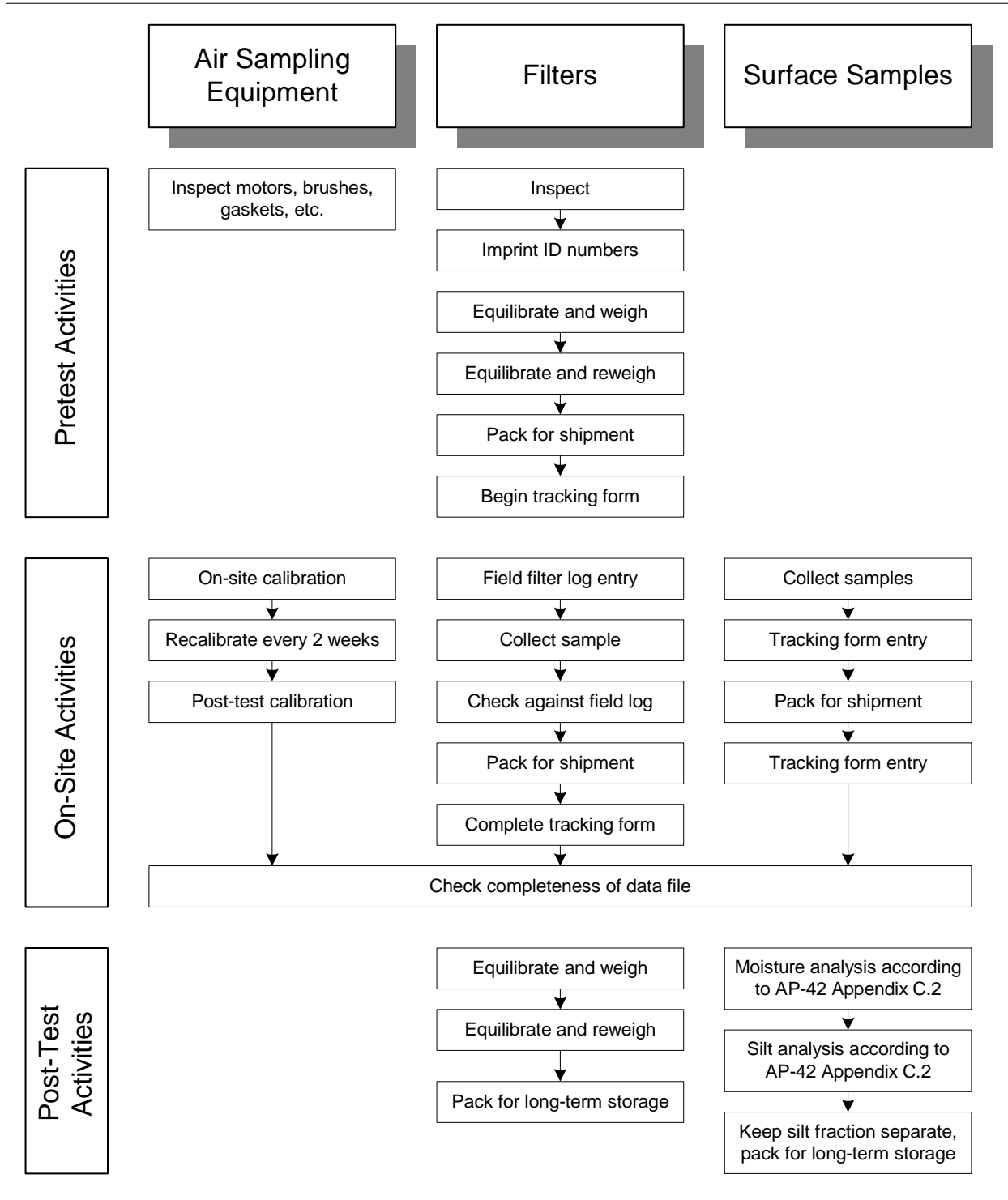
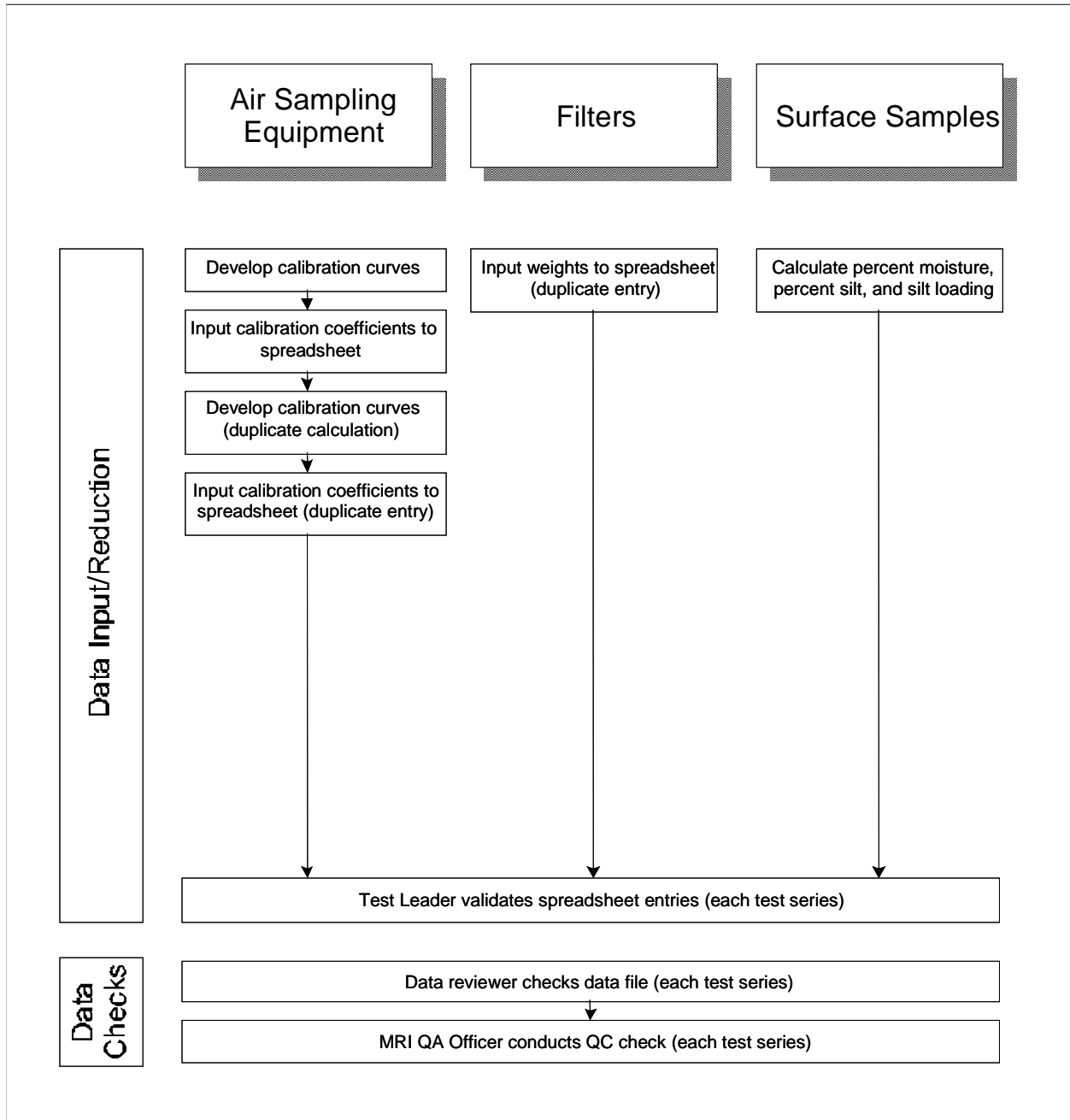


