1994 meeting

Infrared User's Group (IRUG) for the Analysis of Artistic and Historic Materials

at the
Philadelphia Museum of Art
March 24-25, 1994

Co-sponsored by
Philadelphia Museum of Art
The Getty Conservation Institute

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Meeting Schedule

March 24-25, 1994

Schedule for Thursday March 24, 1994:

Five minutes are allowed for questions directly following each talk. There will also be cards on which additional questions may be written down and time for more questions at the end of each session.

8:45 am A-2 door for handicap visitors is available for entry (Museum opens at 10 am).

9:00 am Welcoming remarks (Marigene H. Butler, Michele Derrick)

Session A: Sampling and Analysis Techniques (Beth Price - session chair)

9:10 - 9:35 Concetta Paralusz, Permacel "Miscellaneous Techniques for Handling Small Samples Including Polymers and Coatings"

9:40-10:05 Ray Meilunas, Grumman Corporate Research Center, "The Application of Diffuse Reflectance FT-IR to the Analysis of Paint Surfaces

10:10-10:25 Boris Pretzel, Victoria and Albert Museum "Silicon Carbide Sampling for FT-IR"

10:30-10:45 Break with coffee, tea and danish

10:45-11:10 Jim Cronin, E.I. Dupont "Applications of Photoacoustic Spectroscopy to Real World Samples"

11:15-11:40 John Reffner, Spectra-Tech Inc. "The Evolution of Infrared Microspectroscopy- Its Instrumentation and Techniques for Sample Analysis"

11:45-12:10 Michele Derrick, The Getty Conservation Institute "Microtoming and Embedding Methods to Prepare Thin Sections for IR Analysis"

12:10-12:20 Questions

12:30-1:30 Lunch (sponsored by The Getty Conservation Institute)

Session B: Analysis of Art Materials (Eugena Ordonez - session chair)

1:45 - 2:10 Pam Martoglio, Spectra-Tech Inc. "The Application of Infrared Microspectroscopy to Archaeological Fabrics and Dyes"

2:15 - 2:35 Richard Newman, Boston Museum of Fine Arts "Applications of IR Microspectrometry to Identification of Inorganic Materials in Works of Art"

2:40 -3:00 Elizabeth Moffatt, Canadian Conservation Institute "Infrared Spectroscopic Analysis of Artists' Materials with Examples from the Works of Three Canadian Painters"

3:00 - 3:15 Questions
3:15 - 3:30 Break
3:30 - 5:30 Demonstrations

- Sadtler IR Search Master Software and Sadtler Libraries - Michael Frey, Sadtler Nicolet FT-IR bench and OMNIC software - Beverly Bostwick Nicolet
- Infrared Microprobe (IRuS) - John Reffner Spectra-Tech Inc. ATR objective - Spectra-Tech Inc.
- Micro diamond cell - High Pressure Diamond Optics
- Silicon Carbide sampling for Diffuse Reflectance - Boris Pretzel V&A Museum Microtoming and Embedding Techniques - James Martin, Williamstown
- Glass Knife making - Michele Derrick GCI
- Gettens Raw Materials FT-IR Spectral Library - Beth Price Philadelphia Museum of Art and Amy Snodgrass Harvard Art Museums
- Lab Calc software from Galactic - Jan Carlson Winterthur

(Self-guided tour of Philadelphia Museum of Art - closes at 5 pm)

Schedule for Friday March 25, 1994:

9:00 am A-2 door for disabled visitors is available for entry (Museum opens at l0am).

Session C: Instruments and Data Analysis/Transfer (Michele Derrick - session chair)

9:15 - 9:35 Mike Odem, Nicolet Instruments "FT-IR House Cleaning: The Care and Maintenance of Your System"

9:40 -10:05 Jim Landry, Loyola Marymount University "Spectral Manipulations: The What, When and How"

10:10-10:30 Break with coffee, tea and danish

10:30 -10:55	Jerry Lillquist, 3M Center, "Interpretation of Infrared Spectra of Adhesives"
11:00-11:25	Michael Boruta, Sadtler, "Optimization of Infrared Techniques for Collection and Searching of Digital Library Data"
11:30-11:50	Angela Santiago University of Bridgeport "Introduction to Internet"
11:50-12:05	Questions
12:15-1:30	Lunch
1:30-2:00	Business meeting (future of IRUG and spectra collection)
2:00-5:00pm	Discussions and working groups: Talk about problem spectra, successful analysis, sample preparation methods, etc. Also this time is available to test out many of the items that were demonstrated on Thurs. Self-guided tour of Philadelphia Museum of Art - closes at 5 pm.

