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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Guide for Removal of Oily Soils from Metal Surfaces[1]

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1. Scope

1.1 This guide covers the determination of the amount of an oily surface removed from a metal surface.

1.2 This guide employs the use of a fluorescent dye as a tracer to measure the level of residual oil.

1.3 This guide is designed to evaluate metal cleaners designed or developed for the removal of oily and greasy soils from metal surfaces such as stamping presses, metal cans, metal tanks, and other such items.

1.4 This guide employs the use of a generic oily soil. As there is no one universal oily soil, the choice of the soil and substrate used should be agreed upon by the testing laboratory(s) and those using the data to evaluate cleaning performance prior to testing.

1.5 This guide is not intended for use as an evaluation tool for critical areas, which might include surfaces to be painted.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.*

2. Terminology

2.1 *Definitions:*

2.1.1 *soil*—foreign matter present on a metal surface.

2.1.2 *substrate*—any metal-based surface that is to be cleaned.

2.1.3 *water break*—visual rating of a metal surface rinsed with deionized water whereby the rinse water film breaks into beads or runs off exposing the surface, or both, which is indicative of residual soil.

2.1.4 *water break free*—visual rating of a metal surface rinsed with deionized water whereby the rinse water forms a uniform wetted layer covering the surface.

3. Summary of Guide

3.1 An oily based soil is artificially tagged with a fluorescent dye tracer. The soil is artificially applied to a test metal coupon. The soiled coupon is then cleaned in a beaker of wash liquor using a magnetic stir plate and stir bar. Following cleaning, the metal coupon is immediately removed to a beaker of extraction solvent to remove any remaining residual oily soil. The solvent extract is then measured for fluorescent dye absorbance using a spectrophotometer.

4. Significance and Use

4.1 The guide suggests a laboratory guide for use in the development of cleaners designed to remove oily based soils from metal surfaces. This guide can be used to evaluate the removal of numerous oily type soils from a myriad of metal surfaces. This guide should find use in those industries required to clean, among other, metal cans, rolled metals, large industrial production machinery, and blending vessels.

4.2 This guide employs the use of a fluorescent tracer, which will allow for the accurate quantitative measurement of the amount of soil removed and will assist in the ranking of cleaner performance.

4.3 This guide will provide a fast and efficient test protocol for the determination of oily soil removal by a wet cleaning process. This guide will result in a time savings over other methods which traditionally must allow for a drying step prior to weight loss determinations. Further, the guide will quantitate the level of soil removed, thus providing a means of evaluating the cleanliness of a surface which may not be water break free.

5. Apparatus

5.1 *Beakers*, 150 and 250 mL.

5.2 *Volumetric Flasks*, 100 mL.

5.3 *Magnetic Stir Bars*, 1 in. length, and combination hot plate stirrers.

5.4 *Adjustable Wavelength Spectrophotometer*.

5.5 *Graduated Serological Pipets*.

5.6 *Black Light*.

5.7 *Fluorescent Yellow 131SC Dye*.

5.8 *White Mineral Oil*.

5.9 *Propylene Glycol n-Butyl Ether (PnB)*.

5.10 *Metal Coupon*, 1 by 3 in., 16 gage, #14 finish, 316 stainless steel.

6. Preparation of Soil/Substrate Combination

6.1 Clean metal test coupons in an industry specified standard alkaline surfactant based detergent. Scrub the panels to remove residual soils and avoid surface damage. Rinse the panels with appropriate rinse water followed by terminal rinsing with deionized water. Passivate the cleaned and rinsed coupons in a solution of 20 % by weight nitric acid for a

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minimum of 4 h, but not to exceed 24 h. Remove the coupons from the nitric acid solution, rinse in deionized water, and set on clean paper towels to dry. Cover the coupons are with additional paper toweling to provide an appropriate barrier against contamination.

6.2 Tag the oily soil, white mineral oil, or other oil deemed appropriate for specific evaluation, with a fluorescent tracer. Quantitatively add 2 mL of fluorescent dye 131SC into a 100 mL volumetric flask. Fill the flask to mark with mineral oil. This mixture represents a 20 000 ppm solution of tracer dye. The mixture can be homogenized via the inclusion a magnetic stir bar post dilution.

