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Ti-Pure®

TITANIUM DIOXIDE

GLOSS, 30J AUTOMOTIVE ENAMEL

METHOD: T4400.010.02.WP

TITANIUM DIOXIDE  
(TI-PURE®)

Determination of Alkyd Automotive Gloss (20 degrees)  
(T4400.010.02WP)

I. Principle

Paints are prepared from dry, finished pigment samples and low and high gloss pigment standards. The pigments are dispersed in an alkyd vehicle, sand-milled and reduced with resins and thinners to spray consistency. Aluminum panels are spray painted using automatic spraying equipment under controlled conditions to produce uniform film thickness and surface characteristics. The paint films are then baked. Finally gloss is determined by measuring 20° specular reflectance of the panels and calculating gloss in relationship to the reflectance values of the high and low gloss standards.

II. Applicability

A. General

This method can be used to determine pigment gloss in an alkyd vehicle.

The paint system is solvent borne, melamine-formaldehyde fortified alkyd at 59 wt.% solids, pigmented with 19 vol.% TiO<sub>2</sub> pigment in the paint film (PVC).

**NOTE:** The above formulation is not more precise because of the usual density and solids content ambiguities in the composition of resins and pigments.

III. Limitations

This method can only be used with enamel grade pigment.

IV. Sensitivity, Precision and Accuracy

A. Sensitivity

Data are unavailable.

B. Precision

1. Single Operator

The average analysis ( $\bar{X}$ ), standard deviation (s) and 95% confidence limits (95% CL) established for the single operator precision of the method were as follows:

	$\bar{X}$	s	95% CL
Alkyd Gloss	59.4	1.19	$\pm 2.49$

The above data were calculated from 20 replicate analyses of 1 sample performed by 1 technician over a period of a few days.

2. Multiple Operator

The average analysis ( $\bar{X}$ ), standard deviation (s) and 95% confidence limits (95% CL) established for the multiple operator precision of the method were as follows:

	$\bar{X}$	s	95% CL
Alkyd Gloss	70.9	1.01	$\pm 1.98$

The above data were calculated from 32 replicate analyses of 1 sample performed by 8 technicians over a period of several days.

C. Accuracy

Not applicable. No absolute standards.

V. Special Apparatus (Equivalent apparatus may be substituted)

Hockmeyer Mixer Model 2 HL, available from Hockmeyer Equipment Corp., PO Box 113, Harrison, NJ 07029, (201) 482-0225.

HunterLab Models D-48-7, D-48-8, D-48-D or PRO-3, for reading gloss at 20°, Hunter Associates Laboratory Inc., 11491 Sunset Hills Road, Reston, VA. 22090 703-471-6870, or BYK Multi Gloss Meter, 20° 60° 85°, from BYK-Gardner, 2435 Linden Lane, Silver Springs, MD 20910, 301-589-4747

## VI. Reagents

1. Acetone, Exxon Houston TX., purchase locally, or Lacquer Thinner, DuPont 3602S.
2. Beckosol 12-035 (R), Alkyd type resin, 60% solids content, Reichold Chemicals Inc., 407 South Pace Blvd., Pensacola, FL 32596.
3. Cymel 248-8 (R), Amine type resin, 55% solids content., American Cyanamide Co., Polymer Products Div., PO box 425, Wallingford, CT 06492.
4. Hi-Sol 10 (R) (high flash naptha), Ashland Chemical Inc., PO Box 13305, Memphis, TN 38101
5. n-Butanol, Ashland Chemical Inc., 91 Van Buren St., Nashville, TN 37208. 615-320-5474 (Ashland Chemical Inc., PO Box 371002M, Pittsburgh, PA 152507002)
6. Xylol (xylene), Exxon Houston TX., purchase locally.
7. Ottawa Silica Sand (R), 20-30 mesh, Ottawa Sand Corp., Ottawa. IL, 61350. 800-635-7263.

## VII. Special Safety Considerations

### A. Product Hazards

Refer to the Material Safety Data Sheets (MSDS) on any chemicals with which you are unfamiliar.

No product hazards have been identified with titanium dioxide. However, titanium dioxide is regulated as an air contaminant and care should be exercised to minimize product dusting. Wipe up all spills with a damp paper towel. For more information, refer to MSDS #2816CR.

## VII. Special Safety Considerations (Cont.)

### B. Procedure Hazards

1. The Hockmeyer mill must be locked out any time the glass discs are being handled or cleaned.
2. Leather gloves must be worn when handling the glass discs.
3. Use caution with glass discs.
4. Spray booth exhaust fan must be on while spraying.
5. The oven is  $300^{\circ}\text{F} \pm 10^{\circ}\text{F}$ . Use heat resistant gloves when handling hot rack and samples.
6. The chemicals, acetone, Beckosol 12-035, Cymel 248-8, Hi-sol 10, n-butanol and Xylol are all combustible or flammable; avoid ignition sources.
7. Follow local safety regulation pertaining to handling of flammable solvents, use of moving machinery, and ovens.