Participant List

(Alphabetical order)

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Abstracts

(alphabetical order)

FT-IR STUDY OF ORIGINAL AND SUPERIMPOSED MATERIALS IN WALL PAINTING CONSERVATION

P. Baglioni, L. Dei, E. Ferroni, and G. Sarti

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The FT-IR spectroscopy coupled with the microscope is a very powerful technique in the field of works of art conservation. One of the most interesting fields of application of this technique is the study of the materials - original and/or superimposed - present on the surface of wall paintings. In this report we summarize some recent results concerning the characterization of pigments, efflorescence products, and organic substances taken from different wall painting surfaces. The spectra were made in the microreflectance mode by using a Bio-Rad FTS-40 spectrometer coupled with a Bio-Rad microscope UMA-500.

During the restoration of the fresco La Madonna del Parto by Piero della Francesca a study on the original pigments used by the artist was performed'. In particular, FT-IR spectra showed that the blue pigment of the Madonna's vest was constituted not only of azurite - as believed -, but also of blue ultramarine. This last pigment presents a particular band 2 at 2346 cm-1, which appeared only with a few samples taken from the fresco, indicating that the pigmentation of the vest was not homogeneous.

Another interesting result was obtained with samples taken from a fresco by Antonio da Fabriano in the church San Domenico in Fabriano, Italy. In many zones of this wall painting a whitish efflorescence was present. The analysis performed by FT IR showed a spectrum with many bands, but it was possible to recognize three typical absorbances at 2396, 1763, and 826 cm-1 attributable to potassium nitrate3. This result was afterwards co-validated by X-ray diffractometry and X-ray fluorescence4. Finally, from the same fresco the FT-IR spectra showed also the presence of organic material. This material was demonstrated to be carboxymethylcellulose, which was used in the past years to protect the wall painting surface.

[1] L. Dei, and G. Sarti, I pigmenni azzurri della veste della Madonna del Parto, in Piero della Francesca, La Madonna del Parto, Restauro e iconografia, Marsilio Ed., Venezia, Italy, 1993, pp. 8687.

[2] M. R. Derrick, personal communication.

[3] The Infrared Spectra of Minerals, V. C. Farmer Ed., Mineralogical Society Monograph 4, Mineralogical Society, London, 1974.

[4] P. Baglioni, L. Dei, E. Ferroni, and G. Sarti, paper in preparation.

Photoacoustic Spectroscopy of Real World Samples

James T. Cronin

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Photoacoustic Spectroscopy (PAS) has at least two advantages over traditional Infrared sampling techniques: the ability to run non-transparent samples and the potential for depth profiling. A brief description of PAS theory will address the advantages and limitations of PAS. The surface sensitive nature of PAS can be successfully applied to a wide variety of samples, but can also be used to provide spectral information near and below the surface of the sample. Typical examples of non-transparent samples will be followed by a discussion of depth profiling and its application to carpet fibers treated with a stain resist.

Microtoming and Embedding Methods to Prepare Thin Section for IR Analysis

Michele Derrick

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Infrared analysis of the cross-sectional structure of some samples can provide important information on sample construction and on surface degradation. However, in order to obtain infrared transparent thin sections for small, often delicate samples from works of art, it necessary to embed the sample for support then microtome the sample to produce sections of 1-10 micron thicknesses. This paper will describe how-to microtome and embed samples. The causes and solutions for common problems such as sample pop-out, curling and chatter will be discussed.

Several factors make a significant difference in the ease of producing good sections. It is important to use the smallest sample that will contain all of the layers to be analyzed. To achieve this, a sample should be prepared in a pie shape and oriented with its point to the outer edge of the embedding. Then all the excess embedding resin should be trimmed away from the sample to produce a pointed facet (like a pencil). A sharp knife should be used for cutting. Glass knives are the preferred choice. The sample should be clamped soundly in an offset position of approximately 10 degrees from vertical (based on a rectangular sample). Thin sections (1 micron) are the easiest to cut. Samples should be cut very slowly (< 1 mm/sec) with a motorized microtome (if available). The microtome area should be free of vibrations, drafts and temperature changes. Thin sections can be easily handled with a brush containing only a few bristles or with a sliver of cured silicone.

