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# Standard Guide for Using Infrared Spectroscopy in Forensic Paint Examinations<sup>1</sup>

This standard is issued under the fixed designation E2937; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

#### INTRODUCTION

Infrared (IR) spectroscopy is commonly used by forensic laboratories for the analysis of paints and coatings received in the form of small chips, residues, particles, or smears, and serves as a staple comparative technique in the assessment of whether or not questioned paint could have come from a particular source. IR spectroscopy provides molecular structure information on many of the organic and inorganic constituents contained within a single paint layer. This information can be used to classify both binders and pigments in coating materials. The classification information maycan then be <a href="https://doi.org/10.1001/nc.100

# 1. Scope

- iTeh Standards
- 1.1 This guide applies to the forensic IR analysis of paints and coatings and is intended to supplement information presented in the Forensic Paint Analysis and Comparison Guidelines (1)<sup>2</sup> written by Scientific Working Group on Materials Analysis (SWGMAT). This guideline is limited to the discussion of Fourier Transform Infrared (FTIR) instruments and provides information on FTIR instrument setup, performance assessment, sample preparation, analysis and data interpretation. It is intended to provide an understanding of the requirements, benefits, limitations and proper use of IR accessories and sampling methods available for use by forensic paint examiners. The following accessory techniques will be discussed: FTIR microspectroscopy (transmission and reflectance), diamond cell and attenuated total reflectance. The particular methods employed by each examiner or laboratory, or both, are dependent upon available equipment, examiner training, specimen size or suitability, and purpose of examination. This guideline does not cover the theoretical aspects of many of the topics presented. These can be found in texts such as *An Infrared Spectroscopy Atlas for the Coatings Industry* (Federation of Societies for Coatings, 1991) (2) and *Fourier Transform Infrared Spectrometry* (Griffiths and de Haseth, 1986) (3).
  - 1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.
- 1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety safety, health, and health environmental practices and determine the applicability of regulatory limitations prior to use.
- 1.4 This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

# 2. Referenced Documents

2.1 ASTM Standards:<sup>3</sup>

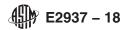
D16 Terminology for Paint, Related Coatings, Materials, and Applications

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 $<sup>\</sup>overline{{}^2}$  The boldface numbers in parentheses refer to the list of references at the end of this standard.

<sup>&</sup>lt;sup>3</sup> For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.



- E131 Terminology Relating to Molecular Spectroscopy
- E1421 Practice for Describing and Measuring Performance of Fourier Transform Mid-Infrared (FT-MIR) Spectrometers: Level Zero and Level One Tests
- E1492 Practice for Receiving, Documenting, Storing, and Retrieving Evidence in a Forensic Science Laboratory
- E1610 Guide for Forensic Paint Analysis and Comparison

