

TECHNICAL GUIDANCE DOCUMENT

**MEASUREMENT OF HEXAVALENT CHROMIUM EMISSIONS FROM  
CHROMIUM PLATING AND CHROMIC ACID ANODIZING OPERATIONS  
FOR CERTIFICATION OF WETTING AGENT CHEMICAL MIST  
SUPPRESSANTS SUBJECT TO SCAQMD RULE 1469**

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SOURCE TEST ENGINEERING BRANCH

MONITORING & ANALYSIS DIVISION

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## ATTACHMENTS

ATTACHMENT I EPA METHOD 306-B - SURFACE TENSION MEASUREMENT  
AND RECORDKEEPING FOR CHROMIUM PLATING TANKS  
USED AT ELECTROPLATING AND ANODIZING  
FACILITIES

ATTACHMENT II FIELD DATA SHEET

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**1.0 OVERVIEW AND BACKGROUND**

On September 19, 2012, the U.S. Environmental Protection Agency (EPA) amended the National Emission Standards for Hazardous Air Pollutant Emissions (NESHAP): Hard and Decorative Chromium Electroplating and Chromium Anodizing Tanks (40 CFR 63 Subpart N). Under the amended NESHAP, if a facility uses a chemical mist suppressant (also commonly referred to chemical fume suppressant) with a wetting agent in a chrome plating or anodizing tank, the required surface tension shall not exceed 40 dynes per centimeter (dynes/cm), as measured with a stalagmometer, or 33 dynes/cm, as measured with a tensiometer. This differs from the SCAQMD Rule 1469 requirement in effect at the time of the NESHAP amendment, as well as the California Air Resources Board (CARB) Airborne Toxics Control Measure (ATCM), which require the mist suppressants to be used at a surface tension not to exceed 45 dynes/cm, as measured with a stalagmometer, or 35 dynes/cm, as measured with a tensiometer. The compliance date for the NESHAP surface tension limits is September 19, 2014.

Unlike the NESHAP, the District Rule and ATCM also require mist suppressant certification. Specifically, Rule 1469(c)(3) requires the owner or operator of a source with any chromium electroplating or anodizing tank using a wetting agent chemical mist suppressant to use only wetting agent chemical mist suppressants

certified pursuant to subdivision (f). Certification involves source testing of representative chrome plating and chromic acid anodizing operations to establish the operating parameters (notably load and surface tension) under which a particular mist suppressant can meet an average emission limit of 0.01 milligrams per ampere-hour (mg/amp-hr) or less. Rule 1469 also requires the Executive Officer to publish and periodically update a list of certified chemical mist suppressants.

In addition to the lower surface tension limits, the NESHAP also prohibits the use of perfluorooctane sulfonic acid (PFOS)-based mist suppressants after September 21, 2015. Currently all mist suppressants certified by SCAQMD and CARB are PFOS-based. The NESHAP does not require a certification of controlled emission rates for mist suppressants, and therefore requires no emissions testing of the non-PFOS based mist suppressants. This Technical Guidance Document establishes the procedure for certification of wetting agent chemical mist suppressants used for compliance with Rule 1469, and the operating parameters under which a mist suppressant is certified. By using a non-PFOS-based certified mist suppressant, facility operators will be able to continue to comply with SCAQMD Rule 1469, as well as the ATCM and NESHAP.

Mist suppressant manufacturers should use an independent laboratory for certification testing which meets the requirements of South Coast Air Quality Management District's Rule 304, Subdivision (k). The source testing contractor

must meet the non-conflict of interest requisite specified per Rule 304(k) and provide a non-conflict of interest statement in the final report. In conducting the test, the laboratory shall prepare a report of findings, including all raw data sheets/charts, and analytical data. Testing must demonstrate to the satisfaction of the Executive Officer that the test data was obtained using the Technical Guidance Document herein.

When equipment at a facility subject to Rule 1469 cannot be tested using this Technical Guidance Document, it may be modified following written approval of the Executive Officer.

Although Rule 1469(c)(14) requires a similar certified mist suppressant emission limit for trivalent chromium ( $\text{Cr}^{+3}$ ) plating baths (0.01 mg total chromium/amp-hr), this document only addresses the certification of mist suppressants in chromic acid ( $\text{Cr}^{+6}$ ) baths. A similar procedure may be used to certify total chromium emissions from trivalent chromium baths, if necessary.



## **2.0 TEST OBJECTIVES**

The objective of the mist suppressant certification testing program is to develop a list of certified mist suppressants for publication, along with the operating parameters approved for use. A separate source test will be required by a facility operator for use of a mist suppressant outside of the range of acceptable operating parameters when the mist suppressant was certified.

The objective of certification testing will be to establish the maximum surface tension, not to exceed 40 dynes/cm (stalagmometer) and 33 dynes/cm (tensiometer), at which a mist suppressant can control hexavalent chromium emissions to a rate of 0.01 mg/ampere-hour or less, given the other operating parameters at the time of the test (e.g. amperage load, chromic acid content, etc.). Once the operating parameters are established for a mist suppressant, each affected facility will have an appropriate requirement to use the mist suppressant at or below the certified maximum surface tension imposed through either an Operating Permit Condition or a Compliance Plan condition.

### **3.0 MIST SUPPRESSANT CERTIFICATION PROCESS AND APPLICABILITY OF RESULTS**

#### **3.1 SUBMITTAL OF PRODUCT INFORMATION AND CERTIFICATION TEST PROTOCOL**

Before certification can occur, a mist suppressant manufacturer, manufacturer's representative, or end user should submit preliminary information on the mist suppressant to be certified. Such information should include the name of the product, material safety data sheet (MSDS) and technical data sheet (TDS), if applicable, and the product chemistry. In addition, the manufacturer, representative or end user should submit a source test protocol in accordance with this Technical Guidance Document. This information should include the facility name and location, date(s) test will occur and other site-specific information, such as tank ventilation, control measures and SCAQMD permit requirements, as required by this Technical Guidance Document.