Figure 7. Data collection activities.





**Figure 7. Data collection activities (continued).**

During tests:

1. Communicate with on-site test personnel during first several days of testing to discuss any problems and resolve any issues that will impact data quality.
2. Communicate with RTI QA staff to discuss any QA issues that they have observed that may need resolution.

3. Conduct an independent, on-site assessment of technical systems that are used for the dust suppression tests.

After each test campaign:

1. Review field test documentation.
2. Make an independent determination, based on the mobile sampler quarterly criteria check (and other information), to see if the tests are on track to meet the DQO. The result of this determination will be reported in the quarterly preliminary test report to the APCTVC.
3. Write short report summarizing QA program and assessing QC data.

### B10.1.2 Data Reduction

Section B2 describes the calculations used to determine emission factors. Measurements of dust suppressant CEs are calculated using Equation 8.

$$CE = 100 * \frac{e_{um} - e_{cm}}{e_{um}} \quad \text{Eq. 8}$$

where:

CE = control efficiency, percent,  
 $e_{um}$  = uncontrolled emission value, mg/m, and  
 $e_{cm}$  = controlled emission value, mg/m.

The CE values determined by the above equations represent values for specific time and location conditions. However, because all unpaved road dust suppressants exhibit time-varying control, the CE values will be plotted against time (or cumulative vehicle passes) since the time of initial application of the dust suppressants.

### B10.1.3 Outline of the Test Report

The final test report for the 6-month verification will be outlined as follows.

1. Summary:
  - a. APCTVC manufacturer/distributor information,
2. Summary of test program,
3. Results of the test, and
4. Brief QA statement;
5. Introduction;
6. Description and identification of the dust suppressant products;
7. Procedures and methods used in testing;

8. Statement of operating range over which the test was conducted;
9. Summary and discussion of results;
10. Results,
11. Deviations from test plan and explanations,
12. Discussion of QA and QA statement, and
13. References;
14. Separate Documentation Report;
15. QA/QC activities and results,
16. Raw test data, and
17. Equipment calibration results.

#### **B10.1.4 Draft Report Preparation**

After each quarterly test series, the MRI Data Reviewer will review the data for the test series for completeness and conduct spot checks. A preliminary test report summarizing the data for the test series will be drafted by the MRI Test Leader, and a QA review will be conducted by the MRI Task QA Officer, including the mobile sampler quarterly criteria check to determine if the tests are on track to meet the DQO. These preliminary test reports will be submitted to the RTI Task Leader, EPA, and the manufacturer/distributor for review.

At the conclusion of the field sampling effort, a copy of all electronic and paper data will be made upon return to Kansas City by the MRI Test Leader. The MRI Test Leader will inspect the data for completeness and make a copy of all data to be reviewed by the MRI Data Reviewer. The MRI Data Reviewer will review the data packets for completeness and conduct spot checks for common errors. The common error checks will be based on the Data Reviewer's experience with dust emission testing.

The MRI Test Leader or designated assistant, under the guidance of the Test Leader, will prepare the draft test report following the format presented in Section B10.1.3. After the draft test report is completed by the MRI Test Leader, the report will be first reviewed by the MRI Project Manager and then by the MRI Task QA Officer. Following all reviews by MRI, the draft test report will be transferred to the RTI Task Leader for RTI's and product manufacturer/distributor's reviews. After comments from RTI and the manufacturer/distributor are addressed, the RTI Task Leader (with assistance and review by the MRI Test Leader) will revise the draft report and prepare a draft verification statement and submit them for EPA's review.

After EPA's approval of the report, the verification statement will be signed by an EPA official and transmitted to RTI for the signature of its official. Verification statements containing original signatures will be sent to EPA and the product manufacturer/distributor, and one original will be retained by RTI. The reports will also be posted on the APCTVC and EPA web sites.

**B10.1.5 Long-Term Storage**

All test data, calibration data, certificates of calibration, assessment reports, and test reports will be retained by MRI's APCTVC Program Office for a period of not less than 7 years after the final payment of the assistance agreement as per Part A, Section 5.3 of the EPA ETV QMP<sup>3</sup>.

**B10.2 Data Recording**

Data for this test will be collected electronically and manually. Observations and test run sheets will be recorded manually in lab notebooks and on data forms developed exclusively for this project. The printed output will be secured in the lab notebook.

