9.2 Fourier Transform Infrared Spectrometer

9.2.1 Introduction

FTIR can provide some important information related to the chemical composition of the paint being examined. This information can be used in the classification of the paint film and/or a means of finding significant differences in the composition of the unknown as compared to the standard. FTIR technique is non-destructive and samples less than 25 microns can be analyzed with the FTIR Microscope.

9.2.2 Safety Considerations

9.2.2.1

Do not look directly into the laser.

9.2.2.2

If the FTIR Microscope is used, use personal protective equipment to cover eyes and skin while working with liquid nitrogen.

9.2.3 Preparations

- Pellet press and die.
- Compression cell with Diamond windows and salt plates.
- 2 mm KBr and/or NaCl Disks (plates).
- Fine-tip tweezers.
- Fine-pointed needles or probe.
- Scalpel.
- Liquid Nitrogen
- Polystyrene Standard
- Microtome
- Paraplast Embedding Medium
- Low-emissivity microscope slide

9.2.4 Instrumentation

Note: Also see TRACE-PM 7.2 FTIR Verification

9.2.4.1

Stereomicroscope

9.2.4.2

Fourier Transform Infrared Spectrometer (FTIR). A microscope accessory is preferred.

9.2.4.3

FTIR Collection Parameters

9.2.4.3.1

Parameters must be adjusted to provide sufficient intensity and resolution for spectra comparison or classification.

9.2.4.3.2

The use of the microscope will require:

- Change in scan speed.
- Use of external mirror.
- Use of MCT Detector.
- Increase number of scans.
- Proper adjustment of apertures with the same setting for sample and background collection.

9.2.5 Procedure or Analysis

9.2.5.1

Samples must be prepared so the sample thickness is sufficient for transmittance of the beam through the sample with minimal saturation as determined by spectral analysis.

9.2.5.2

Samples may be prepared by:

9.2.5.2.1

Use a scalpel to remove individual paint films or slice a thick paint film.

9.2.5.2.2

Use a glass slide as a guide and cut a thin cross section from a paint chip.

9.2.5.2.3

Compress the paint film between two salt plates in a compression cell or diamond windows in the compression cell.

9.2.5.2.4

Place sample in a die and produce a KBr mull in a press.

9.2.5.2.5

Casting of the sample in embedding medium followed by the use a microtome for cross sectioning.

9.2.5.3

Place sample on a salt plate or flatten with a roller knife on low-e glass. It may or may not be retained in the compression cell during analysis.

9.2.5.4

Set instrument parameters.

9.2.5.5

Place sample on microscope stage and adjust apertures to area of interest.

9.2.5.6

Run sample *

9.2.5.7

Move off sample and run background. * The sequence of steps 9.2.5.6 and 9.2.5.7 is instrument dependent.

9.2.5.8

See Classification Appendices and other available known reference spectra for classification. Additional literature may be consulted.

9.2.5.8.1

Refer to Appendix 9.2 A: Automotive Paint Binder Infrared Classification Flowchart

9.2.5.8.2

Refer to Appendix 9.2 B: Polymer Classification Scheme

9.2.5.8.3

Refer to Appendix 9.2 C: Binder Classification Flowchart

9.2.5.8.4

(Appendix 9.2.D: IR of Paint Worksheet - No longer being used)

9.2.5.8.5

Refer to Appendix 9.2 E: Diagnostic IR Peaks of Common Binders/Resins Used in Automotive Paints

9.2.5.8.6

Refer to Appendix 9.2 F: Diagnostic IR Peaks of Common Pigments and Extenders Used in Automotive Paints

9.2.5.9 KBr Pellets

Paints may also be run and compared using the standard pellet method.

9.2.5.9.1

Dissolve paint in suitable solvent.

9.2.5.9.2
Mix with KBr.

9.2.5.9.3
Press into a pellet.

9.2.5.9.4
Run blank and sample by FTIR.

9.2.5.10
Samples may also be run in reflectance mode. Sample preparation for paint run in this mode should be appropriate for the specific reflectance holder device (diamond cell, ATR objective, split pea, etc.). Spectra obtained in absorbance mode should only be compared to spectra run and prepared in the same manner.

9.2.5.11
Documentation must meet standards and controls requirements.

9.2.6 References

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