# 3. Terminology

- 3.1 Definitions—For definitions of terms used in this guide other than those listed here, see Terminologies D16 and E131.
- 3.2 Definitions of Terms Specific to This Standard:
- 3.2.1 100 % line—calculated by ratioing two background spectra taken under identical conditions; the slope and noise of 100 % lines are used to measure the performance of the instrument.
- 3.2.2 absorbance (A)—the logarithm to the base 10 of the reciprocal of transmittance T, written as  $A = log 10 (1/T) = -log^{10}$  T.
- 3.2.3 absorbance spectrum—a representation of the infrared spectrum in which the ordinate is defined in absorbance units (A); absorbance is linearly proportional to concentration and is therefore used in quantitative analysis.
- 3.2.1 *additive* (*modifier*)—(*modifier*), *n*—any substance added in a small quantity to improve properties; additives <del>may</del> include substances such as driers, corrosion inhibitors, catalysts, ultraviolet absorbers, and plasticizers.
- 3.2.5 attenuated total reflectance (ATR)—a method of spectrophotometric analysis based on the reflection of energy at the interface of two media that have different refractive indices and are in intimate contact with each other.
  - 3.2.6 aperture—an opening in an optical system that controls the amount of light passing through a system.
  - 3.2.2 background—background, n—the signal produced by the entire analytical system apart from the material of interest.
- 3.2.8 beam condenser—a series of mirrors that focus the infrared beam in the sample compartment to permit the examination of smaller specimens.
- 3.2.9 beam splitter—an optical component that partially reflects and partially transmits radiation from the source in such a manner as to direct part to a fixed mirror and the other part to a moving mirror.
- 3.2.3 <u>binder—binder</u>, <u>n—</u>a nonvolatile portion of the liquid vehicle of a coating, which serves to <u>bindbond</u> or cement the pigment particles together.
- 3.2.4 *coating—coating*, *n*—a generic term for paint, lacquer, enamel, or other liquid or liquefiable material that is converted to a solid, protective, or decorative film or a combination of these types of films after application.
- 3.2.12 deuterated triglycine sulfate (DTGS) detector—a thermal detector that operates at room temperature but lacks the sensitivity for use with microscope accessories.
- 3.2.5 extraneous material (contaminant, foreign material)—material), n—material originating from a source other than the specimen.
- 3.2.6 <u>interferogram—meaningful difference(s)</u>, <u>n</u>—a plot of the detector output as a function of retardation. feature or property of a sample that does not fall within the variation exhibited by the comparison sample, considering the limitations of the sample or technique, and therefore indicates the two samples do not share a common origin. The use of this term does not imply the formal application of statistics.
- 3.2.7 *microtomy*—*microtomy*, *n*—a sample preparation method that sequentially passes a blade at a shallow depth through a specimen, resulting in sections of selected thickness.
- 3.2.16 mercury cadmium telluride (MCT) detector—a quantum detector that utilizes a semi-conducting material and requires eooling with liquid nitrogen to be operated; this type of detector is commonly used in microscope accessories due to its sensitivity.
  - 3.2.8 *paint—paint*, *n*—a pigmented coating.
- 3.2.9 *pigment*—*pigment*, *n*—a finely ground, inorganic or organic, insoluble, and dispersed particle; besides color, a pigment may pigments provide many of the essential properties of paint such as opacity, hardness, durability, and corrosion resistance; the term pigment includes extenders.
- 3.2.19 representative sample—a portion of the specimen selected and prepared for analysis that exhibits all of the characteristics of the parent specimen.
  - 3.2.20 significant difference—a difference between two samples that indicates that they do not share a common origin.
- 3.2.10 <u>smear—smear, n—</u>a transfer of paint resulting from contact between two objects; these transfers <u>maycan</u> consist of co-mingled particles from two or more sources, fragments, or contributions from a single source.
  - 3.2.22 specimen—a material submitted for examination; samples are removed from a specimen for analysis.

- 3.2.23 transmittance (T)—the ratio of the energy of the radiation transmitted by the sample to the background, usually expressed as a percentage.
- 3.2.24 transmittance spectrum—a representation of the infrared spectrum in which the ordinate is defined in %T; transmittance is not linearly proportional to concentration.
- 3.2.25 wavelength—the distance, measured along the line of propagation, between two points that are in phase on adjacent waves.
- 3.2.26 wavenumber—the inverse of the wavelength; or, the number of waves per unit length, usually conveyed in reciprocal centimeters (cm<sup>-1</sup>).