**B10.3 Data Quality Assurance Checks**

Data QA checks have been discussed in Sections A1.2 and B10.1. Reconciliation with the DQO is discussed in Section D3.

**B10.4 Data Analysis**

The data will be analyzed based on the DQO described in Section A4.1. A value of 12 percent is set, expressed as the half-width interval for the 90 percent confidence limits on CE, for the dust suppression DQO.

**B10.5 Data Storage and Retrieval**

After the completion of a test, labeled three-ring binders containing manually recorded information and data output generated from instrumentation will be stored by MRI's APCTVC Program Office. After the completion of a test, a computer diskette containing spreadsheet data files will be stored by MRI's APCTVC Program Office.

All data and reports will be retained by MRI's APCTVC Program Office for a period of not less than 7 years per Part A, Section 5.3 of the EPA ETV QMP<sup>3</sup>.

## SECTION C: ASSESSMENT/OVERSIGHT

The quality of the project and associated data will be assessed within the project by the project personnel, project manager, and peer reviewers. Management assessment and oversight of the quality for the project activities will be performed through the review of data, memos, audits, and reports by the program and department management and independently by the QA officer.

### C1: Assessments and Response Actions

The effectiveness of implementing the test/QA plan and associated SOPs for a project will be assessed through project reviews, inspections during test data collection, audits, and data quality assessment as described below.

#### C1.1 Project Reviews

The review of project data and the writing of project reports are the responsibility of the MRI Test Leader, who also is responsible for conducting the first complete assessment of the project. Although the project's data will be reviewed by the project personnel and assessed to determine that the data meet the measurement quality objectives, it is the MRI Test Leader who will assure that overall the project activities meet the measurement objectives and DQO. The second review is an independent assessment by a technical peer reviewer. The peer review will be conducted by a technically competent person who is familiar with the technical aspects of the project but not involved in the conduct of project activities. The peer reviewer will present to the MRI Test Leader, MRI QA Task Officer, and project management an accurate and independent appraisal of the technical aspects of the project. The third review of the project is performed by the MRI Project Manager, who is responsible for ensuring that the project's activities adhere to the requirements of the project. The MRI Project Manager's review of the project also will include an assessment of the overall project operations to ensure that the MRI Test Leader has the equipment, personnel, and resources to complete the project as required and to deliver data of known and defensible quality. The final review is that of the MRI Division Director, who is responsible for assuring that the program management systems are established and functioning as required by division procedures and corporate policy. The Division Director is the final MRI reviewer and is responsible for assuring that contractual requirements have been met.

In addition to the MRI reviews, RTI APCTVC and EPA also provide reviews.

#### C1.2 Inspections

Inspections will be conducted by the MRI Test Leader, MRI Project Manager, or MRI Task QA Officer. Inspections assess activities that are considered important or critical to key activities of the project. These critical activities may include, but are not limited to, pre- and post-test calibrations, the data collection equipment, sample equipment preparation, sample analysis, and data reduction. Inspections are assessed with respect to the test/QA plan, SOPs, or other established methods, and are documented in the field records. The results of the inspection are reported to the MRI Test Leader, MRI Project Manager, and MRI Task QA Officer (whomever is

not conducting the inspection). Any deficiencies or problems found during the inspections will be investigated and the results and responses or corrective actions reported in a Corrective Action Report (CAR). This report is discussed later in this section.

### **C1.3 Audits**

Independent systematic checks to determine the quality of the data will be performed on the activities of this project. These checks will consist of self-assessments and independent assessments of technical systems and the quality system and an audit of data quality as described below. These assessments will be conducted according to the procedures that are described in EPA guidance documents for assessments of technical systems and quality systems. In addition, the internal QC measurements will be used to assess the performance of the analytical methodology. The combination of these assessments and the evaluation of the internal QC data allow the assessment of the overall quality of the data for this project.

The MRI Task QA Officer is responsible for ensuring that audits are conducted as required by the test/QA plan. Audit reports that describe problems and deviations from the procedures are prepared and distributed through management. Any problems or deviations need to be corrected. The MRI Test Leader is responsible for evaluating CARs, taking appropriate and timely corrective actions, and informing the MRI Task QA Officer and MRI Project Manager of the action taken. The CAR is initiated by the person finding the problem or deviation. The MRI Task QA Officer is then responsible for ensuring that the corrective action was taken. A summary report of the findings and corrective actions is prepared and distributed to the MRI Project Manager and the RTI Quality Manager.

#### **C1.3.1 Technical System Audit**

The TSA will be conducted by the RTI Quality Manager prior to the start of the project data collection. This audit will evaluate all components of the data gathering and management system to determine if these systems have been properly designed to meet the QA objectives for this study. The TSA includes a careful review of the experimental design, the test plan, and procedures. This review includes personnel qualifications, adequacy and safety of the facilities and equipment, SOPs, and the data management system.

Prior to the TSA, the MRI Task QA Officer may perform a self-assessment of the technical system, following the same pattern of reviews. Final reports of MRI self-assessments and independent assessments, including CARs and followup, will be retained by MRI and will be included in the data packets that are sent to the APCTVC for review.

The TSA begins with the review of study requirements, procedures, and experimental design to ensure that they can meet the DQO for the study. During the TSA, the RTI Quality Manager or designee will inspect the analytical activities and determine they adhere to the SOPs and the test/QA plan. The RTI Quality Manager or a designee reports any area of nonconformance to the MRI Project Manager and management through an audit report. The audit report may contain

corrective action recommendations. If so, follow-up inspections may be required and should be performed to ensure corrective actions are taken.

### **C1.3.2 Performance Evaluation Audit**

A PEA is designed to check the operation of a system that has specific operational parameters. Due to the nature of the task and the type of sampling, the evaluation of performance will be based on verifying that the sampling equipment is operating within the manufacturer's parameters.

The performance of the analytical methods will be assessed using the internal QC requirements as specified in the SOPs for the evaluation.