NOTE 1—Due to the opacity of the dye and the potential adhesion of the dye to a pipet wall, it may be necessary to quantitate the drops of dye in 2 mL. Using a 2 mL glass serological pipet, 2 mL approximates 104 drops.

6.3 Using a serological pipet, or other precision dispenser, dispense 0.2 mL of the white oil/dye test soil onto one half of a passivated metal coupon. The coupons should then be elevated by resting the unsoiled end upon glass stir rods in order to prevent soil from migrating across the entire metal surface. Prepare a minimum of three coupons for each trial. The coupons are allowed to age at room temperature for 2 to 24 h.

7. Preparation of a Calibration Curve

7.1 Using a serological pipet or other precision dispenser, dilute 5.0 mL of the stock white oil/dye solution prepared in 5.2 into a 100-mL flask. Dilute to volume with propylene glycol n-butyl ether (PnB) and mix until homogenous. This represents a 1000 ppm solution of dye.

7.2 Add the following volumes of 1000 ppm solution to individual 100 mL volumetric flasks: 0.2, 0.4, 0.6, 0.9, 1.2, 1.6, 2.0, 2.5, 3.0, 3.5, 4.0 mls. Dilute each flask to volume with PnB. These dilutions represent a range of 2-40 ppm dye.

7.3 Prepare spectrophotometer for use. Set a 0 % transmittance with the test cuvette out of the unit. Set the 100 % transmittance with the test cuvette full of neat PnB. For each dilution record the transmittance and absorbance at 535 nm. Construct a calibration curve using Excel or other graphing program. Run a regression to determine the equation of the line of best fit passing through zero.

8. Experimental Cleaning Test Procedure

8.1 Replication is essential for generation of reliable metal cleaning test results. The number of replicate runs required will depend on the soil/substrate combination and the intended use of the results.

8.2 Prepare the wash liquor in a 250 mL beaker by mixing 0.2 g test metal cleaner to 199.8 g water to make 200 g of solution. The type of water or hardness level, or both, is to be selected based on individual performance requirements.

8.3 Add a 1-in. length magnetic stir bar to the wash liquor, and place the beaker onto a combination hot plate stirrer. Carefully adjust the stirrer to obtain a moderate speed such that a vortex just becomes visible at the air/liquor interface. Add one soiled coupon to the beaker and position it so the soiled surface is facing the center of the beaker and the soiled end is resting on the bottom of the beaker. Allow the wash liquor to mix for 2 min.

NOTE 2—The temperature of the wash liquor is room temperature unless otherwise stated in the specific test design.

NOTE 3—The selection and repeat use of one stir bar and one stir plate removes variables attributed to mechanical action. This practice is recommended. The stir bar should be rinsed in PnB and deionized water between replicates.

8.4 Remove the metal coupon from the wash liquor and place it into a 150 mL beaker containing 100 mL of the extraction solvent (PnB). Then place the beaker on the combination hot plate stirrer and slowly agitate via a magnetic stir bar for 4-5 min. Exercise care to avoid touching the oily soil, which may be floating on the surface of the test wash liquor. This will prevent the accidental transfer of removed soil to the extraction solvent. Further, avoid letting additional oily soil drip from the test coupon into the wash liquor during transfer to the extraction solvent.

8.5 The use of a black light will assist in determining if all residual soil has been removed from the test coupon and is contained in the extraction solvent. Shine the light onto the extracted metal coupon. If there is a residual fluorescence, the coupon should be allowed to continue the extraction process in the beaker of PnB solvent.

9. Performance Evaluation

9.1 Set the spectrophotometer to 535 nm wavelength. The instrument is standardized by setting 0.000 absorbance (100 % transmittance) using PnB in the cuvette.

9.2 The PnB solvent extract from the test coupon is placed into the cuvette and the level of absorbance measured.

9.3 Using the calibration curve from 6.3 and the absorbance value of the PnB extract, calculate the level of fluorescent dye that remained on the test coupon. The residual dye is indicative of the level of residual oil on the coupon and can be used to rank the performance of the wash liquor.

9.4 A completely cleaned surface will remain water break free when rinsed with deionized water. If replicate performance trials suggest with wash liquor has completely removed the soil, the test coupon can be removed from the wash liquor and rinsed with deionized water in place of the PnB solvent extract. A water break free surface is indicative of complete cleaning.