#### **3.2 APPROVAL OF SOURCE TEST PROTOCOL**

An approved test protocol must be used as the basis for a certification test. A test protocol should be submitted at least 60 days in advance of the intended dates of the certification test. The test protocol will be reviewed and fees charged for the review to the entity submitting the protocol, according to the appropriate fee schedule listed in Rule 304.1.

### **3.3 NOTIFICATION OF CERTIFICATION TESTING DATES**

Prior to mist suppressant certification testing, the manufacturer, representative or end user should notify SCAQMD of the intended dates of the certification test at least three weeks in advance of the test, to allow SCAQMD personnel to witness it.

### **3.4 SUBMITTAL OF SOURCE TEST REPORT**

Within 60 days after completion of the certification test, a test report shall be submitted to SCAQMD. The test report will be reviewed and fees charged for the review to the entity submitting the report, according to the appropriate fee schedule listed in Rule 304.1. The test report should demonstrate compliance with the appropriate elements of the Technical Guidance Document, and with the approved test protocol.

### **3.5 APPLICABILITY OF RESULTS**

Use of source test results from this certification testing shall be applicable to chrome plating and chromic acid anodizing operations that meet all of the following criteria:

1. The surface tension of the bath is kept at a level less than or equal to the tested level; and
2. The plating bath concentration of chromic acid is kept at a level less than or equal to the tested level; and

3. The instantaneous current density applied to the parts is similar to the tested level.
4. Other appropriate criteria as identified during the certification process.

#### **4.0 OPERATING PARAMETERS WHICH MAY AFFECT EMISSIONS**

Prior testing has established that several operating parameters may affect the emissions, including surface tension, chromic acid content, and possibly instantaneous load applied during plating. Each of these parameters is discussed in the following sections.

#### **4.1 SURFACE TENSION**

Of all tank operating parameters, reducing surface tension appears to have one of the largest impacts on emissions. The shape of the surface tension vs. emissions curve has been established through prior testing. It is not necessary to conduct an uncontrolled test, as in prior testing in order to calculate control efficiency. Therefore, testing is required only for the controlled condition. Mist suppressant manufacturers should specify the surface tension (using stalagmometer and tensiometer limits) to be tested, in order to meet the required emission limitation of 0.01 mg/ampere-hour or less.

Empirical data indicates there may be a significant difference between the surface tension measurements conducted using a stalagmometer and tensiometer, in the surface tension range that plating and anodizing processes are normally operated. To date, no reliable correlation has been established to convert readings obtained by one instrument to the other. Recent Rule 1469 testing has shown that both instruments are commonly used by plating shops and analytical laboratories to

determine compliance with surface tension limits. Because of this, two separate standards for surface tensions have been established for stalagmometer and tensiometer. However, source test results measured using a tensiometer will be required to be replicated by a facility for compliance purposes using the same type of device; possibly adding to a regulated facility's cost for compliance. Therefore, if a tensiometer is to be used for measurement of surface tension for the current source tests, it is recommended that a stalagmometer also be used to measure the surface tension for each sample. It is also suggested that multiple measurements of surface tension, using both methods, be made for each sample, to confirm the accuracy of the measurements.

#### **4.2 CHROMIC ACID CONCENTRATION**

According to information provided in October 2002 by the Metal Finishing Association of Southern California (MFASC), chromic acid concentrations can vary from 28 to 40 oz/gallon in decorative chrome plating tanks.

For a new decorative chrome bath, the target concentration is 28 to 30 oz/gallon. As the bath ages and contamination builds up, the concentration may increase in the range of 34 – 40 oz/gallon. Contamination increases very slowly, taking months or years to reach 1 oz/gallon (for iron, etc.) If a porous pot is used to plate out impurities, contamination builds up even slower.

For a hard (functional) chrome plating bath, the normal operating range is 32 to 36 oz/gallon, although it may range from 30 to 42 oz/gallon. Hard chrome plating has the same issue as decorative chrome plating with regard to contamination buildup. On average, hard chrome plating bath concentrations may be a few oz/gallon higher than decorative chrome plating bath concentrations.

Chromic acid anodizing baths may range from 30 to 40 oz/gallon (note: other industry estimates suggest anodizing bath concentrations are much lower, nearer 10 to 12 oz/gallon).

Platers try to maintain +/- 2 oz/gallon from their target concentration. Typically, a data log or traveler accompanies each repeat order, so the plater knows the plating parameters of the previous job, including plating time, voltage, amperage, and chromic acid concentration. The solution is typically analyzed once a week to determine chromic acid concentration and contamination.

Since the goal of this testing program is to establish the upper bound operating parameters a mist suppressant can operate at and still be compliant, it is recommended that mist suppressants be tested at the higher chromic acid concentrations expected in use.

### **4.3 LOAD**

Prior testing showed a possible inverse correlation between emissions and amperage applied to the tank during the test. Tests conducted at very low current draw (<500 amps) resulted in higher controlled emission rates (in mg/ampere-hour) than tests conducted at higher current draw. This possible correlation is empirical rather than theoretical. It is recommended that the aggregate applied load during testing be limited to the mid to lower range expected during use of the product.



## **5.0 ENVIRONMENTAL TEST CONDITIONS**

### **5.1 AMBIENT TEMPERATURE**

The temperature shall be monitored and recorded at fifteen-minute intervals during each test run.

### **5.2 AMBIENT RELATIVE HUMIDITY**

The relative humidity shall be monitored and recorded before and after each test run.

### **5.3 AMBIENT BAROMETRIC PRESSURE**

The barometric pressure shall be monitored and recorded before and after each test run.