### **C1.3.3 Audit of Data Quality**

The ADQ is a critical evaluation of the measurement, processing, and evaluation steps to determine if systematic errors have been introduced. During the audit, the MRI Task QA Officer, or a designee, will randomly select at least 10 percent of the data to be followed through the analysis and processing of the data. The purpose of the audit is to verify that the data-handling system is correct and to assess the quality of the data generated.

The ADQ is not an evaluation of the reliability of the data presentation. The review of the data presentation is the responsibility of the MRI Test Leader and the peer reviewer.

### **C1.4 Quality Systems Assessments**

The RTI Quality Manager may conduct an assessment of a quality system, which is a systematic, independent, and documented examination to determine one or more of the following characteristics:

1. Does the organization have a documented and fully implemented quality system?
2. Does the quality system comply with external quality requirements?
3. Do the activities that are being performed by the organization comply with its quality system documentation, particularly in its QMP?
4. Are the quality procedures implemented properly and effectively?
5. Does the quality system support environmental decision making with data that are sufficient in quantity and quality appropriate for their intended purpose?

An assessment is designed to provide objective feedback about the quality system. It evaluates and documents the management policies and procedures that are used to plan, implement, assess, and correct the technical activities that collect or use environmental data. It includes quality system document review, file examination and review, and interviews of managers and staff responsible for environmental data operations.



## **C2: Reports to Management**

During the different activities on this project, the reporting of information to management is critical. To insure the complete transfer of information to all parties involved in this project, a system of reports to management is described below.

### **C2.1 Status and Activity Reports**

The status of the project will be reported to the MRI Test Leader on a regular basis by the project staff. Project status will be reported by the MRI Test Leader to the MRI Project Manager and MRI Task QA Officer at regularly scheduled meetings and monthly by the MRI Project Manager to the RTI Project Manager in the project status report.

Any problems found during the analytical process requiring corrective action will be reported immediately by the project staff to the MRI Test Leader, MRI Project Manager, and the MRI Task QA Officer through the investigation and CAR. The results of the inspection by the MRI Test Leader or Project Manager will be documented in the project files and reported to the MRI Task QA Officer. Inspections conducted by the MRI Task QA Officer will be reported to the MRI Test Leader and Project Manager in the same manner as other audits.

The results of TSAs, inspections, PEAs, and data audits conducted by the MRI Task QA Officer will be written and routed to the MRI Project Manager for review, comments, and corrective action. The results of PEAs will be documented in the project records. The PEAs, issues, and corrective action responses covered by the audit reports will be reviewed and approved by the MRI Test Leader, Project Manager, and Division Director. The results of all assessments, audits, inspections, and corrective actions for the task will be summarized and included in a quality assurance/quality assessment section in the final report.

### **C2.2 Corrective Action Reports**

A corrective action is the process that occurs when the result of an audit or QC measurement is shown to be unsatisfactory or deficient, as defined by the DQO or by the measurement objectives for each task. The corrective action process involves the MRI Test Leader, the MRI Project Manager, and the MRI Task QA Officer. In cases involving the analytical process, the corrective action will also involve the analyst. A written CAR (Figure 8) is required on all corrective actions.

The MRI Test Leader is responsible for and is authorized to implement any procedures to prevent the recurrence of problems.

Project No.: \_\_\_\_\_  
Date: \_\_\_\_\_

**Corrective Action Report**

**Project Title/Description:** \_\_\_\_\_  
\_\_\_\_\_

**Description of Problem:**

Originator: \_\_\_\_\_ Date: \_\_\_\_\_

**Investigation and Results:**

Investigator: \_\_\_\_\_ Date: \_\_\_\_\_

**Corrective Action Taken:**

Originator: \_\_\_\_\_ Date: \_\_\_\_\_

Reviewer/Approval: \_\_\_\_\_ Date: \_\_\_\_\_

cc: Project Leader, Program Manager, Division Manager, QA Unit

**Figure 8. Corrective action report.**

**C2.3 Test and Assessment Reports**

The MRI Test Leader will notify the RTI Project Manager, RTI Task Leader, and RTI Quality Manager when the field test is being conducted. MRI will draft the test reports and submit them

to RTI. The RTI Project Manager will submit the draft test reports to the RTI Quality Manager. After technical assessments, the RTI Quality Manager will submit the assessment report to the RTI Project Manager. The RTI Project Manager will submit test reports to the EPA Project Manager and will submit assessment reports to the EPA Project Manager for informational purposes. Final reports of MRI self-assessments and independent assessments will be retained by MRI and will be included in the data packets that are sent to RTI APCTVC for review.

## SECTION D: DATA VALIDATION AND USABILITY

### D1: Data Review and Validation Requirements

Data review and validation will primarily occur at the following stages:

1. On site following each test run – by the Test Technician,
2. On site following completion of each series of tests in the field – by the MRI Test Leader,
3. After each series of tests - the mobile sampler quarterly criteria check by the MRI Test Leader,
4. After each series of tests - by the MRI Data Reviewer and MRI Task QA Officer,
5. Following the completion of all test runs – mobile sampler DQO check by the MRI Test Leader and MRI Task QA Officer,
6. Before writing the draft test report – by the MRI Data Reviewer, and
7. During QA review of the draft report and ADQ – by the MRI Task QA Officer and MRI Project Manager.

The criteria used to review and validate the data will be the QA/QC criteria specified in each test method (see Table 2) and the DQO analysis of the dust suppression test data (see Section A4.1). Those individuals responsible for on-site data review and validation are noted in Figure 7, Section B10, and above. The MRI Test Leader is responsible for verification of data with all written procedures. Finally the MRI Task QA Officer reviews and validates the data and the draft report using the test/QA plan, test methods, general SOPs, and project-specific SOPs.

The data review and data audit will be conducted in accordance with MRI's SOP 0208 – "Review and Audit of Data and Study Reports." The procedures that will be followed are summarized in Sections C1.3.3 and C2 of this test/QA plan. Form MRI-86 ("blue sheet") will be used for Report review/approval/distribution within MRI. A copy of Form MRI-86 is included as Appendix B.