## VIII. Procedure

### A. Operating Conditions

Refer to the operating manuals for all equipment used in this test.

### B. Calibration

Calibrate the glossmeter according to operating manual or site Standard Job Procedures.

### C. Sample Analysis

1. Dispenser set up.
  - a. Check the manufacturer's operating manual before starting the equipment.
  - b. Lock out the dispenser.

- c. Install sand grinding discs on the Hockmeyer shaft and tighten carefully. The discs are glass; use care in handling. Do not use any discs which show excessive wear Or are cracked or chipped. A fiber washer must be placed on each side of the disc to avoid glass-to-metal contact. The glass discs should be positioned on the shaft so the bottom surface of the bottom disc is ~1/2 inch above the end of the shaft. The opening between the discs should be ~2 inches.
- d. Position the water-cooled pot containing an empty 1000 mL "Tri-Pour" beaker in the Pot Holder Assembly or Vise; do not tighten pot holder assembly.
- e. Lower sand-grinding disc assembly into the beaker and center the beaker so that the distance between the discs and the beaker wall is uniform on all sides. The Hockmeyer support column shaft should have a stop installed so that at the limit of the downward travel the bottom of the shaft is ~1/2 inch from the bottom of the beaker.
- f. Tighten the pot holding assembly. Once this is set up, there is normally no need to move during a grind series.
- g. Raise sand-grinding disc assembly and remove plastic beaker. Hockmeyer is now ready for use as a sand grinder.

2. Preparation of Grind Vehicle

- a. Grind Vehicle should be made up ahead of time, shook for 10-15 minutes on a paint shaker and allowed to stand before use until all air bubbles are gone.
- b. Grind Vehicle Formula

	<u>1 Grind</u>	<u>1 Gallon (15 grinds)</u>
Beckosol 12-035(R)	149.6 ± 1.0 g	2,244 ± 10.0 g
Xylol	79.0 ± 0.5 g	1,185 ± 10.0 g
n-butanol	<u>9.4 ± 0.1g</u>	<u>141 ± 1.0 g</u>
	238.0 ± 1.0 g	3,570 ± 10.0 g

3. Preparation of Reduction Vehicle

- a. The Reduction Vehicle should be made up ahead of time, shook 10-15 minutes on the paint shaker and allowed to stand before use until all air bubbles are gone.

D. Sample Analysis (Cont.)

b. Reduction Vehicle Formula

	<u>1 Grind</u>	<u>1 Gallon (28 Grinds)</u>
Beckosol 12-035(R)	72.0 ± 0.5 g	2,016 ± 10.0 g
Cymel 248(R)	38.6 ± 0.1 g	1,081 ± 10.0 g
Hi-Sol 10(R)	12.0 ± 0.1 g	336 ± 1.0 g
Xylol	1.4 ± 0.1 g	39 ± 0.1 g
n-butanol	<u>1.0 ± 0.1 g</u>	<u>28 ± 0.1 g</u>
	125.0 ± 1.0 g	3,500 ± 10.0 g

4. Paint Grind

- a. Unlock disperser power supply.
- b. Preweigh 387 ± 1.0 g of pigment standards and samples in a paper bag. The TiO<sub>2</sub> can be preweighed into the beaker containing the grind vehicle as long as it is not premixed any earlier than 8 minutes before grinding.
- c. Preweigh 700 ± 2.0 g of sand (Ottawa 20-30 mesh (R)) in a paper bag for each sample (and standard). Sand must be dry. The sand can be weighed as the samples are grinding.
- d. Preweigh 238 ± 1.0 g of premixed grind vehicle into a 1-liter "Tri-Pour" beaker; one beaker for each sample and standard. The top must be placed on the beaker to prevent solvent loss.
- e. Preweigh 125 ± 1.0 g of premixed reduction vehicle into a 1/2 pint can; one can for each sample and standard. The cap must be placed on the can to prevent solvent loss.
- f. Add enough water to the stainless steel pot to raise the water level between the beaker and the pot (after it is inserted) above the level of the mixture in the "Tri-Pour".
- g. Remove the cover from the beaker containing the grind vehicle and add a preweighed sample (or standard) if not added in Step b above. Mix the pigment in with a spatula no earlier than 8 minutes before grinding.