## **6.0 TESTING METHODOLOGY**

Testing will be conducted to demonstrate compliance with Rule 1469(f) emission limitation of 0.01 mg Cr<sup>+6</sup>/amp-hr of current applied, at or near the surface tension limits promulgated in the most recent NESHAP. Variables that affect the emission rate include surface tension (dynes/cm), chromic acid content (oz/gallon) and possibly current load (amperes). Air sparging should not be conducted during certification testing. Testing shall be conducted in triplicate runs, resulting in a minimum number of six testing runs to establish the load and surface tension at which a mist suppressant can meet the required emission factor. The minimum number of runs will consist of triplicate sampling at two surface tensions for a plating tank.

### **6.1 NUMBER OF SURFACE TENSION POINTS TO TEST**

Two surface tensions should be tested to determine the slope of the Emission Factor vs. Surface Tension Curve (see Figure 1). Ideally, the higher surface tension will result in an emission rate that exceeds 0.01 milligrams/ampere-hour, and the lower surface tension will result in an emission rate below 0.01 milligrams/ampere-hour. Certification of the maximum allowable surface tension for a given load may then be determined through exponential interpolation.

The default surface tensions, in the absence of manufacturer data, which may give a better estimate of expected results, are as follows: the higher surface tension test should be conducted near, but not exceeding 40 dynes/cm, and the default lower surface tension test should be conducted below 33 dynes/cm.

## **6.2 CURRENT DENSITY**

During testing, plating current density shall be maintained within accepted industry standards for plating the parts typically plated in the tank. The total surface area of the plated parts should be calculated and the calculations provided in the source test report. The current density range for decorative chrome plating listed in Rule 1469 is 50-223 Amp/ft<sup>2</sup>, and for hard chrome plating is 149-604 Amp/ft<sup>2</sup>. To be consistent with the majority of the previously certified products, the current density should be  $100 \pm 10$  Amp/ft<sup>2</sup> for tests conducted on a decorative chrome tank.

If it is expected or observed that a significant difference in emission rates will occur due to changes in current density, then it may be necessary to certify the mist suppressant separately for decorative and hard chrome applications

## **6.3 VENTILATION SYSTEM**

The initial mist suppressant certification testing program conducted in 2003 and 2004 when Rule 1469 emission limits were less stringent and a large number of tanks were not equipped with ventilation and add-on control systems. This required and allowed a temporary hood to be constructed over the tanks to collect chromium emissions at a rate that simulated the atmosphere around an unventilated tank.

The Rule 1469 amendment in 2008 resulted in the majority of chrome plating facilities installing dedicated add-on control systems to meet the lowered hexavalent chromium emission limits. The tank emissions from most of these facilities are now required to be ventilated to control equipment. A temporary hood on a tank with add-on control equipment might block airflow into the required ventilation system and divert tank emissions into the temporary sampling duct. In addition, the collected emissions from the temporary hood would need to be directed back into the ventilation system so they can be properly controlled. In addition, in the case of many bilateral pull-pull systems, the hood would be in a position above the collection slots. The collection slots would continue to draw emissions away from the temporary sampling duct if they were not physically blocked. There are still a small number of tanks operating in the air basin that solely rely on in-tank controls to meet the Rule 1469 emission limit, and are not equipped with add-on control equipment. These tanks would require a temporary enclosure for certification testing.

This creates two scenarios that may be used to collect and sample emissions for certification: dedicated and temporary exhaust systems. Each scenario should be evaluated on a case by case basis to determine that will result in a representative sampling of emissions while maintaining rule and permit condition compliance.

### **6.3.1 DEDICATED EXHAUST SYSTEM**

The dedicated exhaust system can be one of several designs, including, but not limited to bilateral pull and push-pull systems. Rule 1469(c)(7)(A) requires these systems to meet the requirements of American Conference of Governmental Industrial Hygienists' (ACGIH) Industrial Ventilation. Typically, this requires a collection slot velocity of 2000 feet per minute (fpm) and a capture velocity over the tank ranging from 100 to 250 cubic feet per minute per square foot of tank surface area (cfm/ft<sup>2</sup>), depending on design.

For this ventilation setup, sampling would occur at a suitable location on the existing inlet side of the control system. The increased velocity across the tank may collect a small amount of additional emissions in comparison to atmospheric conditions. This small increase is controlled by the air pollution control system, which provides an overall reduction in emissions; however, this might lead to a high bias in the sampling measurement upstream of the control. This method will be considered as a worst-case scenario. Therefore, if the emissions are less than 0.01 mg/amp-hr, the product will be certified, however, if the emissions are above this level, it might not indicate that the product is incapable of being certified using a temporary draft hood.

Another factor that may interfere with representative sampling is existing contamination inside the collection plenum. Since the ports will be located on the inlet side of the control equipment, it is possible that uncontrolled chromium emissions will be deposited on the inner wall of the duct. Care should be taken to avoid disturbing any of the contamination during sampling, especially when the probe is being inserted and removed from the sampling ports.

Capture efficiency for this configuration shall be performed qualitatively with a smoke test, and quantitatively with anemometer and differential pressure measurements. Please refer to Section 7.3 for capture efficiency test procedures.

### **6.3.2 TEMPORARY EXHAUST SYSTEM**

A temporary ventilation system should simulate emissions to the atmosphere of an unventilated tank. This setup may be used on a ventilated tank if the exhaust system is temporarily re-routed. The ventilation system should consist of a cube-shaped hood of the same cross sectional dimensions as the host tank. The hood should be vented to a blower that is set to achieve a specific velocity through the openings at the base of the hood. The hood should be suspended at a height such that air enters the hood from the space between the hood and the tank at the specified velocity. The height of the hood should be sufficiently high to

evenly collect emissions from the entire tank. A straight run of ducting between the hood and the blower should be used to isolate and measure the emissions from the tank. A schematic of a typical system is shown in Figure 2, and a photo of a temporary ventilation system is shown in Figure 3. If the tank is not standalone, and the permit or Rule 1469 requires the exhaust to be vented to the control system, the temporary exhaust shall be vented into the control system. The existing ventilation system shall not interfere with the collection of the temporary enclosure.