#### D1.1 Mobile Sampler QC Criteria for Quarterly Test Runs

A preliminary study was conducted at Fort Leonard Wood (FLW), MO from October 2001 to January 2002 using the mobile sampler to measure the CE of dust suppressants.<sup>22</sup> The variance of CE was approximated in terms of the component means, variances, and sample sizes. The means and standard deviations of the replicate measurements were then computed and plotted. These plots clearly showed that standard deviations increased as the (mean) levels increased. It appeared that a relationship of the form  $s=Bx$  between the standard deviation ( $s$ ) and the mean ( $x$ ) would adequately approximate the variance of the measurements [i.e., a model that assumes that the relative standard deviation (RSD)= $s/x$  is constant, and equal to  $B$ ]. The geometric mean of the RSDs was used to estimate  $B$ . Estimates of  $B$  from the preliminary study were 0.163 for  $PM_{10}$ , 0.176 for  $PM_{2.5}$ , and 0.150 for TP.

If the estimated  $B$  is taken to be the true RSD value for the planned study and if a sample size of 5 is used, then the observed RSDs would be expected, with approximately 95 percent confidence,

to fall between 0.35B and 1.67B. (This is based on a chi-square distribution with four degrees of freedom.) This analysis provides an estimate for a quantitative QC criterion for the five replicate measurements that comprise a test run during the quarterly tests. The estimated criterion is to achieve a RSD for a test run of 0.334 or less. This is based on a value of B = 0.2. This value was chosen a little above the values of B obtained in the preliminary study because there are no data to assess how test site conditions from test to test may affect the value of B. The above analysis shows that, with 95 percent confidence, actual RSDs are estimated to be between 0.35B and 1.67B; thus, the criterion is set at less than the upper end (RSD of 0.334). The RSD is calculated using Equation 7 as given in Section B5.

$$RSD = \sqrt{\frac{\sum_{i=1}^5 X_i^2 - 5\bar{X}^2}{4}} / \bar{X} \quad \text{Eq. 7}$$

where:

$X_i$  = ith measurement, and

$\bar{X}$  = mean of 5 measurements.

This value will be calculated by the MRI Test Leader after each quarterly series of tests. Derivation of this quarterly QC criterion is described in Appendix C.

### D1.2 DQO for CE for 6-month Test

Consistent with the approach and assumptions described in Section D1.1, half widths of confidence intervals for 6-month CEs should be approximately 0.707 (or  $\sqrt{1/2}$ ) times as long as those expected for quarterly CEs. This results from assuming that the quarterly CEs are the same for all quarters (simplification of Equation 8 in the GVP). This rationale provides an appropriate approach to defining a DQO for a 6-month CE, since no prior data exist as a basis for such a DQO.

Using the above assumptions, and assuming that the quarterly RSD criteria are met for each set of 5 replicate measurements, that B = 0.2, and that RSD/B = 1.67, a DQO for the 6-month CE measurement was set consistent with the quarterly criteria. The DQO is expressed in percent as the half-width interval for the 90 percent confidence limits. The values vary with CE and are set at (100-CE)/5. For example, as shown in Table 6, the DQO is 1 percent when the CE is 95 percent or 12 percent when the CE is 40 percent.

**Table 6. Half-Widths of 90 Percent Confidence Intervals for 6-month CEs**

CE = 95%	CE = 90%	CE = 80%	CE = 70%	CE = 60%	CE = 50%	CE = 40%
1.4	2.8	5.6	7.8	11	14	17

**D2: Validation Methods**

The process for validating and verifying data has been described in Sections B10.1 and D1. If the test is found to not meet the DQO, the process described in Section A4.1 will be followed. Derivation of the DQO is described in Appendix C.

**D3: Reconciliation with Data Quality Objectives**

The DQO was defined in the GVP, as is the mobile sampler quarterly criteria check. After each test campaign, the MRI Task Leader and the MRI Task QA Officer will determine if the tests are on track to attain the DQO, and if not, what corrective actions are needed. They will report this to the APCTVC.

The DQO reconciliation step is an integral part of the test program and will be done after the field tests. Attainment of the DQO is confirmed by statistically analyzing the test data as described in the GVP. The statistical analysis to determine the DQO will be done by a statistician after the conclusion of all scheduled test runs. The statistical analysis will be done using a statistical analysis tool. The MRI Task QA Officer will reconcile the results of this analysis with the DQO.

The reconciliation process starts with the review of the DQO and the sampling design to assure that the sampling design and data collection documentation are consistent with those needed for the DQO. When the preliminary data are collected, the data will be reviewed to ensure that the data are consistent with what was expected and to identify patterns, relationships, and potential anomalies. The data will be summarized and analyzed using appropriate statistical procedures to identify the key assumptions. The assumptions will be evaluated and verified with all deviations from procedures assessed as to their impact on the data quality and the DQO. Finally, the quality of the data will be assessed in terms as they relate to the measurement objectives and the DQO.

Should the test be conducted and the DQO not be met due to excessive data variability, RTI and MRI will present the data to the product manufacturer/distributor after the last field test day and discuss the relative merit of various options. The two primary options will be either to continue the test to obtain additional data, with resulting increases in cost to all parties, or to terminate the test and report the data obtained. The RTI Project Manager will make the final decision after consultation with MRI and the product manufacturer/distributor.

Results from testing of the dust suppression products will be presented in a report as described in Section B10.1.3.





**Appendix A.**

**Mobile Sampler Operating Procedures**

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### Appendix A. Mobile Sampler Operating Procedures

1. Before the initial use of a truck with the mobile sampler, check the vehicle's speedometer in the following manner
  - a. Lay out a test section at least 150 ft long along a straight, flat section of road.
  - b. Drive the truck over the test section, maintaining a steady "target" speed (25 or 35 mph as indicated by the speedometer) over the test section.
  - c. Make at least 20 passes (10 in each direction).
  - d. Have a second person use a stopwatch to accumulate the total time on the test section for the 20 (or more) passes.
  - e. Calculate the mean measured speed in mph as follows.  
(No. of passes \* Test section length) / (Total time)
  - f. Calculate the ratio of the indicated speed / measured speed. This ratio, when multiplied by a "target speed" provides the speedometer indicated speed for test runs using the subject truck.