Polyester embedding resin is recommend for use, due to its clarity, low shrinkage rates, room temperature cure and ease for microtoming the 1-10 micron sections needed for infrared analysis. However, problems of dissolution and infiltration that occur for some samples will be mentioned.

Sadtler IR Search Master Software and Sadtler Libraries

Michael C. Frey

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The discussion will center on building and searching digital databases. Points presented will include the importance of quality and consistency when developing databases. The tools and guidelines that Sadtler uses will be covered. These same procedures can be helpful when building proprietary databases. Also discussed will be how to recognize potential problems when searching databases and how to avoid or minimize their effects.

Interpretation of the Infrared Spectra of Adhesives

Gerald Lillquist

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The interpretation of infrared spectra is based both on experience and on spectra of known compounds, but even then, there are many situations where the exact identification of an unknown may never be determined by its infrared spectrum alone. This talk will be a short review of basic infrared spectral interpretation showing spectra of various commercial adhesives and components as examples. Adhesives are one example of the broad category of compounds called polymers which may have more than one function, i.e., rubbers, polyesters, epoxies, etc. that can be used both as adhesives and as coatings.

The Application of Diffuse Reflectance FT-IR to the Analysis of Paint Surfaces

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The utility of Diffuse Reflectance IR Fourier Transform (DRIFT) spectroscopy for the analysis of neat paint films and inks is described. The paints investigated include those commonly found in paintings of the Northern Italian Renaissance of the 14th and 15th centuries with binders restricted to linseed oil, egg yolk (tempera) and glue. The pigments in the various paints include vermilion, Indian red, red madder, yellow ochre, burnt umber, lead white, terre verte, malachite, copper resinate, ultramarine, and azurite. The inks studied are iron gall, bistre, and sepia. The diffuse reflectance spectra of the various paint combinations are found to be more complicated than those produced by other IR sampling techniques and thus more difficult to interpret. This complexity is shown to be due to the particular nature of the diffuse reflection phenomenon, which is the result of the combined interaction of transmission, specular reflection, internal reflection, and scattering components from the particulate sample. The merits and drawbacks of the DRIFT technique for characterizing binders in neat paint films are detailed. The advantages and drawbacks of the DRIFT technique over standard IR transmission methods are also discussed.

The Application of Infrared Microspectroscopy to Archaeological Fabrics and Dyes

Pam Martoglio

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Infrared microspectroscopy offers a number of advantages such as the ability to analyze extremely small samples without destroying the sample. This particular aspect is important in the study of historic artifacts since sample size is always limited.

Archaeological textiles from two different societies were studied with infrared microspectroscopy. The first textiles are from the Paracas Indian burial mounds. This society flourished between 400 B.C. and 400 A.D. in the region of the Paracas Necropolis in what is now southern Peru. The second group of artifacts studied is from the Etowah Indian Mounds in northwest Georgia. The Etowah Indians inhabited this region around 1200 A.D.

We were able to detect and identify the dyes used in the textiles. Visible microspectroscopy was also used to verify the results. The dyes were either used with a mordant to dye the fiber throughout or painted directly onto the finished textiles. Evidence of pseudomorph formation (mineralized fibers) was also detected on textiles, which had been buried in close proximity to copper artifacts.

Infrared Spectroscopic Analysis of Artists' Materials with Examples from the Works of Three Canadian Painters

Elizabeth Moffatt

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The analysis of paint samples by Fourier transform infrared spectroscopy (FTIR) at the Canadian Conservation Institute will be described using examples from the works of three Canadian artists. FTIR analysis of samples from a selection of paintings by Paul-Emile Borduas, Alfred Pellan and David Milne is being undertaken as part of the Canadian Artists' Painting Materials Research Project. Spectra will be presented to illustrate the identification of inorganic and organic pigments, binding media and varnishes. Spectra indicative of various pigment/medium interactions will also be discussed.

Analysis of the black and white pigments used in paintings executed by Paul Emile Boduas during the late 1950s will be discussed in relation to conservation problems associated with the works. The identification of various organic pigments and ketone resin varnishes in samples form works by Alfred Pellan will be described. Examples of pigment-drying oil interactions in paintings by Borduas and David Milne will be presented. Samples from a painting by Milne will be used to illustrate analytical difficulties encountered with small, multi-layered samples.

