Method of Test for
INFRARED SPECTROPHOTOMETRIC ANALYSIS
DOTD Designation: TR 610M-94

I. Scope

This method determines the infrared spectra of materials when infrared light absorption wavelengths are within the range of 4000 to 650 cm^{-1}. This method is used on a variety of materials such as paint, epoxy resin systems, anti-strip additives, concrete admixtures, thermoplastic, solvents, etc., which can occur as a solid, low volatile liquid or a highly volatile liquid.

II. Apparatus

A. **Infrared spectrophotometer** - having a wavelength range of 4000 to 650 cm^{-1} equipped with a computer for the purpose of monitoring and controlling the functions of the FTIR instrument. A sodium chloride window, a potassium bromide window and closed liquid chambers are integral accessories of the infrared spectrophotometer. A diffuse reflectance accessory, and the Attenuated Total Reflectance (ATR) accessory along with operating manuals are also required.

B. **Amalgamator** - with supply of plastic sample vials and grinding balls.

C. **Analytical balance** - capable of measuring to the nearest 0.0001 g.

D. **Miscellaneous laboratory tools** - tongs, spatula, funnel, syringe, etc.

E. **Mortar and pestle.**

F. **Infrared grade potassium bromide.**

III. Sample Preparation

A. Solid Samples

1. Prepare solid samples by mixing approximately 2 ± 0.1 mg of the sample with approximately 398 ± 2 mg of infrared grade potassium bromide in a vial with the grinding ball. If the sample material is solid bulk, reduce it to a medium fine granular state by using a mortar and pestle.
2. Place the vial with its contents in the amalgamator.
3. Start the amalgamator with the timer switch set to run one minute.
4. When the amalgamator stops, remove the vial and extract the plastic grinding ball.
5. Place the sample and the potassium bromide mixture in the diffuse reflectance sample holder.

B. Liquid Samples

1. Prepare low volatile liquid samples by placing a smear on the ATR window and allowing the solvents to evaporate.
2. Inject highly volatile liquid samples into a closed chamber which has sodium chloride windows on each side.

IV. Procedure

A. Place the prepared sample in the spectrophotometer.

B. Enter the proper parameters on the computer in accordance with the operating manuals.

C. Start the infrared scan as prescribed in the operating manual.

D. Prepare a new sample and produce a new spectrum if the absorption peaks are unsatisfactory.

V. Interpretation of Results

Original spectrums shall be prepared and filed. This test is qualitative and passing is based on a favorable comparison of the infrared spectrum to that of the original sample. A sample is considered rejectionable if its infrared spectrum exhibits significant nonconformity to the spectrum of the original sample, i.e., if

there are different absorption valleys in the two spectra or if an absorption valley in one spectrum is significantly displaced from that in the other.

VI. Report

Mark the appropriate worksheet "pass" or "fail." For qualification samples, mark the appropriate worksheet "complete."

VII. Normal Test Reporting Time

Normal test reporting time is 1 day.