Based on the ability to read a speedometer and hold a truck speed steady, this procedure is expected to provide an accuracy for truck speed within  $\pm 10$  percent.

2. With the vehicle parked, load the 8- by 10-in. filter cartridge and 47-mm filter holder onto the mobile sampler.
3. Fit the high-volume cyclone intake with the appropriate nozzle<sup>a</sup>, matched to the target travel speed (25 or 35 mph).
4. Start the vacuum pump and allow it run for at least 1 minute. Record the start time (to the nearest minute, using local time).
5. Set the flow through the URG at 16.7 Lpm using a rotameter. Record the time that the flow rate is set.
6. Start the high-volume sampler and allow it to run at least 1 minute. Record the start time and note the back-plate pressure.
7. Use the on-site calibration results to determine the back-plate pressure that corresponds to 40 cfm.
8. Set the flow through the high-volume sampler by adjusting the autotransformer ("variac") until the back-plate pressure reading is slightly above the pressure determined in Step 6. Recheck the rotameter and reset to 16.7 Lpm, if necessary.
9. Record the pressure reading and turn off the high-volume sampler. Record the stop time.
10. Check all hoses, electrical cords, and mechanical fastenings for the measurement devices prior to starting the vehicle.
11. Driving slowly, position the truck test approximately 150 ft away from the test section. Slowly accelerate to the target travel speed using the speedometer indicated speed calculated in Step 1.

---

<sup>a</sup> Four sizes of nozzles ("A" through "D") are available to maintain isokinecity within  $\pm 20\%$ . The "C" and "D" nozzles provide intake speeds of 26.3 and 35.1 mph, respectively, when the sampler is operated at 40 cfm. Thus, use the "C" nozzle for a target speed of 25 mph and the "D" nozzle for a target of 35 mph.

12. As the truck passes the start of the 500-ft test section, activate the high-volume sampler using the autotransformer (check the red light to ensure that generator circuit breaker has not tripped).
13. As the truck passes the end of the 500-ft test section, deactivate the high-volume sampler using the autotransformer.
14. Slow the truck gently and reposition for another trip over test section (in opposite direction).
15. Repeat Steps 11 through 14 until 6 to 24 passes (depending upon the level of control) have been completed.
16. Stop the truck and briefly reactivate the high-volume sampler to read the back-plate pressure and rotameter reading. Record values and time of readings.
17. Recover the filter cartridge and holder.

**Appendix B**

**Form MRI-86. Report Review/Approval/Distribution**



**Report review/approval/distribution**

**Project No.** (task and subtasks):

Form MRI-86 starts in the technical department office and is circulated with the document through production and review. The completed original form is attached to the Archives copy and sent to the Records Center by the Word Processing Center. The Records Center sends a copy of the completed form to Contracts for deliverable tracking. Form MRI-86 is used for letter reports, monthly reports, interim reports, QA plans, test plans, draft and final reports, and other internally generated project documents. All portions must be filled in. If an information item is not needed, cross it out, initial, and date it. *Documents will not be mailed until this form is properly completed.* THIS FORM SHOULD BE PRINTED ON **BLUE PAPER**.

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## Appendix C

### Mobile Sampler QC Criteria and DQO Derivation

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### Appendix C. Mobile Sampler QC Criteria and DQO Derivation

#### Calculation of Confidence Intervals for Quarterly Control Efficiencies

A preliminary study was conducted at FLW from October 2001 to January 2002 using the mobile sampler to measure the control efficiency of dust suppressants<sup>22</sup>. The calculation of confidence intervals for an efficiency,  $CE_t$ , was accomplished by first deriving an algebraic expression that approximates the variance of  $CE_t$  in terms of the component means, variances, and sample sizes:

$$\begin{aligned}
 \text{Var}[CE_t] &= \text{Var}[1 - \bar{X}_t / \bar{X}_0] = \text{Var}[\bar{X}_t / \bar{X}_0] \\
 &\approx \frac{1}{\bar{X}_0^2} \left[ \text{Var}[\bar{X}_t] + \frac{\bar{X}_t^2}{\bar{X}_0^2} \text{Var}[\bar{X}_0] \right] \\
 &= \frac{\bar{X}_t^2}{\bar{X}_0^2} \left[ (RSD_t^2 / n_t) + (RSD_0^2 / n_0) \right] \\
 &= (1 - CE_t)^2 \left[ (RSD_t^2 / n_t) + (RSD_0^2 / n_0) \right]
 \end{aligned} \tag{C1}$$

where:

$\bar{X}_t$  denotes the mean of  $n_t$  post-treatment observations,

$\bar{X}_0$  denotes the mean of  $n_0$  pre-treatment (baseline) observations, and

$RSD_t$  and  $RSD_0$  denote the relative standard deviations for the post-treatment and baseline observations, respectively.

In the preliminary study, the sample sizes were 2 for the post-treatment observations and 3 for the baseline observations. The above derivation makes use of a Taylor series approximation. The means and standard deviations of the duplicate measurements (and the time 0 triplicates) were then computed and plotted. These plots clearly showed that standard deviations increased as the (mean) levels increased. It appeared that a relationship of the form  $s=Bx$  between the standard deviation ( $s$ ) and the mean  $x$  would adequately approximate the variance of the measurements (i.e., a model that assumes that the  $RSD=s/x$  is constant and equal to  $B$ ). Substitution of this model into the above variance expression for  $CE_t$  leads to

$$\text{Var}[CE_t] \approx (1 - CE_t)^2 \hat{B}^2 \left[ \frac{1}{n_t} + \frac{1}{n_0} \right] \tag{C2}$$

where  $\hat{B}$  is the estimate of  $B$ .

