

## Colorado Procedure – Laboratory 2214-15

*Standard Method of Test for*

### Verification of Binder Acidity

#### 1. SCOPE

1.1 This method covers the detection of acid modification of asphalt binders.

#### 2. REFERENCED DOCUMENTS

2.1 FHWA Turner-Fairbanks Research Lab Procedures:

**NOTE 1:** Susan P. Needham Test Method for Detecting the Presence of Phosphoric Acid in Asphalt as last revised on 9/20/2013.

**NOTE 2:** This method is adapted from ASTM test method D 515-88 “Standard Test Methods for Phosphorus in Water.”

#### 3. APPARATUS

- 3.1 Disposable, plastic transfer pipettes.
- 3.2 Glass beakers: 150 ml or 250 ml. Glass stirring rods.
- 3.3 Balance that reads a minimum of 500 g, accurate to 0.1 g.
- 3.4 Distilled or Purified water.
- 3.5 Constant temperature chamber or water bath capable of maintaining the Ascorbic Acid Color Reagent temperature at approximately 4°C.
- 3.6 Antimonyl tartrate / Ammonium molybdate Solution: Dissolve 0.13g of potassium antimonyl tartrate hydrate  $[C_8H_4K_2O_{12}Sb_2 \cdot H_2O]$  in 50 ml of distilled water. Add 5.6g of ammonium molybdate  $[(NH_4)_6Mo_7O_{24} \cdot 4 H_2O]$  and swirl until dissolved.
- 3.7 1N Sulfuric Acid Solution:  $[H_2SO_4]$ . This can be purchased in 1-liter polyethylene bottles.

3.8 Stock Solution Mixture: Mix the solution from Subsection 2.6 and approximately 950ml of solution from Subsection 2.7. This can be done by adding solution from Subsection 2.6 to the 1 liter of 1N sulfuric acid if there is sufficient space in the bottle. The exact amount of sulfuric acid is not critical. This solution is stable for 1 year.

3.9 Ascorbic Acid Color Reagent: Dissolve 0.50 g of L-Ascorbic Acid  $[C_6H_8O_6]$  in 100 ml of the Stock Solution Mixture (see Subsection 2.8). This reagent is stable for a week if stored at 4°C; otherwise, prepare the reagent fresh daily as needed.

3.10 Iso Butanol:  $[(CH_3)_2CHCH_2OH]$  (n-butanol also can be used).

3.11 X-ray fluorescence spectrometer

#### 4. SIGNIFICANCE AND USE

4.1 This method is used to determine if asphalt binder was modified with phosphoric acid. The color change of the wash is an indication of the presence of acid modification.

This method serves as a positive or negative detection of acid in asphalt. This process is not quantitative.

#### 5. PROCEDURE

##### METHOD A – WET CHEMISTRY

5.1 Carefully heat the asphalt sample taking care to prevent local overheating. Heat until the sample is sufficiently fluid to pour. Shake or stir the sample thoroughly before pouring.

5.2 Pour 1 to 2 grams of asphalt sample into glass beakers.

5.3 Add 2 ml of butyl alcohol, while swirling the container.

5.4 Continue to swirl the container and add 2 ml of distilled water.

5.5 While still swirling the container, add 2 ml of the Ascorbic Acid Color Reagent.

5.6 If phosphoric acid is present in the asphalt, a blue color will develop within 5-10 minutes.

## **6. DETERMINATION**

6.1 Determination of Acid Modification.

6.1.1 When the color of the wash is blue in color or tint, the binder is positive for acid modification.

6.2 If the sample is suspected of acid modification further analysis may be performed using X-Ray Fluorescence Spectrometry in Method B.

## **7. PROCEDURE**

### **METHOD B – X-RAY SPECTROSCOPY**

7.1 Procedure is internal to CDOT Chemistry Lab.