

Photooxidation and Film Loss in Coatings

Method A: Photooxidation and Film Loss of Coatings on Silicon Disks

Application

This test method determines the amount of photooxidation and film loss in coatings by measuring the changes in IR spectra after weathering. Chemical changes occur in coatings during weathering and can be monitored as a function of exposure time. This information provides a way to evaluate the durability of coatings and monitor film loss.

Required Test Equipment

IR Silicon Disks - 2 mm x 13 mm, available from Wilmad Glass Co., Inc., Rte. 40 and Oak Rd., Buena, NJ 08310 (609)697-3000. Catalog #100N9-SP-1 (notched for alignment purposes)

Glass Capillary Tubes - for applying coating on silicon disks

Laboratory Oven - capable of curing coating

Silicon Disk Holder - this will have to be custom made to align disk in IR beam

Fourier Transform Infrared (FTIR) Spectrophotometer

Materials and Solution Required

Methylene Chloride - certified ACS grade or equivalent

I. Preparation of Coating on Silicon Disks

1. For each clearcoat being studied, make free-standing coating films of varying thickness. Obtain an IR spectrum of each film and integrate the CH absorbance area (see Figure 1) to obtain the CH absorbance area/micrometer correlation.
2. Acquire an IR background on a clean, uncoated silicon disk. Observe that the notched disk rests in the proper orientation.
3. Using a capillary tube, coat a silicon disk with a thin uniform film of coating.
4. Allow the solvent to evaporate for several minutes at room temperature.

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Page 1 of 5

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5. Display the IR spectrum of the coating in absorbance mode. Integrate the CH absorbance area. Convert the integrated area to film thickness in micrometers.
 - a. If the film thickness is between 7 and 10 micrometers, cure the coating on the silicon disk using the recommended temperature and time, unless stated otherwise in applicable Engineering Material Specification.
 - b. If the film thickness is not between 7 and 10 micrometers, remove the uncured coating from the silicon disk by rinsing with methylene chloride. Repeat steps 2 – 4 until the film thickness is in the recommended range.
5. Obtain the IR spectrum of the coating before weathering has occurred. This will be called the "time 0" value.

II. Obtaining IR Spectra

1. Remove the silicon disks from weathering at intervals suggested by the ongoing results. Record the hours of exposure upon removal. Exposure samples from Florida should be tested at least at six month intervals, or more often if suggested by the ongoing data.
2. Obtain the IR spectra of the coatings using a clean, uncoated silicon disk as an IR background. (Place the silicon disks back in the weatherometer as soon as possible to continue weathering. Silicon disks from Florida exposure need not be sent back.)

Note: Absorbed water might contribute strongly to the OH, NH area resulting in a false measure of polymer-based OH, NH photooxidation products. Therefore, it is important to remove samples at the end of the dry, light cycle of the weatherometer. Alternatively you can dry samples for 15 minutes at 104 °C.

III. Photooxidation Rate by Monitoring OH, NH Products

One way to access the photooxidation rate of a coating is to monitor the concentration of -OH (hydroxy) and -NH (amine) products as weathering occurs. The concentration is expressed as the amount of -OH and -NH products per amount of film remaining. The concentration is calculated by ratioing the OH, NH area to the CH area of the FTIR spectrum.

1. Integrate the entire OH, NH, CH stretch area. (See Figure 1.)
2. Integrate the CH stretch area centered around 2900 cm^{-1} .
3. Subtract the CH stretch area from the total area to obtain the OH, NH area at time t.
4. Divide the OH, NH stretch area by the CH area to obtain the concentration of hydroxy and amine functional groups present in the coating.