In the previous study, three different estimates of  $B$  were considered: the geometric mean of the relative standard deviations (RSDs), the median of the RSDs, and the mean of the RSDs. For

estimating B, cases in which a duplicate had a zero measurement (either one or both) were not used. The estimate based on the geometric mean was recommended. (It is less sensitive to large RSDs than the third method and can be derived from the least squares estimate for log(B) in the model  $\log(s) = \log(Bx)$ ; this log-scale model has appeal because it should have fairly homogeneous error structure – since standard deviations of standard deviations tend to increase proportionally with their magnitude.) Estimates of B from the prior study at FLW were 0.163 for PM<sub>10</sub>, 0.176 for PM<sub>2.5</sub>, and 0.150 for TP.

Forming a confidence interval for a quarterly CE in future verification tests can be accomplished in two ways. The first way assumes:

1. a model like that used in the prior study (i.e.,  $s=Bx$ ) will be used to produce an estimate of B, and
2. the estimate of B is used, along with the  $CE_t$  value, to produce the estimated variance of  $CE_t$  via equation C2.

Then a 90% confidence interval would be formed via

$$CE_t \pm t_{k,0.95} \sqrt{\text{Var}[CE_t]} \quad (C3)$$

where  $t_{k,0.95}$  is the upper 95<sup>th</sup> percentile of the t distribution with k degrees of freedom. The degrees of freedom, k, can be taken to be equal to the number of RSDs upon which the estimated B is based. Hence, this first approach will be useful only after a substantial amount of testing has been performed. In this context, the subscripts t and 0 in the above equations now represent something different than they did in the preliminary study. In that study, as noted above, the 0 subscript represented a baseline, pre-treatment condition for a given road segment and t represented that same segment after treatment (of a given type); in the planned study, the 0 subscript identifies measurements for an untreated segment at a given time and the t subscript identifies measurements on a similar segment at that same time that was treated with product t.

The second way of forming a confidence interval for a CE does not rely on the variance versus mean model; rather it uses only the data from  $n_t + n_0$  observations used in calculating the  $CE_t$ . In this case, Equation C1 [last part] is used to compute the variance of the control efficiency and the 90% confidence interval is determined as:

$$CE_t \pm t_{K,0.95} \sqrt{\text{Var}[CE_t]} \quad (C4)$$

where  $t_{K,0.95}$  is the upper 95<sup>th</sup> percentile of the t distribution with K degrees of freedom. The degrees of freedom, K, in this case, is determined (after rounding the result down to the nearest integer) by Satterthwaite's formula:

$$K = \frac{\left( RSD_t^2 / n_t + RSD_0^2 / n_0 \right)^2}{\frac{\left( RSD_t^2 / n_t \right)^2}{n_t - 1} + \frac{\left( RSD_0^2 / n_0 \right)^2}{n_0 - 1}} \quad (C5)$$

This approach for forming confidence intervals can be implemented early in the testing. The value of  $K$  will tend to be maximized if the  $RSD$ s and the  $n$ s are the same; in that case,

$$K = n_t + n_0 - 2 .$$

The formation of confidence intervals in either of the above two ways assumes that the estimated quarterly efficiencies are approximately normally distributed. The former way (Equation C3) also relies on the accuracy of the variance-versus-mean relationship. The former way also has the advantage that the estimation of the  $B$  can make use of data from all of the different treatments used in a study. For example, if five products are tested at each of two quarters, there will be 6x2 standard deviations that can be used in the modeling.

### Anticipated Half-Widths of Confidence Intervals for Quarterly Control Efficiencies

The values of  $B$  obtained in the prior study can be used to provide some insight into the expected widths of the confidence intervals. If the estimated  $B$  is taken to be the true  $RSD$  value for the planned study and if a sample size of five is used, then the observed  $RSD$ s would be expected, with approximately 95% confidence, to fall between 0.35 $B$  and 1.67 $B$ . (This is based on a chi-square distribution with four degrees of freedom.)

Table C1 provides half-widths of 90% confidence intervals for  $CE$  generated for four different  $B$  values ranging from 0.15 to 0.30 and for seven different efficiencies ranging from 40% to 95%. Values of the  $RSD$ s appearing in Equation C1 were allowed to take on various multiples of  $B$  – namely, as shown in Table C2.

These were combined with the four choices for  $B$  and the seven efficiency values to produce the estimated half-widths. Equation C1 was used to produce the variance estimate, Equation C5 was used to determine  $K$ , and the half-width was determined as indicated in Equation C4.

### QC Criteria for Quarterly Test Runs

The analysis above provides an estimate for a quantitative QC criteria for the five replicate measurements that comprise a test run during the quarterly tests. The estimated criterion is to achieve a  $RSD$  for a test run of 0.334 or less. This is based on a value of  $B = 0.2$ . This value was chosen a little above the values of  $B$  obtained in the preliminary study because there are no data to assess how test site conditions from test to test may affect the value of  $B$ . The above analysis shows that, with 95% confidence, actual  $RSD$ s are estimated to be between 0.35 $B$  and

**Table C1. Half Widths of Confidence Intervals for CE for Selected Combinations of RSDs and Estimated Efficiencies (%)**

