Test Procedure for

OBTAINING THE INFRARED SPECTRUM OF ORGANIC MATERIALS

TxDOT Designation: Tex-888-B

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1. SCOPE

1.1 Use this method to obtain the infrared spectrum of organic materials.

1.2 The values given in parentheses (if provided) are not standard and may not be exact mathematical conversions. Use each system of units separately. Combining values from the two systems may result in nonconformance with the standard.

2. APPARATUS

2.1 *Infrared spectrophotometer,* dual-beam type, capable of recording infrared spectrum from 4000 cm^{-1} to 600 cm^{-1} (such as Perkin-Elmer 783) plus hardware or a Fourier Transform Infrared Spectrometer that generates equivalent spectra, such as Perkin-Elmer FT-IR Spectrometer Spectrum 1000.

2.1.1 *Salt plate blanks,* of materials such as NaCl, KI, CsI, KRS-5, etc., depending on the sample material, its availability, and the wave-number range to be scanned. Plates must fit the infrared instrument used, have parallel faces, and be free of deep scratches and etchings on both faces.

2.1.2 *Mounting block,* for holding salt plates in the beam of the infrared instrument.

2.1.3 *Pellet holder,* for holding pressed pellets in the beam of the infrared instrument.

2.1.4 *Liquid cells,* utilizing various window materials and of varying path lengths.

2.2 *Vacuum oven,* capable of attaining a temperature of 140°C (284°F) and holding a vacuum of at least 711 mm (28 in.) of mercury for three hours.

2.3 *Mechanical vibrator-grinder,* such as Wig-L-Bug Amalgamator (Crescent Dental Manufacturing Co., Chicago, Illinois).

2.4 *Ring press,* with die suitable for producing pressed pellets, such as Research Industrial Instrument Co., Number 0025, 25 Ton.

2.5 *Heat lamp,* 250 watt, infrared, such as Kem Rad's Heat Ray.

2.6 *Polished agate mortar and pestle.*

2.7 *Medicine dropper.*

2.8 *Aluminum cup,* approximately 51 mm (2 in.) diameter by 15 mm (0.6 in.) deep.

2.9 *Various glassware.*

3. MATERIALS

3.1 *Matrix material,* potassium bromide (KBr) is useful down to 400 cm^{-1}; however, other alkali halides including NaCl, KCl, CsBr, and ammonium halides may be used.

3.2 *Selected hydrocarbon solvents,* of reagent grade purity. Acceptable solvents include:

- Methylene chloride
- Ethanol
- Toluene
- Benzene
- Carbon tetrachloride
- Methyl ethyl ketone.

4. PREPARING SAMPLES

4.1 *Cast Film Technique:*

4.1.1 Prepare a solution of the resinous material to be examined at a concentration of approximately 15% by weight of solids in the appropriate solvent.

4.1.1.1 If the material is received as a solution, such as an alkyd resin solution, reduce the concentration to approximately 15% solids with an appropriate solvent.

4.1.1.2 If received at a concentration below 15% solids, use the material as received.

4.1.2 Place two or three drops of the sample solution on one face of an appropriate salt plate, using the medicine dropper.

4.1.3 Roll the salt plate from side to side until the solution is distributed over the entire surface.

4.1.4 Place the salt plate in the aluminum cup.

4.1.5 Place the cup and plate in the vacuum oven at a temperature of 60°C (140°F).

4.1.6 Slowly pull a vacuum on the oven to a minimum of 711 mm (28 in.) mercury. The salt plate must remain in the vacuum oven for 30 minutes.

4.1.7 Remove the salt plate from the oven.

4.1.8 Cool and examine the dried film for transparency, bubbles, or gross surface irregularities.

4.1.8.1 If the film is opaque, prepare another sample using a different solvent or combination of solvents.

4.1.8.2 If the dried film contains bubbles or gross surface irregularities, clean the salt plate and prepare a new film using slightly lower temperature and slower rate of pull of vacuum.

4.2 *Pressed Film Technique:*

4.2.1 Using a medicine dropper, place two to three drops of the material to be examined on one face of an appropriate salt plate.

4.2.2 Use a second salt plate to sandwich the material to be examined between the two plates and press the plates together to obtain a uniform film of material between the plates.

4.2.3 Examine the pressed film for transparency, bubbles, or irregularities. Prepare another sample if observing any film defects.

4.3 *Pressed Pellet Technique:*

4.3.1 Grind the sample, using a mortar and pestle or vibrator-grinder, and add it to the matrix material.

4.3.2 Regrind the mix to sufficient particle size (200 to 400 mesh).

Note 1—If adding a volatile solvent to the mix to aid grinding, dry the sample-matrix mix with an infrared heat lamp.

4.3.3 Transfer the sample-matrix mix to a suitable die and place it in a ring press.

4.3.4 Press a pellet of the sample-matrix mix into the ring press. 300 mg of sample-matrix material mix yields a pellet of satisfactory thickness. A sample-loading ratio of 1 mg sample to 299 mg matrix material is a good reference starting point. If the 1 to 299 mg loading ratio does not give good analytical response, adjust the sample weight until obtaining a satisfactory response.

4.3.5 Examine the pellet for transparency or irregularities. If excess cloudiness or excessive irregularities exist, remake the pellet changing the parameters if necessary

4.4 *Liquid Cell Technique:*

4.4.1 For pure liquid materials, fill a liquid cell with sample material.

4.4.1.1 If the infrared spectrum is too weak, select a cell with a greater path length.

4.4.1.2 If the infrared spectrum is too strong, select a cell having a lesser path length, or, in the case of nonvolatile or slowly volatile liquids, use the pressed film technique.

4.4.1.3 If it becomes necessary to dilute the sample material, use the next method.

4.4.2 For solutions, fill the liquid sample cell with the solution and a matched reference cell with dilution solvent.

4.4.2.1 Adjust the concentration and/or cell thickness to provide a satisfactory infrared spectrum.

5. PROCEDURE

5.1 Turn on the infrared spectrophotometer and make sure that it is operational as per the operations manual.

5.2 Mount the sample by placing the salt plate or plates in a mounting block and the pressed pellet in a pellet holder.

5.3 Place the mounted sample in the sample beam of the infrared spectrophotometer.

5.4 Obtain the infrared spectrum of the sample over the desired wave-number range and examine for compliance with the specification.

5.5 Adjust sample presentation so that major absorption peaks show a maximum transmittance of between 5% and 25%. Vary transmission of major peaks beyond these limits to display peaks of interest effectively.

5.6 Adjust percent transmittance by varying:

- amount of test solution or the percent solids of the test solution placed on the salt plate for the cast film technique
- amount of test solution placed between the salt plates for the pressed film technique
- sample loadings (sample to matrix material ratio) for the pressed pellet technique
- concentration and cell thickness for liquid cell samples.