

Webinar Transcript

Techniques for Obtaining Infrared Spectra

Access a recording of this webinar and download the slides.



www.mccrone.com/webinar-archives

Introduction

Hello, my name is Charles Zona, and I'd like to thank everyone for attending today's webinar. Our topic today is Techniques for Obtaining Infrared Spectra, and it will be presented by Andrea Champagne, Mary Stellmack and Kate Martin. But before we get started, I would like to talk a little bit about The McCrone Group, for those of you who aren't familiar with our services.

The McCrone Group consists of three divisions: Hooke College of Applied Sciences, McCrone Microscopes & Accessories, and McCrone Associates.

Hooke College of Applied Sciences—our education division—offers professional development courses in materials science, including instrument-based courses such as polarized light microscopy, scanning and transmission electron microscopy, micro-FTIR—which we're going to hear a little bit about today; and micro-Raman.

We also offer special topics courses, such as gunshot residue analysis, specimen isolation and preparation, fiber identification, pharmaceutical contaminants, and a whole host of other courses.

PRESENTER: Andrea B. Champagne

Andrea is a research chemist for McCrone Associates, Inc. (MA) who specializes in small particle analysis utilizing light microscopy, micro-Fourier transform infrared spectroscopy, and gas and liquid chromatography. Prior to joining MA, Andrea worked as a method development chemist in the nutraceutical industry, and worked as a forensic scientist specializing in controlled substance analysis, fire debris analysis, and clandestine laboratory response.



Mary Stellmack and Kate Martin, Ph.D., both senior research chemists at MA, joined Andrea for the Q&A session.

Hooke College is also an academic partner with North Central College and Concordia University, offering 3+1 programs which result in a bachelor's degree in applied microscopy, or chemical microscopy.

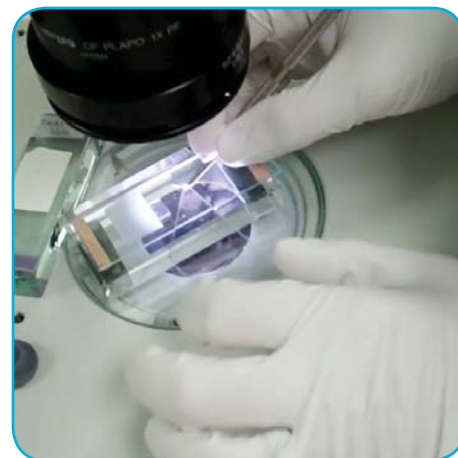
For a complete listing of our professional development courses, or more information about our 3+1 programs, please visit our website.

McCrone Microscopes & Accessories is our instrument sales group. They are a preferred dealer for Nikon industrial microscopes and metrology systems. They're also the national dealer for the JEOL NeoScope benchtop SEM, and are a preferred national dealer for Linkam thermal stage systems, along with many other microscopy-based laboratory tools and supplies.

McCrone Associates, the analytical division of The McCrone Group, is an A2LA accredited laboratory providing materials analysis services, specializing in solving industry's toughest problems, using light and electron microscopy, X-ray diffraction, Raman, FTIR, and other cutting-edge techniques and applications.

For more information on any of the three divisions of The McCrone Group, please visit The McCrone Group website.

And now I'd like to introduce today's presenters: Andrea Champagne is a research chemist at McCrone Associates; also joining Andrea today are Mary Stellmack and Kate Martin, both are senior research chemists at McCrone Associates. And together, our presenters have over 65 years of combined industry experience.



Watch Kristen Wiley demonstrate sample preparation for this IR webinar. View the webinar "Preparing Polymer Samples for Microspectroscopy" at www.mccrone.com/webinar-archives.

Mary and Kate are also instructors for Hooke College of Applied Sciences, where they co-instruct our upcoming [micro-FTIR course](#), and for those of you interested in attending that course, it will be offered November 3rd through 5th.

We're also holding an FTIR [Spectral Interpretation](#) course this year, and that's going to be taught by Gretchen Shearer.

That course will be held September 22nd through the 24th, and for more information about these courses, please visit the [Hooke College website](#).

At the end of the webinar, Andrea, Mary and Kate will field questions from the audience, and you can type your questions into the questions box.

For those of you who attended the previous webinar on the [Preparation of Polymer Samples for Microspectroscopy](#), this is a continuation of that webinar.

Today's presenters will be talking about the spectra collected from the samples that were discussed in the previous webinar by Kristen Wiley and Heidi Talesky. Kristen is also here with us to field any sample prep questions that you may have.