Samples are typically mounted in a low-pressure diamond anvil microsample cell for analysis using one of two Bomem spectrometers in the laboratory. For relatively large samples, the diamond cell is positioned in the microbeam compartment of the Michelson MB-100. Smaller samples are analyzed using the diamond cell and a Spectra Tech IR-Plan microscope coupled to a Michelson MB-120 spectrometer.

Applications of IR Micro spectrometry to Identification of Inorganic Materials in Works of Art

Richard Newman

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This paper reviews the applicability of IR to identifying inorganic materials. Specific applications include several ongoing projects on the materials used in Asiatic paintings (Tibetan thangka paintings and Japanese Buddhist paintings), projects in which the technique has been invaluable because of the very small sample sizes. A second application is to identification of corrosion products from metal objects, including organic acid salts. This paper will describe some of the available spectral libraries of inorganic materials and limitations in the types of compounds that can be identified.

FT-IR House Cleaning: The Care and Maintenance of Your System

Mike Odem

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The paper will cover the procedures for the maintenance of infrared spectrometers. Optimum environmental conditions can prolonge the life of the instrument and minimize sources for contamination. Methods for evaluating and optimizing the performance of the spectrometer and their relationship to the enhancement of spectral quality will be discussed.

Silicon Carbide Sampling for FTIR

Boris Pretzel

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Silicon carbide paper provides a suitable substrate for rapid sampling of organic residues for investigation by diffuse reflectance FTIR. Spectra can be recorded directly from thin layers of sample collected on the abrasive paper and no further sample preparation is necessary. The spectra of many organic residues taken in this way resemble standard transmission spectra. (Many inorganic materials, which are highly absorbing and have a high refractive index, give spectra distorted by components due to specular reflectance.) The technique allows discrete sampling of small amounts (1 mg or so) of material from the surfaces of objects. It is therefore particularly applicable to the study of museum and archaeological artifacts and can readily identify the class of material present.

The technique is routinely used at the Victoria & Albert Museum to identify various surface coatings (e.g. varnishes, waxes, resins, gums etc.). It is also being used to identify the principal components of plastic objects in the Museum's collection currently being surveyed to ascertain their condition.

The Evolution of Infrared Micro spectroscopy--Its Instrumentation and Techniques for Sample Analysis

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The invention of the ATR objective was a major advance in infrared microspectroscopy. The objective has reduced the role of sample preparation in obtaining high-quality spectra while providing a reproducible sample path length for both quantitative analysis and spectral subtraction. The micro-ATR technique is completely nondestructive, as it generates spectra from a sample's surface. MicroATR is another tool for the analyst to use when probing the composition of materials. In infrared microspectral analysis, the quality of spectra is determined by the spectrometer, the nature of the sample, and the preparation of the sample for analysis. Most often, it is the preparation of the sample that limits a spectrum's quality. While this is true of all spectroscopic measurements, precise sample preparation is most important in infrared microspectroscopy. Advances in sample preparation have led to higher-quality spectra, keeping pace with developments in instrumentation. The current state of sample preparation and instrumentation will be presented, along with a view of the future of FT-IR microscopy.

Miscellaneous Techniques For Handling Small Samples

Concetta Paralusz

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This talk is an overview of FT-IR methods for obtaining infrared spectra. Examples include use of ATR techniques, a mini diamond cell, a novel mini-diffuse reflectance accessory and use of beam condensers alone and in conjunction with other accessories. A short description will be given of applications to fibers, polymeric coatings, powders, gemstones, cured thermosets, and rubber compounds. A recommended practice for optimizing ATR sampling for different chemical types is presented. Also, the speaker will describe the work of ASTM subcommittee E-13.03 on infrared, in developing standard recommended practices for the field of infrared spectroscopy.

Questionnaire Responses

What kind of sample do you find the easiest to analyze?

1. A transparent coating, which turns out to be what it was suspected of being, e.g., "Incralac" or "cellulose nitrate".

2. Synthetic adhesives, films coatings

3. Pure organic materials - most coatings, adhesives, films or bulk polymers.

4. A pure coating

5. Sample coating on furniture, waxes. Although find it difficult to distinguish mixtures of plant and insect resins.

6. Powders, films, small crystals, liquid samples

7. Liquid samples

8. Samples that are solid, homogeneous, present in a relatively thick layer.

What has been your most difficult sample?