Testing has recently been completed on several standalone tanks for Rule 1469. Temporary enclosures were constructed as described, and the inward flow was maintained at 200 feet per minute. This velocity is regarded in EPA Method 204 as the minimum velocity required to establish a Temporary Total Enclosure, in which all of the emissions can be assumed to be collected. Several tanks were tested in this configuration with in-tank controls and determined to meet the Rule 1469 emission limit of 0.0015 mg/amp/hr. Although the configuration does not meet the EPA Method 204 Natural Draft Opening distance requirements, collection efficiency will also be verified qualitatively with a smoke test.

### **6.3.3 OTHER APPROVED METHODS**

An alternative method may be proposed in lieu of the configurations discussed in this document, which will be subject to approval of the

Executive Officer. The alternative test protocol shall provide evidence that the collection of emissions and representativeness of the sampling is equivalent to the methods presented in this document.

#### **6.4 CONTINUOUS VS. INTERMITTANT PLATING DURING TESTING**

Among plating sources, decorative chrome plating operations are most likely to use certified mist suppressants as a means of control. Decorative plating times are very short compared to the sampling time necessary to collect measurable sample. Therefore, a pre-test estimate should be made to determine whether sufficient sample can be collected during the minimum sample time of 120 minutes required by Section 7.2. An emission rate of 0.01 milligrams/ampere-hr should be assumed in the calculation.

Efforts should be made to ensure that a real-world plating operation is simulated as closely as the practical. This includes cycling plating current on and off on a schedule that is representative of parts that would be plated using the subject mist suppressant. However, in order to minimize sampling time, parts (actual or dummy) may be plated continuously for the entire sampling period. New parts should be placed in the tank prior to each of the three tests runs. Parts should be well distributed throughout the tank.



## **6.5 PRE-EXISTING IN-TANK CONTROLS**

In many cases, tanks are permitted and required to have in-tank controls as well as add-on pollution control devices. These in-tank controls can be chemical mist suppressants, or mechanical in nature, such as polyballs. Tanks utilized for mist suppressant certification testing cannot include any additional in-tank controls, other than the mist suppressant to be certified, that may decrease emissions from the tank. Similarly, processes which increase emissions, such as air sparging, should not take place during emissions sampling. Facilities should be aware of their in-tank control requirements prior to submittal of a test protocol, so proper adjustments and modifications can be made prior to testing to maintain compliance with District Rules.

### **6.5.1 PRE-EXISTING MIST SUPPRESSANT**

Any pre-existing chemical mist suppressant shall be sufficiently burned off prior to certification testing. A surface tension of 60-70 dynes/cm shall be considered as having no mist suppressant. The burn off period can often take weeks or months, so advanced planning will be required to set a certification source test date. If it is necessary to burn off mist suppressant, surface tension records shall be taken during the burn off process to show the increasing surface tension trend over time. These records shall be included in the source test protocol or report.

Facilities should verify their mist suppressant requirements so proper adjustments and modifications can be made prior to the removal of the mist suppressant and testing to maintain compliance with District Rules and permit conditions.

## **7.0 TEST PROCEDURES**

The locations of the sampling ports are shown in Figure 2 (for a temporary ventilation system). The required number of traverse points for each will be twelve points on each of two diameters, 90° apart. The locations of the traverse points will be based on SCAQMD Method 1.2. The sampling will be conducted in triplicate during periods of plating activity corresponding to test conditions delineated in this section.

### **7.1 FLOW RATE**

The gas velocity at the sampling ducts should be measured during each run using SCAQMD Method 2.3. The gas velocity determination should be conducted simultaneously with the chromium sampling using a Standard Pitot tube with a differential pressure gauge, and a type "K" thermocouple with temperature readout (see Figure 4). After assembly, the Pitot tube and differential pressure gauge must be checked for leaks before and after each test by introducing a pressure head and blocking the flow at the Pitot tip. The stack's access ports should be located based on the requirements of SCAQMD Method 1.2.

The volumetric flow rate should be calculated using the stack's cross section area and average gas velocity. The flow rate must be corrected to standard conditions by using the stack temperature and pressure along with the barometric pressure measured with a calibrated aneroid barometer. The flow rate must also be

corrected to dry conditions using the moisture content as determined by SCAQMD Method 4.1.

## **7.2 EMISSION SAMPLING**

A hexavalent chromium sample shall be collected during each sampling run using CARB Method 425 or EPA Method 306. The emission samples shall be collected isokinetically from the same sampling locations in the exhaust stack as the velocity measurements. The acceptable isokinetic range is 90% - 110%. The samples shall be collected over a minimum period of 120 minutes for each run. The sampling apparatus must be checked for leaks both before and after sampling by blocking the flow at the probe tip. Before sampling, the leak check must be performed at a minimum vacuum of 20 in. Hg. After sampling, the leak check should be performed at the highest vacuum recorded during the test. The sample impingers must be cooled in an ice bath in order to condense water and other condensable matter present. In addition to hexavalent chromium, the samples should also be analyzed for total chromium.

Recovery and analysis of the samples shall be done in the timeframes stated in the reference methods. The samples shall also be stored at or below the specified temperatures.

### **7.3 CAPTURE EFFICIENCY**

The capture efficiency shall be determined both qualitatively and quantitatively by a smoke test and hot-wire anemometer measurements. Required capture efficiency tests are described for both ventilation configurations discussed in this document. If an alternative configuration is proposed, then it should also include and plan to determine capture efficiency, which will be subject to approval by the Executive Officer. All of these collection parameters and tests and should be verified to meet the applicable rule, permit and Industrial Ventilation standards prior to sampling, and reported in the final certification test report.

