
Standard Method of Test for

Analysis of Structural Steel Coatings for Hindered Amine Light Stabilizer (HALS)

AASHTO Designation: TP 66-06 (2007)¹



1. SCOPE

- 1.1. This method is designed to determine the presence of hindered amine light stabilizer (HALS) in the polyol portion of a two-component acrylic urethane coating.
- 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. Gas Chromatograph with Mass Spectrometer Detector.
- 2.2. 5 percent Phenyl Methyl Siloxane Capillary Column.
- 2.3. Reference HALS sample.
- 2.4. Hexane, HPLC grade.
- 2.5. Centrifuge.

3. INSTRUMENT CONDITIONS

- 3.1. Install and condition the column in the chromatograph in accordance with manufacturer's recommendations. Establish operating conditions which yield acceptable separation of the components. See Table 1 as a guide for initial instrument conditions. Values may need to be altered to achieve necessary resolution of components. Allow sufficient time for the instrument to reach equilibrium and to produce a stable base line.

Table 1—Initial Instrument Conditions

Injection Port	
Mode	Splitless
Carrier gas	Helium
Initial temperature	250°C
Pressure	7.6 psi
Total flow	54 mL/min
Purge flow	50 mL/min
Purge time	2 min
Injection volume	1.0 µl
Column Parameters	
Nominal length	30 m
Nominal diameter	0.25 mm
Nominal film thickness	0.25 µm
Maximum temperature	325°C
Mode	Constant flow
Initial flow	1.0 mL/min
Oven Parameters	
Initial temperature	50°C
Maximum temperature	325°C
Equilibration time	0.5 min
Initial time	1.0 min
Temperature ramp 1	25.0°C/min to 250°C
Hold time 1	15.0 min
Temperature ramp 2	50.0°C/min to 300°C
Hold time 2	5.0 min
Mass Spectrometer	
Transfer line heater	280°C
Solvent delay	2.0 min

4. PROCEDURE

- 4.1. Prepare standards by adding reference HALS compounds to hexane to obtain the desired concentrations. Record the weights of hexane and reference HALS used to the nearest 0.1 mg. These standards will be used to obtain retention time data and atomic masses incidental to the specific HALS.
- 4.2. Prepare samples by thoroughly mixing two parts hexane to one part polyol in a centrifuge tube. [Note: 10.0 mL of hexane and 5.0 mL of polyol is a suitable working volume.] Record the weights of hexane and sample polyol used to the nearest 0.1 mg. Cap the centrifuge tube and centrifuge for five minutes at a minimum relative centrifugal force (RCF) of 43. See Section 5.1 for RCF equation. Fill a sample vial with the resulting supernate.