#### D. Sample Analysis (Cont.)

- h. Place the "Tri-Pour" beaker containing the pigment and the grind base into the water-cooled pot. Beaker should be firmly seated and under the clips on top of the stainless steel pot. Water level in stainless steel pot should be higher than the level inside the "Tri-Pour".
- i. Lower sand-grinding disc assembly into the beaker to the preset position. Turn on cooling water to maximum flow. Be sure to close guards.
- j. Set timer for  $5 \pm 0.25$  minute grind. Turn on sand mill and adjust speed to  $2750 \pm 10$  rpm.
- k. Add  $700 \pm 2.0$  g sand in 20-30 seconds.
- l. At the end of the grind period, raise the disc assembly out of the beaker, shutting off the Hockmeyer as the discs clear the surface of the mill base. This will spin the excess paint from the discs.
- m. Remove the plastic "Tri-Pour" beaker containing the mill base from the water-cooled pot.
- n. Strain the base through a medium or coarse mesh paint filter inside of a fine filter on a ring stand. The strained mill base should be collected in a plastic-lined paper cup or a plastic beaker. Discard the strainers, sand and the "Tri-Pour" beaker.
- o. Weigh  $100 \pm 1.0$  g of the strained mill base into a preweighed 1/2 pint can of reduction vehicle. Mark can with proper identification.
- p. Add the same amount of Xylol to each 1/2 pint can, approximately 25-30 mL, using a 50 mL syringe. The amount of Xylol needed to give the correct viscosity to paints should be checked periodically, as determined by supervision, or when spraying problems are encountered.
- q. Shake the cans for ~10 minutes, off center, on a Paint Shaker. Refer to the manufacturer's operating instructions before using this machine.

#### D. Sample Analysis (Cont.)

- r. To adjust viscosity of paints, add Xylol until 50 mL of paint will pass through a 10F Parlin cup in  $18 \pm 1$  second. Xylol must be added with a glass syringe. A #4 Ford Cup at  $24 \pm 1$  second or a #2 Zahn Cup at  $26 \pm 1$  second may also be used.
- s. Repeat g through r for each standard and sample.
- t. After all paints have been ground, turn off the water, lock the mill out, clean the glass discs with solvent (Xylol) and wipe dry.

#### 5. Spraying Panels

- a. Clean a 4 x 12 inch panel for each standard and sample by dipping the panel in a bath consisting of 2/3 acetone and 1/3 Xylol and drying with a lint-free towel or by wiping with a towel wet with Xylol and drying with a lint-free towel.
- b. Check the spray gun settings.
- c. Turn on the reciprocating panel carrier. The speed should be adjusted to make three passes in 4-7 seconds. The panel holder should have a turn or two of masking tape around each end -- adhesive side out -- to hold the panels in place or a vacuum equipped panel holder can also be used.
- d. Place a clean aluminum panel on the panel carrier after stopping it at the extreme right or left end of its travel. All panels in a series should be sprayed with the panel carrier starting from the same end.
- e. Turn on the spray booth exhaust fan.
- f. Open the air valve on the gun and spray ~1/2 pint or more of solvent through to flush the gun.
- g. Place the paint can in position under the spray gun suction tube so that the end of the fluid tube is 1/2 - 1 inch below the level of the paint and off the bottom of the can when the panel carrier is turned on.

#### D. Sample Analysis (Cont.)

- h. Open air valve. Let spray for 5-10 seconds before turning panel carrier on, make three passes, turn air off, and turn panel carrier off when it has returned to its original position.
- i. Remove the panel from the carrier immediately and examine it. There should be no air bubbles, large lint particles or "fish eyes" present. The surface should have a slight orange peel effect. If it is satisfactory, place it on the baking rack, sprayed side up.
- j. Remove paint can, put cap on and set aside.
- k. Repeat Steps g through j for each paint in the series, flushing the spray gun with the next paint for a minimum of 10 seconds.
- l. The panels should air dry for 25-30 minutes after the last panel is sprayed. Air dry away from strong air currents or drafts or in an air drying cabinet purged with air or nitrogen. Panels should then be baked for  $45 \pm 1.0$  minutes in the oven at  $300 \pm 10^\circ\text{F}$ .
- m. Examine the panels for uniformity of film thickness and orange peel. All panels in a series should be uniform. Any panels with defects that cannot be avoided when the gloss is read must be resprayed along with the standards.
- n. Clean the spray gun by running  $\sim 1/2$  pint or more of solvent through it. Wipe the outside of the feed tube with a towel.

#### 6. Reading Gloss

- a. Check the gloss meter to make sure that it is set on  $20^\circ$  angle and is properly warmed up.
- b. Calibrate the instrument following local site procedures.
- c. Place a baked panel on the gloss meter port. Adjust the hold-down clamp to give a slight pressure on the panel if necessary or use a large, flat weight on the panel.

d. Make at least three readings per panel, one in the center and one near each end. Average the readings and record.

e. Read and record each sample and standard in the series.

#### E. Calculations

1. Correct the raw gloss values using a chart for the current standards or by calculating the value from the following expression:

$$\text{Gloss} = L + \frac{(\text{MX} - \text{ML}) \times (\text{H} - \text{L})}{(\text{MH} - \text{ML})}$$

Where:

L = Assigned Low Standard Value  
H = Assigned High Standard Value  
MX = Measured Sample Value  
ML = Measured Low Standard Value  
MH = Measured High Standard Value  
(MX - ML) may be negative

#### IX. Quality Control

1. Three standards are run with every series of samples. (high, low and reference).
2. The reference standard reading is used for Statistical Process Control.

#### X. Comments

##### A. General

Thorough training and attention to all the details of the procedure are needed to achieve reliable and reproducible results.