#### 4. Summary of PracticeGuide

- 4.1 The film forming portion of a paint or coating is the organic binder, also referred to as the resin. The binder forms a film that protects as well as displays the bonds to substrates and can contain organic and inorganic pigments that make a coating both decorative and functional. Infrared spectroscopy is commonly employed for the analysis of paint binders, pigments and other additives that are present in detectable concentrations.
- 4.2 Paints and coatings absorb infrared radiation at characteristic frequencies that are a function of the coating's composition. These absorption frequencies are determined by vibrations of chemical bonds present in the various components.
- 4.3 The analysis of coatings using infrared spectroscopy can be carried out using either transmission or reflectancereflection techniques. These measurements can be taken with a variety of equipment configurations and sampling accessories, the most common being the use of an infrared microscope. A variety of accessories can also be utilizedused in the system's main bench. However, the use of a nonmicroscope non-microscope accessory typically requires a larger sample size than those that can be analyzed using a microscope.
- 4.4 For transmission FTIR, a thin-peel of each paint layer, or a thin cross-section of a paint sample is made either by hand with a sharp blade or using a microtome. It is then analyzed using either a microscope attachment or other suitable accessory, such as a diamond anvil cell. When thin samples suitable for transmission FTIR are not obtainable, reflectance reflection techniques (ATR, internal total reflection) may can be employed using microscope objectives or bench accessories.
  - 4.5 Basic Principles:
- 4.5.1 Infrared spectroscopy (mid-range) is eapable of utilizing a the spectral range between 4000 and approximately 400 cm<sup>-1</sup>. Extended range instruments are needed to take measurements down to approximately 200 cm<sup>-1</sup>. The actual spectral cutoff depends upon the type of detector and optics used.
- 4.5.2 An FTIR spectrometer measures the intensity of reflected or transmitted radiation over a designated range of wavelengths. The spectrum of a sample is produced by ratioing the calculating the ratio of the transmitted or reflected infrared spectrum to a background spectrum. Televal/catalog/standards/sist/6d41b3d5-a5e6-43ed-9834-f29ca52b7ddb/astm-e2937-18
- 4.5.3 Transmission spectra <u>maycan</u> be plotted either in percent transmittance (%T)(%T), or in absorbance (A). <u>Reflectance Reflection spectra maycan</u> be plotted either in percent reflectance (%R) or in <u>absorbance (A).</u>—log10 (R) units.
  - 4.6 *Instrumentation:*
- 4.6.1 An FTIR instrument consists of a source to produce infrared radiation, an interferometer, a detector and a data processing device. A micro-FTIR instrument also has a microscope equipped with a detector and infrared compatible optics.
- 4.6.2 Most FTIR systems are equipped to collect data using the main bench in the range of 4000 to 400 cm<sup>-1</sup>. Extended range systems are equipped with a beamsplitter and optics that allow transmission down to approximately 200 cm<sup>-1</sup>. Systems equipped with an FTIR microscope utilizeuse a more sensitive detector type. Depending on the specific detector type, microscopic samples can be analyzed in the range of approximately 4000 to 450 cm<sup>-1</sup>.

# 5. Significance and Use

- 5.1 FTIR spectroscopy maycan be employed for the classification of paint binder types and pigments as well as for the comparison of spectra from known and questioned coatings. When utilized for comparison purposes, the goal of the forensic examiner is to determine whether any significant meaningful differences exist between the known and questioned samples.
- 5.2 This guide is designed to assist an examiner in the selection of appropriate sample preparation methods and instrumental parameters for the analysis, comparison or identification of paint binders and pigments.
- 5.3 It is not the intent of this guide to present comprehensive theories and methods of FTIR spectroscopy. It is necessary that the examiner have an understanding of FTIR and general concepts of specimen preparation prior to using this guide. This information is available from manufacturers' reference materials, training courses, and references such as: Forensic Applications of Infrared Spectroscopy (Suzuki, 1993) (4), Infrared Microspectroscopy of Forensic Paint Evidence (Ryland, 1995) (5), Use of Infrared Spectroscopy for the Characterization of Paint Fragments (Beveridge, 2001) (6), and An Infrared Spectroscopy Atlas for the Coatings Industry\_(2).

