Method of Test for

**Determination of Titanium Dioxide in White Waterborne Traffic Paint via X-Ray Diffractometer**

DOTD Designation: TR 523

I. **Scope**
   A. This method of test is designed to determine the amount of titanium dioxide in white waterborne traffic paint by using an X-ray diffractometer.
   
   B. Reference Documents:
      2. ASTM D 2369 – Standard Test Method for Volatile Content of Coatings
      3. ASTM D 3723 – Standard Test Method for Pigment Content of Water-Emulsion Paints by Low-Temperature Ashing
      4. LA DOTD Sampling Procedure S 608

II. **Apparatus**
   A. **X-Ray Diffractometer**, XRD
   B. **Tin sheet**, 2 x 4 inches
   C. **Metal Cutting Shears**
   D. **Paper Towel**
   E. **Personal Nuclear Gage Badge**
   F. **Personal protective equipment** – eye protection and apron for handling paint.
   G. **Waterborne traffic paint worksheet**, Figures 1a and 1b

III. **Health Precautions**
    Wear eye protection and apron while handling paint. Maintain proper ventilation to prevent exposure to solvents when handling waterborne paints.

IV. **Sample**
    Sample white waterborne traffic paint in accordance to LA DOTD Sampling Procedure S 608.

V. **Procedure**
   A. Determine and record the total solids in accordance with ASTM D 2369 on waterborne traffic paint worksheet.
   B. Determine and record the weight of the sample in accordance with ASTM D 1475 on waterborne traffic paint worksheet.
   C. Dip the tin sheet into the paint, collecting a sample about ¾ the length of the tin.
   D. Tap off excess paint and wipe back of tin clean with a paper towel.
   E. Allow specimen to dry for at least 15 minutes.
   F. Ensure the X-ray diffractometer is on and proceed with testing per manufacturer’s instruction.
G. Determine and record the percent pigment (P) in accordance with ASTM D 3723 on waterborne traffic paint worksheet.

VI. Calculations
A. Refer to the rich text format, RTF, report (Figures 2a and 2b), which is generated from the X-Ray diffractometer, and collect the following height data: Silicone Dioxide (SO$_2$), Titanium Dioxide (TiO$_2$), and Calcium Carbonate (CaCO$_3$). Calculate the total height of the peak positions of the compounds above to the nearest 0.01 counts (cts), using the following formula:

$$T_H = H_S + H_T + H_C$$

Where:

- $H_S$ = height of Silicone Dioxide peak, cts
- $H_T$ = height of Titanium Dioxide peak, cts
- $H_C$ = height of Calcium Carbonate peak, cts
- $T_H$ = total height, cts

Note: To identify the height of each component, use the following peak position ranges: Silicone Dioxide (26.2 to 26.8), Titanium Dioxide (27.1 to 27.7), and Calcium Carbonate (29.1 to 29.7).

Example:

- $H_S = 105.44$
- $H_T = 195.23$
- $H_C = 1218.31$

$$T_H = 105.44 + 195.23 + 1218.31$$
$$T_H = 1518.98$$

B. Calculate the percent of Titanium Dioxide, to the nearest 0.01% using the following formula:

$$R = \frac{H_T}{T} \times 100$$

Where:
$H_T = \text{height of Titanium Dioxide peak, cts}$

$T_H = \text{total height, cts}$

$R = \text{percent of Titanium Dioxide}$

$100 = \text{conversion factor for percentage}$

**Example:**

$H_T = 195.23$

$T_H = 1518.98$

$100 = \text{conversion factor for percentage}$

$$R = \frac{195.23}{1518.98} \times 100$$

$$R = 0.1285 \times 100$$

$$R = 12.85\%$$

**C. Calculate the amount of TiO}_2\text{ to the nearest 0.01 lb/gal, using the following formula:**

$$\text{TiO}_2 = \frac{R \times P \times W}{90 \times 100}$$

Where:

$R = \text{percent of Titanium Dioxide}$

$P = \text{percent pigment (refer to ASTM D 3723)}$

$W = \text{weight of paint, per gallon (refer to ASTM D1475)}$

$90 = \text{purity of Titanium Dioxide}$

$100 = \text{conversion factor}$

**Example,**

$R = 12.85$

$P = 61$

$W = 13.7$

$$\text{TiO}_2 = \frac{12.85 \times 61 \times 13.7}{90 \times 100}$$

$$\text{TiO}_2 = \frac{10738.745}{9000}$$

$$\text{TiO}_2 = 1.2\text{lbs/gal}$$
VII. Report
   Record the amount of titanium dioxide to the nearest 0.1 lb/gal on the waterborne traffic paint worksheet, LA DOTD 346.

VIII. Normal Test Reporting Time
   Normal test reporting time is 2 days.
### Traffic Paint Worksheet (Front)

#### Remarks 2

<table>
<thead>
<tr>
<th>Test Results</th>
<th>P/F</th>
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<tbody>
<tr>
<td>XXXXXXXX</td>
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#### Type

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<tr>
<th>Color</th>
<th>P/F</th>
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</thead>
<tbody>
<tr>
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<td>P</td>
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</tbody>
</table>

#### PIGMENT, % [(D-E)+C] x 100 (ASTM D 3723)

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<tr>
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#### Total Solids, % (B+A) x 100 (ASTM D 2369)

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#### Weight:

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#### Per Gallon, LB (F) [(D-E) ÷ 10] (ASTM D 1475)

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<td>P</td>
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#### Per Liter, kg (F x 0.11983)

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#### Viscosity (ASTM D 652)

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<td>85</td>
<td>P</td>
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<tr>
<td>Property</td>
<td>Value</td>
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<tr>
<td>----------------------------------------------</td>
<td>-------</td>
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<tr>
<td>DRYING TIME, MINUTES (ASTM D 711)</td>
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<tr>
<td>DRY THROUGH, MINUTES (ASTM D 1640)</td>
<td></td>
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<tr>
<td>INFRARED SPECTRA (TR 610)</td>
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<tr>
<td>X-RAY DIFFRACTION (TR 523)</td>
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</tr>
<tr>
<td>FLEXIBILITY (Fed. Spec. TT-P-1962)</td>
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<tr>
<td>HIDING POWER @ .260 μm (Fed. Test 141 B)</td>
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<tr>
<td>DRYING TO NO PICKUP, MINUTES (ASTM D 711)</td>
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<tr>
<td>DAYLIGHT REFLECTANCE, %: (Fed. Test 141 B)</td>
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<tr>
<td>WHITE</td>
<td>811</td>
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<td>YELLOW</td>
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<td>FINENESS OF GRIND (ASTM D 1210)</td>
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<td>FREEZE-THAW (ASTM D 2243)</td>
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<td>HEAT STABILITY (Fed. Spec. TT-P-1962)</td>
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<td>VOLATILE ORGANIC COMPOUNDS, g/L</td>
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<td>PIGMENT COMPOSITION:</td>
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<td>WHITE PAINT: (ASTM D 476)</td>
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<tr>
<td>TITANIUM DIOXIDE WEIGHT, Weight, lb/gal (A)</td>
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</tbody>
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Tested by: MC Date: 1/5/10 Checked by: JT Date: 1/8/10

APPROVED by: _____________________________ Date: __________
Figure 2a
X-Ray Diffractometer RTF Report
Figure 2b
X-Ray Diffractometer RTF Report