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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 chromium electroplating 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 provided the procedures specified in Section 11.1.2 are followed.

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 using a tensiometer, stalagmometer, or any other equivalent surface tension measuring device for measuring surface tension in dynes per centimeter.

11.1.1 If a tensiometer is used, the procedures specified in ASTM Method D 1331-89 must be followed.

11.1.2 If a stalagmometer is used, the procedures specified in Sections 11.1.2.1 through 11.1.2.3 must be followed.

11.1.2.1 Check the stalagmometer for visual signs of damage. If the stalagmometer appears to be chipped, cracked, or otherwise in disrepair, the instrument shall not be used.

11.1.2.2 Using distilled or deionized water and following the procedures provided by the manufacturer, count the number of drops corresponding to the distilled/deionized water liquid volume between the upper and lower etched marks on the stalagmometer. If the number of drops for the distilled/deionized water is not within ± 1 drop of the number indicated on the instrument, the stalagmometer must be cleaned, using the procedures specified in Section 11.1.3 of this method, before using the instrument to measure the surface tension of the tank liquid.

11.1.2.2.1 If the stalagmometer must be cleaned, as indicated in Section 11.1.2.2, repeat the procedure specified in Section 11.1.2.2 before proceeding.

11.1.2.2.2 If, after cleaning and performing the procedure in Section 11.1.2.2, the number of drops indicated for the distilled/deionized water is not within ± 1 drop of the number indicated on the instrument, either use the number of drops corresponding to the distilled/deionized water volume as the reference number of drops, or replace the instrument.

11.1.2.3 Determine the surface tension of the tank liquid using the procedures specified by the manufacturer of the stalagmometer.

11.1.3 *Stalagmometer cleaning procedures.* The procedures specified in Sections 11.1.3.1 through 11.1.3.10 shall be used for cleaning a stalagmometer, as required by Section 11.1.2.2.

11.1.3.1 Set up the stalagmometer on its stand in a fume hood.

11.1.3.2 Place a clean 150 (mL) beaker underneath the stalagmometer and fill the beaker with reagent grade concentrated nitric acid.

11.1.3.3 Immerse the bottom tip of the stalagmometer (approximately 1 centimeter (0.5 inches)) into the beaker.

11.1.3.4 Squeeze the rubber bulb and pinch at the arrow up (1) position to collapse.

11.1.3.5 Place the bulb end securely on top end of stalagmometer and carefully draw the nitric acid by pinching the arrow up (1) position until the level is above the top etched line.

11.1.3.6 Allow the nitric acid to remain in stalagmometer for 5 minutes, then carefully remove the bulb, allowing the acid to completely drain.

11.1.3.7 Fill a clean 150 mL beaker with distilled or deionized water.

11.1.3.8 Using the rubber bulb per the instructions in Sections 11.1.3.4 and 11.1.3.5, rinse and drain stalagmometer with deionized or distilled water.

11.1.3.9 Fill a clean 150 mL beaker with isopropyl alcohol.

11.1.3.10 Again using the rubber bulb per the instructions in Sections 11.1.3.4 and 11.1.3.5, rinse and drain stalagmometer twice with isopropyl alcohol and allow the stalagmometer to dry completely.

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 40 dynes per centimeter limit when measured using a stalagmometer, above 33 dynes per centimeter when measured using a tensiometer, 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.

12.1.3 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]