### 6. Sample Handling

- 6.1 The general collection, handling, and tracking of samples shall meet or exceed the requirements of Practice E1492 as well as the relevant portions of the SWGMAT's Trace Evidence Quality Assurance Guidelines (7).
- 6.2 The work area and tools used for the preparation of samples shall be free of all extraneous materials that could transfer to the sample.
- 6.3 As stated in Guide E1610, a paint specimen should first be examined withusing a stereomicroscope, noting its size, appearance, layer sequence, heterogeneity within any given layer, and presence of any material that could interfere with the analysis (for example, traces of adhesive, surface abrasion transfers, or zinc phosphate conversion coating residue on the underside of the base primer on electro-coated parts). Some surface materials maycan be of interest and therefore maycan be worthy of analysis before removal.
  - 6.4 Each layer of a multi-layered paint should be analyzed individually.
- 6.5 When analyzing difficult items (for example, smears, dirty or heterogeneous specimens) care shall be taken when sampling the paint and in choosing appropriate analytical conditions. An attempt should be made to remove any extraneous material from the exhibit before sampling. It may be necessary to If possible, analyze a number of samples areas to ensure reproducibility and understand inter/intrasample compositional variation.
- 6.6 Extraneous material should be removed either by scraping with a suitable tool such as a scalpel or washing with water. If needed, alcohols or light aliphatic hydrocarbons can be useful for cleaning. However, it should be noted that the use of organic solvents for cleaning paint can alter the composition by extracting soluble components such as plasticizers or dissolve the paint binder. Therefore, cleaning with organic solvents should be considered the method of last resort. If solvents are used, known and unknown samples should be treated the same, making sure no residual solvent remains.
  - 6.7 For the accurate-comparison of paint evidence, samples should be prepared and analyzed in the same manner.

# 7. Analytical Techniques and Operating Conditions

- 7.1 Paints <u>maycan</u> be analyzed by transmission or <u>reflectance utilizing reflection methods using</u> the microscope accessory or <u>the</u> bench accessories. The technique chosen is dependent upon the physical nature of the paint, the quantity of sample, preparation and analysis time, available equipment, and access to reference libraries for that technique. The same technique should be used on both known and questioned samples. <u>It may be necessary to use Use</u> multiple preparation or analytical techniques, or both, in <u>orderas necessary</u> to analyze all layers and characteristics.
- 7.2 The type of detector and beam splitter dictates the spectral range of the FTIR spectrometer. Mid-range infrared instruments use alkali halide beam splitters that are made from either cesium iodide (CsI) or potassium bromide (KBr).
- 7.3 The most common infrared detector used on the main bench is a deuterated triglycine sulfate (DTGS) detector. The DTGS detector operates at room temperature. A spectrometer equipped with a DTGS detector and CsI optics has an approximate spectral range of 4000 to 200 cm<sup>-1</sup>. With KBr optics and a DTGS detector, the spectral range of the spectrometer is approximately 4000 to 400 cm<sup>-1</sup>.
- 7.4 The detector commonly used with the microscope accessory is a mercury-cadmium-telluride (MCT) detector. The MCT detector is approximately 40× more sensitive than the DTGS detector, but has a narrower spectral range with a lower limit of 700 to 450 cm<sup>-1</sup>, depending on the type.
- 7.5 Infrared data are collected from both the sample and a previously stored or newly acquired background. Taking the ratio of the sample spectrum to the background enables removal of absorptions from the cell or support material (for example, diamond absorptions) or from the atmosphere (for example, carbon dioxide and water vapor), or both. The latter absorptions can be minimized by purging with dried and filtered air desiccant packs or nitrogen gas. The number of scans acquired for each specimen can vary depending on sample type and size.
  - 7.6 Main Bench Transmission Techniques:
- 7.6.1 The most <u>A</u> common bench transmission technique for the analysis of paint is the use of the diamond cell with a beam condenser.
- 7.6.1.1 Either prior to or after analysis, a background spectrum of the empty diamond cell is collected. The same background spectrum maycan be used for multiple samples or a new one maycan be collected for each sample analysis.
- 7.6.1.2 A sample from a single paint layer is placed on the clean diamond anvil cell and compressed between the windows to a desired thickness. Both high-pressure and low-pressure diamond cells can be used in conjunction with a beam condenser. Sample compression is normally done under a stereomicroscope to ensure uniform coverage. The cell is then placed in the sample holder in the main bench of the instrument. The instrument is allowed to equilibrate. This process is dependent upon the type of instrument and efficiency of the purge. A spectrum is then collected with the sample in place. Typically 16 to 256 scans are collected with a resolution of 4 cm<sup>-1</sup>. These parameters maycan vary depending on the instrument and size and nature of the sample. The same instrumental parameters, including the number of scans, should be acquired for the background as for the sample.