1. Aside from failures, blue spatters on a human skull, which I suggested to consist of a mixture of polyvinyl chloride, ester plasticizer, sodium carbonate and inorganic silicates (dirt) - soft PVC blowing material. The composition varied from spatter to spatter. An absolute minimum of sample preparation proved to be best. Even pressing a spatter to obtain a thin film was too much. I finally got an interpretable spectrum from a spatter put onto a bed of loose potassium bromide powder and doing reflectance on the microscope.

2. Multiple-layer or multiple component samples

3. An insoluble epoxy resin filled with glass fibers to form an extremely hard block was very difficult. Any type of mixtures is challenging. Charcoal, coals, bitumen and most black pigments have been very difficult.

4. Any complex mixture

5. Mixtures/multicomponent samples.
6. Sloppy sampling - ground mixed in with paint layers.

7. Solid alumino silicates (zeolites and binding materials)

8. A very small paint sample consisting of 10 layers Gels, pastes and glues

Special techniques for analysis of difficult samples:

1. Ultra-thin pressed wafers on vacuum chamber.

2. Diffuse reflectance cell (near IR)

3. I use a selective solvent extraction method on most mixtures that I have. The sample is placed on a non-reactive window (BaF2) and analyzed initially. If it is a mixture, I then start with a solvent series (hexane, chloroform or ethyl acetate, ethanol, acetone, and water). Which solvents are actually used depends on the bulk spectrum and whether the previous solvent had any effect, but the progression is always from least polar to more polar. The solvent drop (0.1 - 1 microliter) is placed directly on the bulk sample. It will evaporate quickly and any material that was soluble in the solvent will deposit in a ring around the sample as the solvent evaporates. A spectrum is then collected from the ring. Waxes are beautifully separated with the hexane or chloroform. Natural and synthetic resins will usually come out with chloroform, ethyl acetate. Ethanol and acetone can be used for further separations. When water is applied the pellet and holder are placed under an incandescent bulb to facilitate dissolution and increase evaporation rate. Always after the series and sometimes in-between solvents, the bulk sample is reanalyzed. It will typically contain, pigments, fillers and insoluble organics, such as drying oils. This method is an easy way to make sense of mixtures. Multiple solvents can be sequentially placed on the same sample - allowing for drying and analysis in between. The small drop size allows several samples to be extracted at the same time on the same pellet without the solvents running into each other. It is very important to run a blank drop of each solvent, to check for contamination. This may be due to solvent, bottle, capillary, or previous sample residue on pellets.

4. Do mini extractions in well slides to separate components.

Which method do you use the most often for analyzing samples? Why?

1. Opened diamond anvil cell on infrared microscope; a quick method, which applies to almost all of my samples.

2. Flattening sample directly on mini-salt plate or in a diamond cell.

3. For most samples, I place a small section on a BaF2 pellet, flatten, and then analyze in a microspectrometer. If the material appears to be a mixture, I use a sequence of solvents (micro-drops directly on the sample on the pellet) to separate the components. The BaF2 is not affected by most solvents.

4. Microspectroscopy. Small sample sizes and ease of mounting samples.

5. Mount samples with roller on diamond cell and analyze in micro accessory.
6. Extract samples in well slides for mini-separations.

7. Pressing sample in diamond cell, analyzing by transmittance microscopy.

8. Liquid samples concentrated over a Kr salt plate (organic solvent based). Run using regular transmission technique.

9. Microreflectance (Microscope Bio-Rad UMA 500 and Spectrometer Bio-Rad FTS-40) when the samples are sufficiently thin and transparent; diffuse reflectance (the same spectrometer equipped with the Drift accessory Specac Graseby) when the sample are powders or can be powdered.

10. Low-pressure diamond anvil microsample cell. It is very easy to mount the sample; the sample is not contaminated and can be easily recovered for other analysis. It is compatible with IR microscope accessory.

What type (KBr NaCl, AgC1...) and brand of salt pellets/powder do you use?

