



NEW YORK STATE DEPARTMENT OF TRANSPORTATION  
MATERIALS BUREAU ALBANY, NY 12232

## TEST METHOD

Method No.: 727-22C

Issue Date: February 2012

Subject Code: 7.42-5

SUBJECT:

**DETERMINATION OF TITANIUM DIOXIDE (TiO<sub>2</sub>) IN PAINT PIGMENT BY WAVE DISPERSIVE X-RAY FLUORESCENCE**

APPROVED:

Gary Fredrick, Acting Director, Materials Bureau

Supersedes: n/a

### 1. SCOPE

- 1.1 This method covers the pigment commercially known as white reflectorized epoxy. The material specified in this standard shall be a two-component (Part A and Part B). The entire pigment composition shall consist of Titanium Dioxide. No Extender pigments are permitted.

A loose powder sample is weighed and placed in a sample cup. The sample is analyzed by WDXRF in a helium environment, and the concentration is measured based upon the wavelength of the spectral line of the desired element.

### 2. REFERENCE DOCUMENTS:

- 2.1 ASTM STANDARDS:  
D476 Type II Test Methods for Titanium Dioxide Pigments.
- 2.2 *ARL OPTIM'X Users Manual*
- 2.3 UniQuant<sup>®</sup> 5 User Manual, chapters 6-7.

### 3. INTERFERENCES AND LIMITATIONS

- 3.1 These procedures were developed primarily for the analysis of pigment commercially known as white reflectorized epoxy. The material specified in this standard shall be a two-component (Part A and Part B). Limitations are noted in the procedure.

### 4. APPARATUS:

- 4.1 Wave Dispersive X-ray Fluorescence Spectrometer: Power supply 50 watts (50 kV max. or 2 mA max), Goniometer with: Fixed collimator (medium angular admittance), Crystal changer with 3 crystals fitted:

- PET, AX06, and LiF200 and 2 detectors fitted: Flow Proportional Counter and Scintillation Counter.
- 4.2 Simultaneous analysis of Magnesium and Sodium with curved AX06 multilayer crystal and sealed detector (no gas required).
  - 4.3 Gas cylinder of Helium (46) with a two-gauge, two stage pressure reducing regulators compatible with the gas required.
  - 4.4 Double Open Ended, 40mm sample cups with rings and caps
  - 4.5 Polypropylene, 6 $\mu$ m X-ray thin film.
  - 4.6 Analytical balance
  - 4.7 Kimwipes 11 x 21 cm

**NOTE1:** The text of this method is most applicable to the use of an ARL OPTIM'X with UniQuant<sup>®</sup>. Laboratories using instruments other than ARL should utilize the method to the fullest extent possible.

## **5. PREPARATION OF SAMPLE**

- 5.1 Remove a representative sample of pigment from sides of glass vial previously prepared by the paint section. This material is used in the determination of % pigment. Center X-ray film over the bottom of double open ended sample cup. Slide external cup ring over film and cup to secure X-ray film in place. Place sample cup, open end up, on a Kimwipe on an analytical balance and record weight. Add enough pigment to sample cup for a minimum depth of 1mm evenly distributed across the X-ray film. Record weight. Secure lid on sample cup. Subtract total weight from sample cup weight. This value will be used in the analysis.
- 5.3 Analyze for Ti on WDXRF using OptiQuant method. Report in SM/LIMS.

## **6. PROCEDURE FOR TIO<sub>2</sub>**

- 6.1 Samples are prepared and analyzed in accordance with ARL OPTIM'X User Manual AA83612, Chapter 4 Samples, Thermo Scientific, 2002 and UniQuant<sup>®</sup> 5 User Manual, chapters 6-7.

## **7. PROBLEMS ENCOUNTERED**

- 7.1 Interferences may arise due to scatter, absorption or other elements; therefore standardization is based upon internal calibration and standards for the WDXRF Instrument. The calibration for Ti is checked

routinely with Ti Standard Sample 222. Calibration discrepancies can be resolved with drift correction which is performed utilizing Setting-Up Samples provided by the manufacturer.

## **8. CALCULATIONS:**

- 8.1 The WDXRF's software program gives a direct reading of the concentrations in percent.