- 7.6.1.3 Diamond absorbs infrared radiation in the 2300 to 1900 cm<sup>-1</sup> region; therefore, sample absorptions in this region may can be obscured if the diamond path length is too long.
  - 7.7 Attenuated Total Reflectance (Main Bench):
- 7.7.1 A number of Numerous in-bench single reflection ATR accessories are commercially available. The general principles of operation are the same for each accessory. The sample of interest is placed in direct contact with the internal reflecting crystal, such as diamond or KRS-5. Some accessories employ a viewing microscope to facilitate proper placement of the sample or area of interest
- 7.7.2 In contrast to transmission methods, ATR methods require little or no sample preparation, although the <u>pressureforce</u> applied when using the ATR accessory <u>maycan</u> deform the sample.
- 7.7.3 Once in contact with the crystal, multiple scans are collected. The material is then removed and the crystal is cleaned. Background scans are collected with the item removed, either before or after the sample scans. Typically 16 to 256 scans are collected at a resolution of 4 cm<sup>-1</sup>. These parameters maycan vary depending on the instrument and size and nature of the sample.
  - 7.8 FTIR Microscope Accessory:
- 7.8.1 In forensic science, infrared microspectroscopy is the most-commonly used method-for acquiring the infrared spectrum of a paint. Spectra can be obtained from samples as small as 10 to 20  $\mu$ m in diameter, using transmittance, reflectance transmission and ATR\_reflection methods. MCT detectors are commonly used with microscopes due to the higher sensitivity needed for small samples. They-These detectors are available in configurations usually designated as narrow band and broad (wide) band with the lower energy cut-off ranging from approximately 700 to 450 cm<sup>-1</sup>. There is a compromise between sensitivity and spectral range with these detectors. A detector with the spectral range of 4000 to 650 cm<sup>-1</sup> and an area of  $0.25 \times 0.25$  mm<sup>2</sup> is typically used for paint examination since it offers the optimal balance between spectral range and sensitivity. These detectors shall be cooled by liquid nitrogen before use. When using the lower sensitivity/broader spectral range detector, larger samples are required.
  - 7.8.2 Transmission Measurements:
- 7.8.2.1 Transmission methods generally require more extensive sample preparation. The sample shall be thin enough not to over-absorb. For transmission data viewed in % transmittance, spectral peaks optimally should not fall below 10 % T. For spectra displayed in absorbance, the maximum absorbance optimally should be 1.0 or less.
- 7.8.2.2 A prepared and mounted sample is placed on the microscope stage and focused. The condenser on some instruments may have to can be adjusted to account for the thickness of a support window. The sample is observed with visible light and the area to be analyzed is centered in the field of view. The area of interest is isolated from the remainder of the field of view with one or two apertures.
- 7.8.2.3 The number of apertures corresponds to the instrument configuration. The apertures control the area and location of the infrared beam striking the sample and the transmitted light reaching the detector.
- 7.8.2.4 Apertures also block unwanted radiation originating outside of the area of interest. If stray light is allowed to reach the detector, absorption intensity is reduced.
- 7.8.2.5 As a sample area of interest becomes smaller, or as the aperture(s) are reduced so that a portion of the sample can be isolated, diffraction effects rapidly increase. These effects can be experienced when using aperture sizes smaller than 25  $\mu$ m  $\times$  25  $\mu$ m.
- 7.8.2.6 To minimize the effects of heterogeneity, aperture areas greater than  $2500 \, \mu m^2$  (for example,  $50 \, \mu m \times 50 \, \mu m$  or  $25 \, \mu m \times 100 \, \mu m$ ) should be used when possible. Alternatively, multiple areas of the sample can be analyzed to determine the range of spectral characteristics.
- 7.8.2.7 Once the area of interest is isolated by adjusting the magnification and apertures of the microscope, the infrared spectrum of the sample is collected. Typically 16 to 256 scans are collected at a resolution of 4 cm<sup>-1</sup>-or better.
- 7.8.2.8 The background spectrum is collected from an unused area of the support window using the same aperture configuration as used for the sample.
- 7.8.2.9 If sample size is limited, the resulting spectrum maycan be noisy. To increase the signal to noise ratio (S/N), the number of scans can be increased. It is important to collect spectra with goodhigh S/N to permit visualization of fine detail such as small sharp peaks or shoulders in the resultant spectrum.
  - 7.8.3 *Reflection Measurements (Microscope):*
- 7.8.3.1 The FTIR can also be used in the reflectance mode. However, most of the reference IR spectral data of coatings, binders, pigments and additives consist of transmission spectra. Furthermore, being surface analysis techniques, inconsistencies in the preparation of surfaces subject to comparative assessments can be problematic for data interpretation. Additionally, when analyzing individual layers in cross section and using the requisite small apertures, signal-to-noise constraints are even greater than those encountered in transmission analyses.
  - 7.8.4 Reflection Measurements (Microscope): Reflection/Transmission (Transflection):
- 7.8.3.1 The FTIR microscope can also be used in the reflection mode. However, in most cases, transmittance methods are preferred for several reasons. Refractive index changes, and differences in infrared absorption coefficients for different wavelengths, give rise to distortions in reflectance spectra. Reflectance spectra are not absorption spectra and cannot be compared in detail to transmission spectra due to shifts in spectral peak wavelengths and variations in spectral peak intensities (8). Also, most of the reference data of coatings, binders, pigments and additives consist of transmission spectra. Furthermore, being surface