B	Smaller RSD/B	Larger RSD/B	Ratio of RSDs	Smaller RSD	Larger RSD	Half-Widths of 90% Confidence Intervals for CEs						
						CE <sub>i</sub> =95%	CE <sub>i</sub> =90%	CE <sub>i</sub> =80%	CE <sub>i</sub> =70%	CE <sub>i</sub> =60%	CE <sub>i</sub> =50%	CE <sub>i</sub> =40%
0.15	0.35	0.35	1.000	0.053	0.053	0.3	0.6	1.2	1.9	2.5	3.1	3.7
	1.00	1.00	1.000	0.150	0.150	0.9	1.8	3.5	5.3	7.1	8.8	10.6
	1.67	1.67	1.000	0.251	0.251	1.5	2.9	5.9	8.8	11.8	14.7	17.7
	1.00	1.67	1.670	0.150	0.251	1.3	2.5	5.1	7.6	10.1	12.7	15.2
	0.35	1.00	2.857	0.053	0.150	0.8	1.5	3.0	4.5	6.1	7.6	9.1
0.20	0.35	1.67	4.771	0.053	0.251	1.2	2.4	4.9	7.3	9.8	12.2	14.6
	0.35	0.35	1.000	0.070	0.070	0.4	0.8	1.6	2.5	3.3	4.1	4.9
	1.00	1.00	1.000	0.200	0.200	1.2	2.4	4.7	7.1	9.4	11.8	14.1
	1.67	1.67	1.000	0.334	0.334	2.0	3.9	7.9	11.8	15.7	19.6	23.6
	1.00	1.67	1.670	0.200	0.334	1.7	3.4	6.8	10.1	13.5	16.9	20.3
0.25	0.35	1.00	2.857	0.070	0.200	1.0	2.0	4.0	6.1	8.1	10.1	12.1
	0.35	1.67	4.771	0.070	0.334	1.6	3.3	6.5	9.8	13.0	16.3	19.5
	0.35	0.35	1.000	0.088	0.088	0.5	1.0	2.1	3.1	4.1	5.1	6.2
	1.00	1.00	1.000	0.250	0.250	1.5	2.9	5.9	8.8	11.8	14.7	17.6
	1.67	1.67	1.000	0.418	0.418	2.5	4.9	9.8	14.7	19.6	24.6	29.5
0.30	1.00	1.67	1.670	0.250	0.418	2.1	4.2	8.5	12.7	16.9	21.1	25.4
	0.35	1.00	2.857	0.088	0.250	1.3	2.5	5.1	7.6	10.1	12.6	15.2
	0.35	1.67	4.771	0.088	0.418	2.0	4.1	8.1	12.2	16.3	20.3	24.4
	0.35	0.35	1.000	0.105	0.105	0.6	1.2	2.5	3.7	4.9	6.2	7.4
	1.00	1.00	1.000	0.300	0.300	1.8	3.5	7.1	10.6	14.1	17.6	21.2
	1.67	1.67	1.000	0.501	0.501	2.9	5.9	11.8	17.7	23.6	29.5	35.4
	1.00	1.67	1.670	0.300	0.501	2.5	5.1	10.1	15.2	20.3	25.4	30.4
	0.35	1.00	2.857	0.105	0.300	1.5	3.0	6.1	9.1	12.1	15.2	18.2
	0.35	1.67	4.771	0.105	0.501	2.4	4.9	9.8	14.6	19.5	24.4	29.3

**Table C2. RSD Values for Multiples of B**

Selected values of RSDs	Description of RSD Values	Ratio of RSDs	K, determined from eq. C5	t <sub>K,0.95</sub>
0.35B and 0.35B	Both values near lower end of expected range	1.00	8	1.86
0.35B and 1.00B	One value near lower end, one near expected value	2.86	4	2.13
0.35B and 1.67B	One value near lower end, one near upper end of expected range	4.77	4	2.13
1.00B and 1.00B	Both values near expected value	1.00	8	1.86
1.00B and 1.67B	One value near expected value, one near upper end of expected range	1.67	6	1.94
1.67B and 1.67B	Both values near upper end of expected range.	1.00	8	1.86

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1.67B; thus, the criterion is set at less than the upper end (RSD of 0.334). The RSD is calculated (for a given product or uncontrolled segment at a given time) as:

$$RSD = \sqrt{\frac{\sum_{i=1}^5 X_i^2 - 5\bar{X}^2}{4}} / \bar{X} \quad (C6)$$

where:

$X_i$  =  $i^{\text{th}}$  measurement ( $i=1,2,\dots,5$ ) for the given product (or uncontrolled segment), and  
 $\bar{X}$  = mean of five measurements.

### Calculation of Confidence Intervals for 6-Month Control Efficiencies

Assume that the 6-month control efficiency for a given product is estimated as:

$$A_t = \frac{1}{2} \sum_q CE_{tq} = 1 - \frac{1}{2} \sum_q (\bar{X}_{tq} / \bar{X}_{0q}) \quad (C7)$$

where:

the index  $q$  denotes quarters ( $q=1,2$ ),

$CE_{tq}$  is the estimated control efficiency for quarter  $q$  and treatment  $t$ ,

$\bar{X}_{tq}$  denotes the quarterly mean of observations for quarter  $q$  and treatment  $t$ , and

$\bar{X}_{0q}$  denotes the quarterly mean of observations for quarter  $q$  and the untreated segment.

If Equation (C2) is used to estimate the variance of the quarterly control efficiencies, then the variance of the 6-month estimate is given approximately as

$$Var[A_t] \approx \frac{\hat{B}^2}{8n} \left[ \sum_q (1 - CE_{tq})^2 \right] \quad (C8)$$

Equation (C8) assumes the sample size is  $n$  for each quarter and treatment ( $n$  is expected to be five). The degrees of freedom,  $k$ , associated with Equation C8 can be taken to be equal to the number of RSDs upon which the estimated  $B$  is based. Then a 90% confidence interval for a 6-month control efficiency for product  $t$  would be formed as

$$A_t \pm t_{k,0.95} \sqrt{Var[A_t]} \quad (C9)$$

where  $t_{k,0.95}$  is the upper 95<sup>th</sup> percentile of the  $t$  distribution with  $k$  degrees of freedom.



**DQO for 6-Month CE**

Half widths of confidence intervals for 6-month CEs, as determined via equation (C9), should be approximately 70 percent as long as those expected for quarterly CEs (see Table C1). This can be seen by assuming that the CE values that appear in equation C8 are the same for both quarters; simplification of Equation C8 then results in a variance that is  $\frac{1}{2}$  as big as that given by Equation C2; that is, the resultant confidence intervals will be  $\frac{1}{\sqrt{2}}$  as long. This rationale provides an appropriate approach for defining a DQO, since no prior annual data exists as a basis for such a DQO.

Using the above assumptions and assuming that the quarterly RSD criteria are met for each set of five replicate measurements, a DQO for the 6-month CE measurement can be set consistent with the quarterly criteria. The DQO is expressed as the half-width interval for the 90percent confidence limits and is set at  $\frac{1}{\sqrt{2}}$  the value in Table C1 for a B of 0.2 and RSD/B of 1.67. For example, the DQO is 1.4 percent when the CE is 95 percent or 17 percent when the CE is 40 percent.

## **Appendix D**

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