#### **7.3.1 CAPTURE EFFICIENCY FOR DEDICATED VENTILATION**

The collection slot dimensions and tank surface dimensions should be measured and the slot velocities measured in feet per minute with a hot wire anemometer (with a suitably sized probe to fit in the collection slots). The exhaust flow rate per square foot of tank surface area should be calculated from this data. If applicable, the push air hole size and distances should be measured, and the push air plenum pressure should be measured (using tubing and a magnehelic gauge, or other acceptable methods). The values measured and calculated, should meet the recommendations in Industrial Ventilation for Open Surface Tank design. In addition, a smoke test shall be performed in accordance with the procedures specified in Rule 1469 Appendix 9.

### **7.3.2 CAPTURE EFFICIENCY FOR TEMPORARY VENTILATION**

A hot wire anemometer should be used to measure the inward airflow between the base of the temporary hood and the top of the tank. The inward velocity shall be at least 200 fpm. The velocity shall be measured at points no further than one foot apart and shall include all corners of the enclosure. A smoke test shall also be performed at all of the same velocity measurement points. The smoke test shall be documented with pictures or video to verify inward air flow into the enclosure. The temporary hood must collect all smoke. In addition, it should be verified that if the tank is equipped with a ventilation system that it does not interfere with the collection of the temporary hood.

### **7.4 SURFACE TENSION MEASUREMENT**

A sample of plating solution should be taken at the beginning of each sampling run to determine surface tension with a well maintained and/or calibrated tensiometer and stalagmometer. If additional mist suppressant is added during the certification test, the surface tension shall be measured and recorded before and after the addition. Surface tension measurement should be conducted with a stalagmometer and tensiometer, in accordance with standards set forth in EPA Method 306-B, *Surface Tension Measurement and Recordkeeping for Chromium*

*Plating Tanks Used at Electroplating and Anodizing Facilities.* This method is contained in Attachment I.

If a tensiometer is used to measure surface tension, such measurement should be conducted in accordance with the standards set forth in ASTM Method D 1331-89, *Standard Test Methods for Surface and Interfacial Tension of Solutions of Surface-Active Agents.*

## **7.5 CHROMIC ACID CONCENTRATION**

Chromic acid concentration during testing should be maintained at a level corresponding to standard practice for the plating type, as discussed in Section 4.2. A sample of plating solution should be taken at the beginning of each sampling run to determine chromic acid concentration. Analysis of chromic acid concentration shall be done by an acceptable test method. The concentration should be reported in the source test report in weight percent.

## **7.6 MEASUREMENT OF OTHER OPERATING PARAMETERS**

During sampling, the following operating parameters should be measured and recorded, and the results reported in the Source Test Report. When appropriate, the parameters should be recorded periodically through the sampling runs.

Plating solution temperature

Totalizer readings (ampere-hours) during test

Sampling period

Average amperage

Plating period within sampling period

Freeboard height

Number of parts and placement in tank

Surface area of parts

Part agitation

Air agitation

Solution recirculation

Part removal and addition

Differential pressures of any downstream air pollution control equipment



## **8.0 QUALITY ASSURANCE / QUALITY CONTROL**

Reliability and accuracy of the test data is assured by using the following work practices:

### **8.1 USE OF STANDARD TEST PROCEDURES**

SCAQMD Method 1.2 must be utilized to determine the sampling point locations. SCAQMD Method 2.3 must be utilized to determine flow rate. CARB Method 425 or EPA Method 306 must be used to determine the emission rate of total and hexavalent chromium.

### **8.2 USE OF TRAINED TEST PERSONNEL**

Because of the complexity of typical source testing methods, the testers should be trained and experienced with the test procedures in order to assure reliable results for this testing program.

### **8.3 KNOWLEDGE OF SOURCE OPERATIONS**

The source testing team should have sufficient knowledge of the process to be tested to document the process parameters during the tests. Without documentation of the process parameters used, results are much less meaningful.

#### **8.4 EQUIPMENT MAINTENANCE AND CALIBRATION**

Use of properly maintained and calibrated test equipment is essential for minimizing systemic errors in results. The totalizer or ammeter, whichever is used to calculate the emission factor, should be calibrated within two months prior to the test. All sampling devices to be used should be constructed, maintained, and calibrated as suggested in EPA documents APTD-0576, and APTD-0581 (these are commonly accepted construction and maintenance manuals for source testing equipment). The dry gas meters should be calibrated with a transfer gas meter with NIST traceability. These calibrations should be included in the Appendices to the final source test report along with those for the nozzles, thermocouples, digital potentiometers, and Pitot tubes.

#### **8.5 THOROUGH RECORDKEEPING**

All data relating to the operation of the sampling train should be immediately recorded to ensure that it is not lost or misinterpreted. Field data sheets similar to those shown in Appendix II should be utilized. Any unusual occurrences in the process operations, unusual test instrument readings, or any other items that could affect the test results should be noted.

#### **8.6 PROPER SAMPLE HANDLING PROCEDURES**

Inaccurate source test results can be caused by delays in retrieving samples, contamination of the samples, insufficient sample identification, tampering, and mishandling of samples. The chances of these errors are greatly increased when

too many people are permitted to handle the samples. For this reason, a chain of custody procedure should be used. The samples should be recovered at the site and kept in secure areas until delivery to the analytical laboratory. The chromium samples should be kept refrigerated until analysis. A sample submittal/chain of custody form should be completed and submitted with the chromium samples to document that each sample analyzed was taken under the conditions reported.

#### **8.7 USE OF THOROUGHLY CLEANED GLASSWARE**

All glassware and probe lines should be cleaned prior to the tests with hot tap water and then with 40% nitric acid solution. The trains should then be cleaned with 0.1 normal sodium hydroxide solution, laboratory grade distilled water, air dried, and sealed until the tests.