1. Formerly used KBr powder and pellets. Infrequently use KRS-5 powder - all from Spectra Tech.

2. NaCl, KCl mini (1 mm x 6mm diameter) salt plates. Do not use pellets.

3. BaF2 windows (either 13 x 1 or 13 x 2 mm) from McCarthy Scientific, Fullerton CA

4. None

5. Almost never use pellets.

6. KBr from Merck, Darmstadt, Germany or from Carlo Erba, Milan Italy

7. KBr essentially for mid IR. If one would like to venture into far IR, CsI is the choice and for environmental chamber CaF2 is perfect due to resistance and durability (not for far IR).

8. We rarely do any work with salt pellets/windows.

9. We have KBr, but have not used pellets in 3 years.

What type of packaging do you use to store or transport samples?

1. Pierce's Reacti-vials for transportation of microsamples. Folded-up weighing paper/glassine for storage.

2. Glass vials with inert plastic caps. Glass microscope slides or well slides with glass cover slip.

3. For small samples, they are collected, transported and stored in glass well slides with a regular slide as a lid. This is easy to label, great for viewing under an optical microscope and easy to remove samples for subsequent analyses.

4. Glass cup slides for storage

5. Well slide/microscope slides taped together. Vials for architectural samples.

6. Microcentrifuge vials for larger samples, or sometimes gelatin capsules. Smaller samples are stored in cup slides (well slides).

7. Desiccator with desiccating agent (drierite).

8. Storing in glass or polyethylene vials hermetically plugged and transporting the vials in pocket equipped in the interior with air bubble nylon, to prevent breaking.

9. Glass microscope slides with concavities.

10. 2.5 ml polyethylene vials with snap-on plastic caps.

Describe the worst sample packaging that you have seen.

1. Particulate samples submitted on the sticky side of adhesive tape.

2. Sample removed from object with solvent-moistened swab. Still moist swab then placed directly in a plastic cup, which promptly dissolved. Do not like plastic vials or gelatin capsules.

3. I received some wall painting samples (that were mostly wall) inside glassine paper that had been stapled to form an envelope. This probably would not have been to disastrous, except in the same package, reference samples of oils were sent and one of the vials of oil had leaked onto the samples and into the packages.

4. Zip-loc baggy with lots of static.

5. Paint sample was placed on a strip of Scotch tape, which was folded over to seal in the sample. It was extremely difficult to remove the sample without it breaking up. Adhesive residue clung to the paint fragments.

6. We have never seen bad sample packaging.

7. Architectural paint sample in a baggy.

What type of contamination problems have you noticed?

1. Samples scraped from painted objects in such a way that ground, pigment, medium, coating, surface grime are all mixed together.

2. In some KBr powders, I have noticed a sharp absorption at 1384 cm-1 that is really annoying. I also have noticed that commercially prepared q-tips have an adhesive that attaches the cotton to the stick. It is typically PVAC. In using the solvent extraction method, I always run a blank drop of solvent, because the solvent, the capillary tubes and the bottles have at one time or another been sources of contamination.

3. Nothing significant

4. It depends on the complexity of the sample in study. if the sample is a composite of different materials (as often samples encountered in art conservation) is very difficult to establish contamination in an environmental chamber.

5. Nothing with IR. We are careful to keep diamond cells clean, and tools clean.

6. No contamination problems related to the packaging. We have noticed some problems of contamination when analyzing substances, i.e. pigments, into solid matrices - like plaster, mortar, wood, paper, etc..... due to the impossibility to discriminate between the signals of the substance and the support.

7. Contamination has been observed in surface coating samples taken with a solvent moistened swab.. Are there soluble materials in the cotton wool?

Please describe any problems or questions you may have with you instrument.

1. Mattson 4326 Update requires resetting after a few hours in 'noninteractive' collection mode. It is not immediately apparent that the reset is needed.

2. Would like to make better use of reflectance capabilities.

3. Water-cooling unit for the source has had problems with the lines clogging. We have had to flush it about every 6 months and the last time we had to take it apart and replace quick disconnects.

4. Software is finicky.

5. We have lost vacuum and alignment on the MCT detectors.

6. Spectrometer BIO-RAD FI'S 40 and Microscope BIO-RAD UMA 500:
    1) Problems in performing spectra with the microscope in the transmission mode with the NaCl window because of the difficulties in preparing then section of the samples: 2) problems of software with the name of background spectrum;
    2) The maximum distance between platform in which the samples are put below the microscope and the zoom lens does not permit the direct analysis of big samples (high > 3.5 cm).