analysis techniques, inconsistencies in the preparation of surfaces subject to comparative assessments can be problematic for data interpretation. Additionally, when analyzing individual layers in cross section and using the requisite small apertures, signal-to-noise constraints are even greater than those encountered in transmission analyses.

- 7.8.4.1 If samples are compressed directly on This method is used to analyze thin films of a sample on an infrared reflective surface, such as a glass slide made of infrared light reflecting architectural glass (low e-glass), the microscope's reflection mode can be used to produce spectra mimicking double-pass transmission spectra. or metal. The sample is viewed using visible light and the area to be analyzed is centered in the field of view. The area of interest is isolated from the remainder of the field of view with an aperture and the infrared spectrum is collected. Typically, 16 to 256 scans are collected at a resolution of 4 cm<sup>-1</sup>. The technique is sometimes referred to as "transflection" or "reflection/absorption". Some wavelength maxima shifts may be observed in intense absorption bands:background spectrum is collected from an unused area of the reflective support using the same aperture configuration and number of scans as used for the paint sample.
- (1) For transflection, the thinned sample is placed on an infrared reflective surface, such as a glass slide made of infrared light reflecting architectural glass (low e-glass), or a gold mirror, and placed on the microscope stage. It is viewed using visible light and the area to be analyzed is centered in the field of view. The area of interest is isolated from the remainder of the field of view with an aperture and the infrared spectrum is collected. Typically, 16 to 256 scans are collected at a resolution of 4 cm<sup>-1</sup> or better. The background spectrum is collected from an unused area of the reflective support using the same aperture configuration and number of scans as used for the paint sample.
  - 7.8.5 ATR Objectives for Infrared Microscopes: ATR:
- 7.8.5.1 ATR microscope objectives maycan be fitted with a silicon, ZnSe, diamond, KRS-5, or germanium internal reflecting crystal offering a wide element. Different internal reflection elements (IREs) offer a variety of penetration depths and erystal physical or chemical attributes. The sample is viewed using visible light and the area to be analyzed is centered in the field of view. The crystal is then placed in direct contact with the area of interest. Monitoring the single beam spectrum provides an indication of whether there is sufficient contact between the sample and crystal. Typically 64 to 512 scans are collected at a resolution of 4 cm<sup>-1</sup> or better and ratioed against an air background. The number of scans collected for each sample can vary with size or type.