#### **8.8 USE OF STANDARDIZED DATA REDUCTION TECHNIQUES**

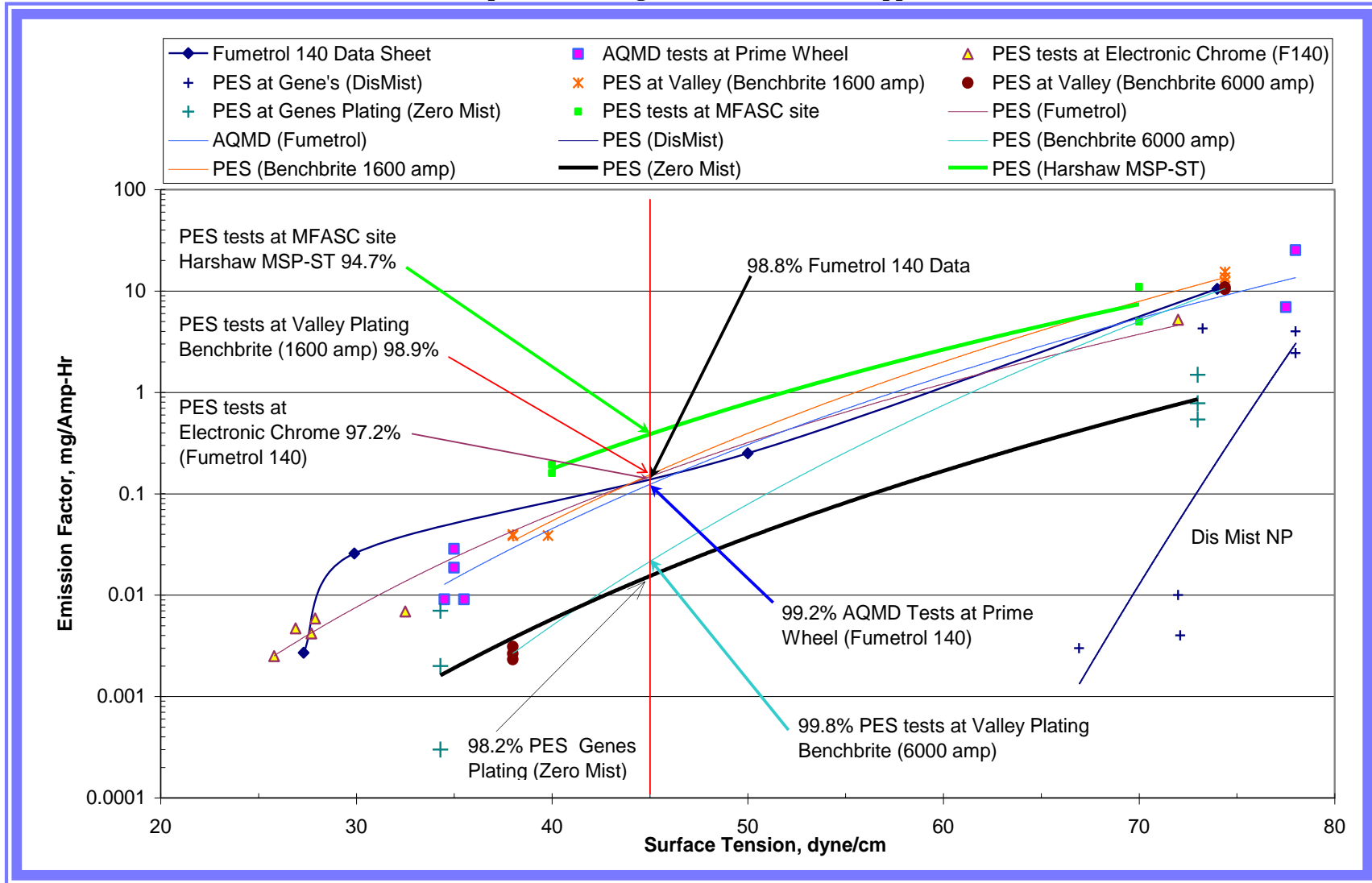
Data reduction should be accomplished by the use of step-by-step calculation sheets. The calculations should be systemic and easy to follow. All calculations for the source test should be included in the final source test report.