7. Tube connecting funnel to detector on microscope frequently slips out and has to be re-inserted to pour in liquid N2.

<u>For dual aperture microscope users: Have you ever rechecked the alignment of the apertures with a pellet in place? Are they still in alignment?</u>

1. I did this once after I was not getting any throughput on a very small aperture (20 x 40) and I found that it was indeed not aligned with the pellet in place although the alignment without the pellet was still fine. Now I only align with a pellet in place.

2. I check the alignment of the microscope regularly and generally it stays in alignment.

3. Alignment is checked weekly, but generally only minor adjustments are required.

4. Check the alignment once a month; it is almost always aligned.

<u>How often do you run background spectra?</u>

1. Once per spectrum.

2. Routinely, about twice a day. More frequently if sample sizes or operating conditions change substantially.

3. With nearly every spectrum. I found that the baseline is better if the size of the aperture window is exactly the same for the background and the sample.

4. Almost every spectrum.

5. Run a new background when change the aperture size, when change to different diamond cell, or if start to notice C02 peaks and if change to a NaCl plate.

6. Every sample, or if more than part of a given sample is being analyzed, the same background may be used more than once.

7. Every day.

8. Every time we change mode. With the same mode, once a week.

9. Daily in the spectrometer; more often with the microscope.

How many scans do you collect for a spectrum?

1. 512 (or 1024) scans

2. As few as 32 for very good samples; as many as 1000 if sample is extremely small.

3. Typically run 200 scans for every sample. If I am checking several spots on a sample or extraction ring, I drop down to 50 scans, and then when I find a good area I usually run 200 scans.

4. 200 scans.

5. 200 scans

6. It depends on the mode and the type of samples. Usually:
    1) Micro reflectance scans >200;
    2) Diffuse reflectance 50 <scans<500;
    3) Transmittance with KBr pellet scans <50;
    4) Circle cell (liquid sample) 100<scans<500.

7. 200 scans generally, but if sample is very small, about 300 scans. 32 scans for pure samples, large quantity.

8. 64 scans

9. 100-300 depending on the sample

What resolution do you use?

1. 8 cm-1
2. 2 or 4 cm-1

3. 4 cm-1
4. 8 cm-1
5. 4 cm-1
6. 2 cm-1
7. 4 cm-1; sometimes 8 cm-1
8. 8 cm-1 and 4 cm-1
9. 4 cm-1

What libraries do you have? Do you like them?

1. Mattson ICON and KS (free with Win First software, 600 spectra)

2. Analect Polymers, Plasticizers, Surfactants, Reagents, Inorganic and minerals, Art materials, Ga. State Crime Lab, EPA vapor Phase, plus several I have created using reference material - resins, oils, fibers, polymers, dyes, miscellaneous materials.

3. Several Sadder libraries on disk, Hummel and Scholl and Coatings Society books are the most useful.

4. Gettens Raw Materials and Pigments we've run ourselves. Most useful Hummel Polymer - OK Sigma natural pigments - OK USGS minerals - OK. I rarely get hits from the commercial libraries, except the Hummel polymer.

5. We don't use the computerized libraries, just published atlases and our own reference spectra.

6. 225 representative spectra of the Sadtler library which are in the software of the instrument; Sadder IR Mentor Version 1.0

7. Hummel Polymer, Aldrich Solvent, Commercial Materials (this library is difficult to use when it lists only trade names.), Industrial Coatings

8. Art Conservation, reagents, Oils, Resins, Polymers, Fibers, Dyes, Ambers

9. Several Nicolet libraries and several Sadtler libraries. They are all valuable.

Do you have access to Internet? Would you like a spectral library on-line?

1. Bad access. Probably not.

2. No access to Internet.

3. Yes. Yes

4. Yes. Yes.

5. No access.

6. No. Yes.

7. No. Yes.

8. Yes in a near future. Yes we would like to see a spectral library on line if the access will not be very expensive.

Other comments or questions that you would like to see discussed.

1. I am particularly interested in learning to do thin sectioning of multi-layer materials. Would like to see this demonstrated and have some hands-on practice.

2. Methods for the deconvolution of bands; methods to subtract, in particular spectral range, one spectrum from another differing only for the presence of a component (for example plaster + pigment minus plaster) when analyzing in reflectance mode (diffuse or micro) where the Absorbance units are in Kubelka-Munk.