# 8. Sample Preparation Methods and Sampling Accessories 2002 COS

- 8.1 The method chosen for sample preparation depends on the size, nature, and condition of the specimen, as well as the particular FTIR technique/accessory that is employed for analysis. It may be necessary to use <u>Use</u> multiple preparation or analytical techniques, or both, in orderboth as necessary to analyze all layers and characteristics.
  - 8.2 Transmission Techniques (Main Bench):
- 8.2.1 Samples prepared for analysis by main bench transmission techniques shall be thin enough to allow infrared radiation to pass through without over-absorption. For transmission data that are viewed in absorbance, the sample optimally should be thin enough to produce a maximum absorbance of 1 absorbance unit. For transmission data viewed in % transmittance, spectral peaks ideally should not fall below 10 % T. This typically requires thicknesses of approximately 5 to 10 µms.
- 8.2.2 The separation and analysis of individual layers is recommended in order to determine chemical composition and detailed spectral characteristics of each layer. This <u>maycan</u> be achieved by microtomy or by hand using a sharp blade while observing the evidence under a stereomicroscope.
- 8.2.3 Sample preparation techniques which <u>maycan</u> be employed for transmission analysis using the main bench include a thin peel stretched over an aperture, an alkali halide pellet (for example, potassium bromide), or a diamond cell.
- 8.2.4 Thin peels of each layer can be placed on a glass microscope slide and compressed with the flat beveled surface of a scalpel blade, a roller bearing tool, or other equivalent technique, and then placed directly over a small masking aperture for analysis. Given the small sample sizes encountered, a beam condenser is typically employed.
- 8.2.5 Potassium bromide (KBr) pellets are made by grinding a small aliquot of the individual paint layer with dry spectroscopic grade KBr using a mortar and pestle. The powder is then transferred to a die maker and a press is used to generate the pellet. As KBr is deliquescent, the pellets, KBr powder and die maker should be stored in an oven or desiccator. Given the small sample sizes encountered, one to three millimeter diameter pellets mounted in a beam condenser are typically employed.
- 8.2.6 The diamond anvil cell is a useful sampling technique when the greatest spectral range is required and for laboratories that do not have a microscope accessory for their infrared spectrometer. It is possible to obtain spectra down to approximately 200 cm<sup>-1</sup> with cesium iodide (CsI) optics, or down to 400 cm<sup>-1</sup> with KBr optics. Both high- and low- pressure diamond cells can be used in conjunction with a beam condenser. Pliable and powdered samples are amenable to low-pressure diamond cells, while harder paints maycan require the use of high-pressure diamond cells. Low pressure diamond cells have the advantages of costing less, are compatible with an infrared microscope accessory and do not obscure spectral information in the 2400 to 1800 cm<sup>-1</sup> region because they contain thinner diamonds than high pressure cells.
- 8.2.7 Diamond cells permit relatively simple sample preparation. The cell consists of two diamond windows, a holder for each diamond and a means of compression that achieves an appropriate sample thickness. There are a variety of designs available. The paint is simply placed on one of the diamond faces, the second diamond is positioned on top, and sufficient pressure is applied to form a film. This is normally done under a stereomicroscope to ensure uniform coverage of the diamond face.