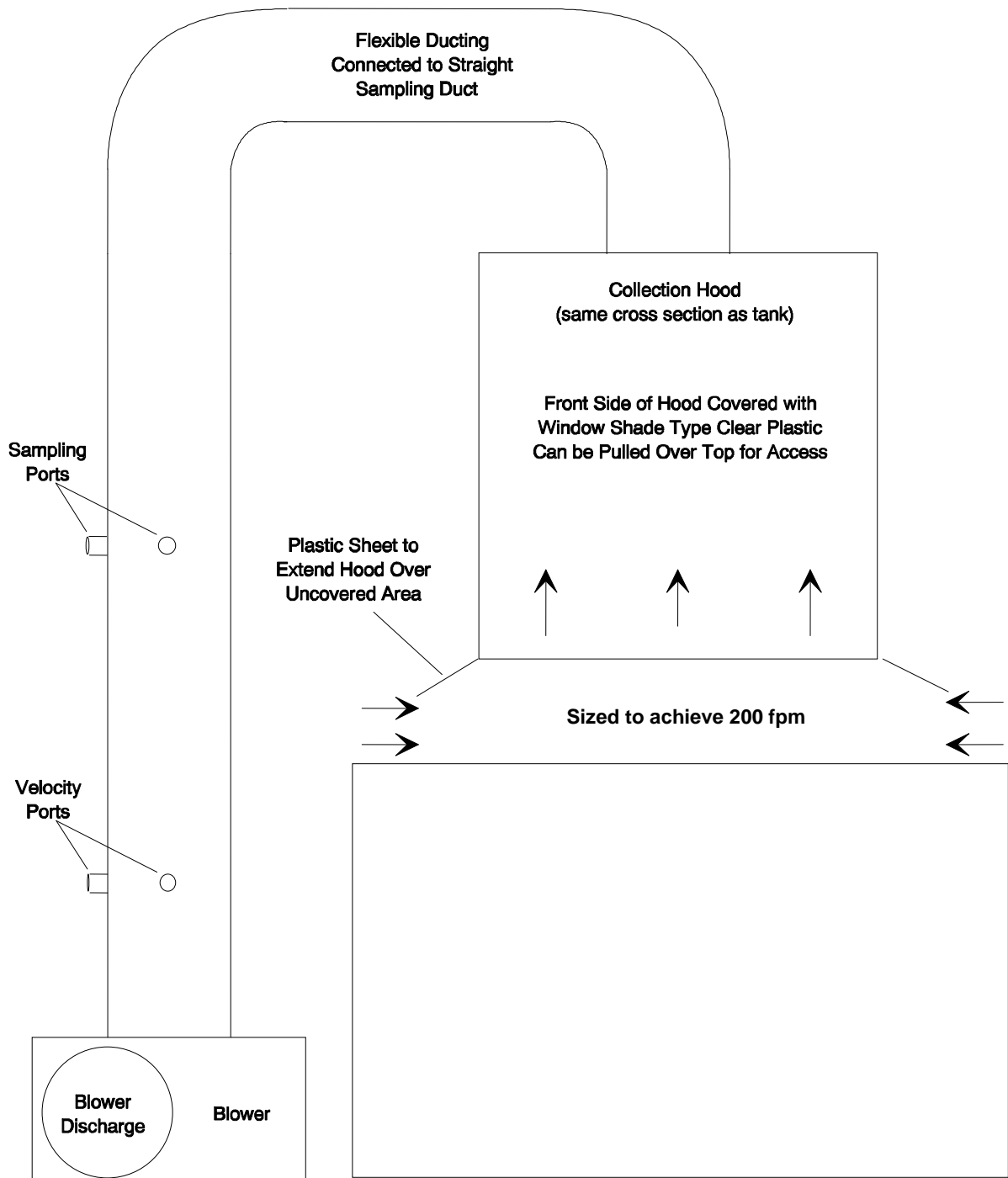
#### **8.9 ANALYTICAL QUALITY ASSURANCE PROCEDURES**

Filter and reagent samples from an unused sampling train carried to the field should be submitted to the laboratory and analyzed with the other samples to detect any possible contamination of sampling media or problems with lab analyses. No corrections should be made to the measured concentrations of the

collected samples, but the blank train results should be reported on the calculation sheets. In addition, one sample in the group submitted should be spiked and reanalyzed to check for matrix effects, and duplicate analysis should be performed on one sample in the group.

**Figure 1 Chromium (Cr+6) Emission Factor vs. Surface Tension**  
 (previous testing of PFOS-based mist suppressants)

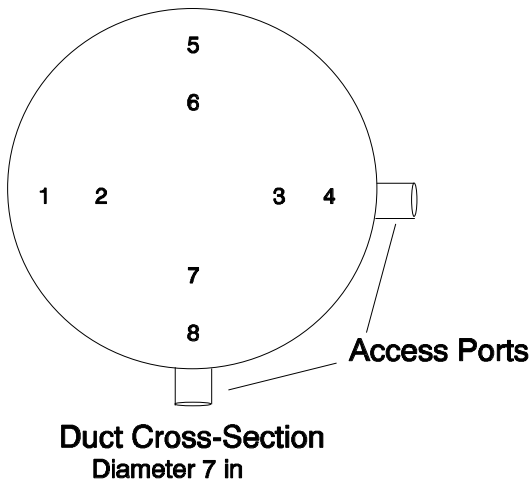
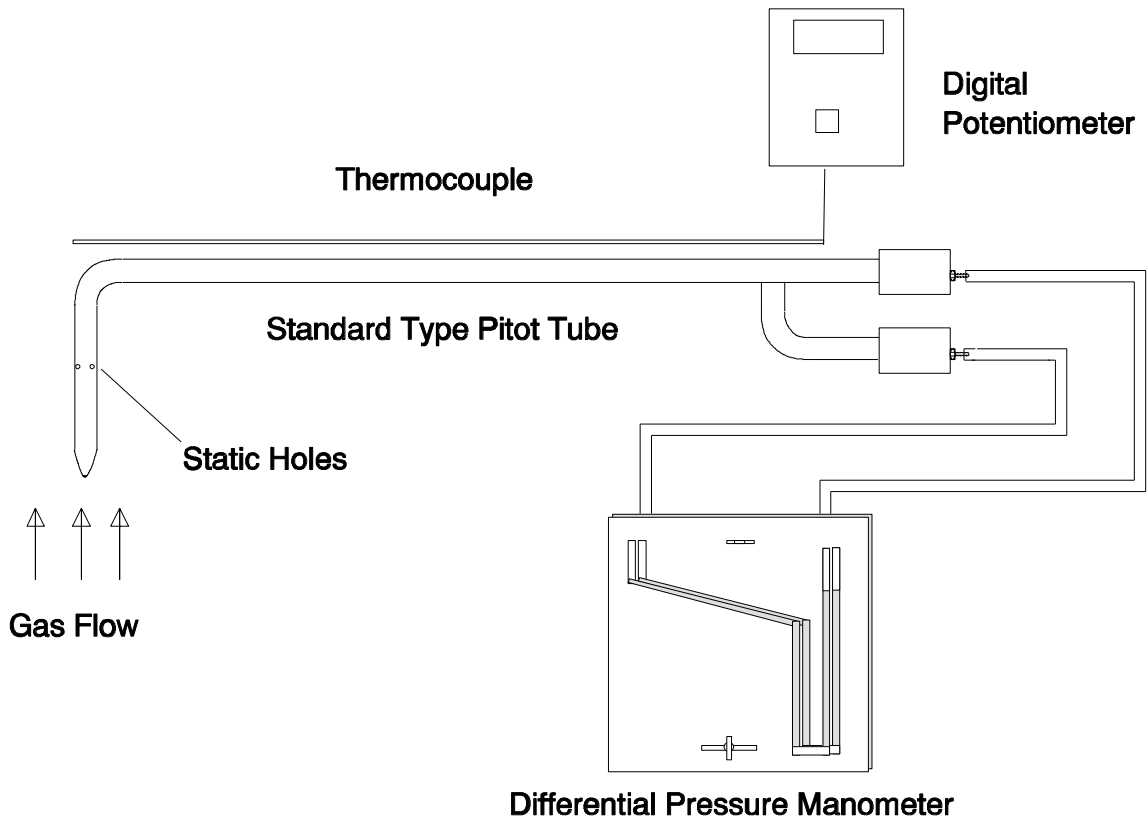




