
Standard Method of Test for

Analysis of Structural Steel Coatings for Isocyanate Content

AASHTO Designation: TP 67-06 (2007)¹



1. SCOPE

- 1.1. This method is designed for the determination of the isocyanate content of the resin system found in polyurethane paints.
- 1.2. The values stated in SI units are to be regarded as the standard.
- 1.3. *This standard does not purport to address all of the safety concerns associated with its use. It is the responsibility of the user of this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. APPARATUS AND MATERIALS

- 2.1. Analytical balance.
- 2.2. High-speed centrifuge.
- 2.3. Centrifuge tubes, minimum 50-mL heavy-wall glass with vinyl-lined screw caps or 50-mL high-speed (9400 relative centrifugal force (RCF)) polypropylene tubes with screw caps.
- 2.4. 10-mL syringes with Luer slip tip and caps.
- 2.5. Automatic titrator with stirrer (Mettler-Toledo DL 50 or equivalent).
- 2.6. pH electrodes.
- 2.7. Fleaker or other vessel with stopper or cap, 200 mL minimum volume.
- 2.8. o-xylene, reagent grade, dried over 3A molecular sieve (see Section 4.1).
- 2.9. Standard buffer solutions, pH 4.00 and pH 7.00.
- 2.10. Dibutylamine, 99 percent assay.
- 2.11. Methanol, ACS reagent grade, dried over 3A molecular sieve (see Section 4.1).
- 2.12. Hydrochloric acid solution, 1.0 N.

3. PROCEDURE

- 3.1. To analyze a one-component material, the resin system must first be separated from the whole paint.
- 3.2. Place a 45-mL sample of the whole paint into a high-speed centrifuge tube and cap immediately.
- 3.3. Centrifuge the sample to produce a minimum RCF of 4300 until the pigment and resin have separated. See Section 5.1 for RCF equation.
- 3.4. No separation is required for multi-component materials when the resin system is packaged separately. The resin system is generally labeled “curing agent” or “part B.”
- 3.5. Calibrate the titrimeter and pH electrodes with standard buffer pH 7.00 and pH 4.00 solutions.
- 3.6. Place 25 mL of dried o-xylene into an appropriate-sized vessel.
- 3.7. Weigh a 10.0 g sample of resin solution from materials having an expected isocyanate value of less than 15.0 percent or 4.0 g sample of resin solution from materials having an expected isocyanate value of 15.0 percent or greater into the vessel from Section 3.6.
- 3.8. Record the sample weight to the nearest 1.0 mg.
- 3.9. Add 20.00 mL of 1.7 N dibutylamine solution to the vessel from Section 3.6. The 1.7 N dibutylamine solution is prepared by diluting 290 mL of dibutylamine to 1.0 L with dried o-xylene.
- 3.10. Cap the vessel immediately.
- 3.11. Allow the sample to stir for a minimum of 15 minutes but no longer than 60 minutes at $25 \pm 2^\circ\text{C}$.
- 3.12. Prepare a blank in the same manner, omitting the resin solution.
- 3.13. Wash down the sides of each vessel with 100 mL of dried methanol.
- 3.14. Titrate the stirred blank with 1.0 N hydrochloric acid to the end point, which occurs at pH 5.0.
- 3.15. Record the volume of titrant used to the nearest 0.01 mL.
- 3.16. Titrate the stirred sample with 1.0 N hydrochloric acid to the end point, which occurs at pH 5.0.
- 3.17. Record the volume of titrant used to the nearest 0.01 mL.
- 3.18. Prepare and titrate a duplicate sample.
- 3.19. Calculate the weight percent NCO. Duplicate analyses should not differ by more than 0.1 weight percent. See Section 5.2 for weight percent NCO calculation.