**Figure 2 - Temporary Ventilation System with Sampling Location**



**Figure 3 – Photo of Temporary Ventilation System**



Traverse Point #	Distance from Inner Stack Wall (in)
1, 5	0.5
2, 6	1.75
3, 7	5.25
4, 8	6.5

**Figure 4 - Flow Rate Measuring Apparatus (Example Only)**



**ATTACHMENT I**

**EPA METHOD 306-B - SURFACE TENSION MEASUREMENT AND  
RECORDKEEPING FOR CHROMIUM PLATING TANKS USED AT  
ELECTROPLATING AND ANODIZING FACILITIES**

METHOD 306B - SURFACE TENSION MEASUREMENT FOR TANKS USED AT  
DECORATIVE CHROMIUM ELECTROPLATING AND CHROMIUM ANODIZING  
FACILITIES

NOTE: This method does not include all of the specifications (e.g., equipment and supplies) and procedures (e.g., sampling and analytical) essential to its performance. Some material is incorporated by reference from other methods in 40 CFR Part 60, Appendix A and in this part. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least Methods 5 and 306.

*1.0 Scope and Application.*

1.1 Analyte. Not applicable.

1.2 Applicability. This method is applicable to all decorative chromium plating and chromium anodizing operations, and continuous chromium plating at iron and steel facilities where a wetting agent is used in the tank as the primary mechanism for reducing emissions from the surface of the plating solution.

*2.0 Summary of Method.*

2.1 During an electroplating or anodizing operation, gas bubbles generated during the process rise to the surface of the liquid and burst. Upon bursting, tiny droplets of chromic acid become entrained in ambient air. The addition of a wetting agent to the tank bath

reduces the surface tension of the liquid and diminishes the formation of these droplets.

2.2 This method determines the surface tension of the bath using a stalagmometer or a tensiometer to confirm that there is sufficient wetting agent present.

3.0 *Definitions.* [Reserved]

4.0 *Interferences.* [Reserved]

5.0 *Safety.*

5.1 *Disclaimer.* This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to performing this test method.

6.0 *Equipment and Supplies.*

6.1 *Stalagmometer.* Any commercially available stalagmometer or equivalent surface tension measuring device may be used to measure the surface tension of the plating or anodizing tank liquid.

6.2 *Tensiometer.* A tensiometer may be used to measure the surface tension of the tank liquid provided the

procedures specified in ASTM Method D 1331-89, Standard Test Methods for Surface and Interfacial Tension of Solutions of Surface Active Agents (incorporated by reference -- see §63.14) are followed.

7.0 *Reagents and Standards.* [Reserved]

8.0 *Sample Collection, Sample Recovery, Sample Preservation, Sample Holding Times, Storage, and Transport.*

[Reserved]

9.0 *Quality Control.* [Reserved]

10.0 *Calibration and Standardization.* [Reserved]

11.0 *Analytical Procedure.*

11.1 Procedure. The surface tension of the tank bath may be measured by using a tensiometer, a stalagmometer or any other equivalent surface tension measuring device approved by the Administrator for measuring surface tension in dynes per centimeter. If the tensiometer is used, the procedures specified in ASTM Method D 1331-89 must be followed. If a stalagmometer or other device is used to measure surface tension, the instructions provided with the measuring device must be followed.

11.2 Frequency of Measurements.

11.2.1 Measurements of the bath surface tension are performed using a progressive system which decreases the frequency of surface tension measurements required when the proper surface tension is maintained.

11.2.1.1 Initially, following the compliance date, surface tension measurements must be conducted once every 4 hours of tank operation for the first 40 hours of tank operation.

11.2.1.2 Once there are no exceedances during a period of 40 hours of tank operation, measurements may be conducted once every 8 hours of tank operation.

11.2.1.3 Once there are no exceedances during a second period of 40 consecutive hours of tank operation, measurements may be conducted once every 40 hours of tank operation on an on-going basis, until an exceedance occurs. The maximum time interval for measurements is once every 40 hours of tank operation.

11.2.2 If a measurement of the surface tension of the solution is above the 45 dynes per centimeter limit, or above an alternate surface tension limit established during the performance test, the time interval shall revert back to the original monitoring schedule of once every 4 hours.

A subsequent decrease in frequency would then be allowed according to Section 11.2.1.

*12.0 Data Analysis and Calculations.*

12.1 Log Book of Surface Tension Measurements and Fume Suppressant Additions.

12.1.1 The surface tension of the plating or anodizing tank bath must be measured as specified in Section 11.2.

12.1.2 The measurements must be recorded in the log book. In addition to the record of surface tension measurements, the frequency of fume suppressant maintenance additions and the amount of fume suppressant added during each maintenance addition must be recorded in the log book.

2.1.3 The log book will be readily available for inspection by regulatory personnel.

12.2 Instructions for Apparatus Used in Measuring Surface Tension.

12.2.1 Included with the log book must be a copy of the instructions for the apparatus used for measuring the surface tension of the plating or anodizing bath.

12.2.2 If a tensiometer is used, a copy of ASTM Method D 1331-89 must be included with the log book.

*13.0 Method Performance. [Reserved]*

14.0 *Pollution Prevention.* [Reserved]

15.0 *Waste Management.* [Reserved]

16.0 *References.* [Reserved]

17.0 *Tables, Diagrams, Flowcharts, and Validation Data.*  
[Reserved]

**ATTACHMENT II**

**FIELD DATA SHEET**