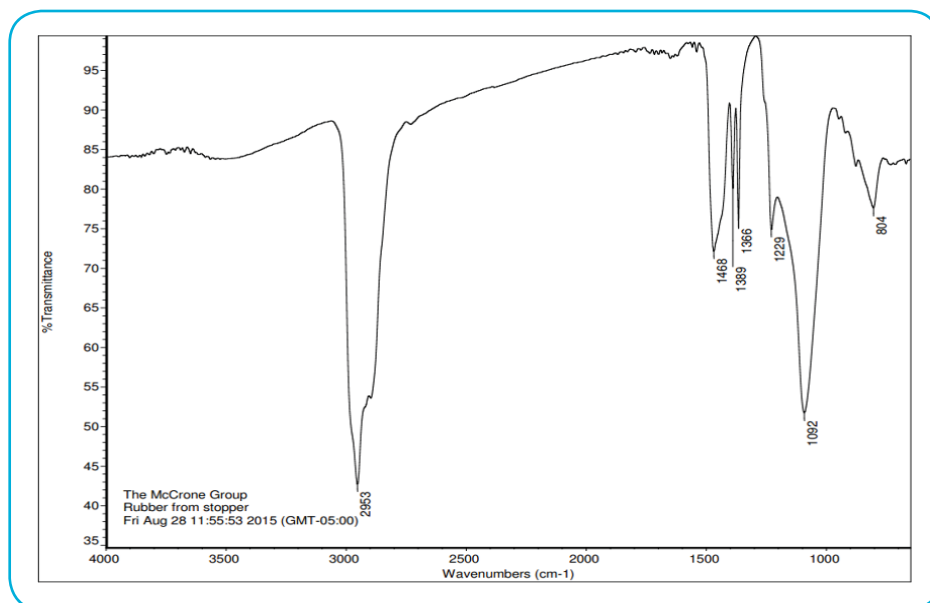
Today's webinar is being recorded, and will be available on The McCrone Group website under the webinars tab.

And now I will hand the program over to Andrea.

Infrared Spectroscopy

Thank you Chuck for such a great introduction. Good afternoon everyone. I am so pleased you chose to join us today while we discuss infrared microspectroscopy. We have only a short time so I will dive right in.

The first question you might ask is why infrared spectroscopy? Well, IR is a great identification technique. It can be used with a lot of different sample types including liquids, solids, powders, whether they're crystalline or non-crystalline, and even fibers.



Spectrum from the grey elastomeric material.

At McCrone we often use IR for the identification of unknown particulate contamination.

One of the strengths of infrared is that it can be applied to microscopic size samples. Using an infrared spectrometer – with a microscope accessory isolated particles as small as 20 microns can be analyzed while still maintaining good spatial resolution and low noise.

So how would you proceed if you want to analyze small particle contaminants? Let's use a case study example typical of some of the samples received here at McCrone Associates. This sample consists of a dark particle found floating in a liquid. The client has requested that we identify what the particle is so that they can try to narrow down the possible sources of this contamination.

The first step to any analysis is to assess the sample. For small particulates the first stage is always examination under a stereomicroscope. This allows us to get a better visual assessment of the sample. What does it look like? What kinds of physical characteristics are present? Is the particle hard? Is it brittle? Does it spread out and stay spread out, or does it stretch and then retract – like an elastic? Does it look metallic? All of this information will guide our analysis throughout.

When we examine this particle under magnification we see that it is not a homogeneous sample. We have a clear coating over a gray elastomeric core material. We are going to sample both portions and prepare them for infrared analysis. The samples will need to be pressed out on an IR transparent substrate. In last week's webinar, Kristen Wiley prepared both of these samples for us. She used a glass wedge to press out the clear sample and used a micro coverglass and carbide scribe to press out the elastomeric sample. Her webinar is archived on our website if you would like to see the preparation in detail.

After we have obtained our spectra we can see that the two portions of the particle provide very different infrared spectra. If we take a closer look we can confirm that no peaks from one spectrum are present in the other. So the two components were cleanly separated. So we can go ahead and start working to identify each part.

Let's look at the spectra of the clear coating first. A quick visual scan tells us that this is a good quality spectrum. There is very little noise, despite how small that sample was, the baseline is flat, none of the peaks are bottoming out and we are not seeing large interferences from water or carbon dioxide. Good quality spectra are easier to interpret and analyze.

Infrared spectral interpretation is based on the principle that molecular groups absorb infrared light at specific and predictable energies. So the frequencies (or wavenumbers) of the bands in a spectrum can be related to the presence of certain functional groups. This predictability makes spectral searching against libraries or databases very simple. Better quality spectra are easier to search and provide more reliable matches.

There are a multitude of commercial libraries available. A general search against the database is not a bad start for identifying a sample. Even if the library does not produce a perfect match, the spectra provided may assist in classifying the sample, giving you a good start to subsequent interpretation. If we search a polymer library against the clear coating spectra we find a very nice match to a fluorinated polymer: ethylene-tetrafluoroethylene. We do see an extra peak at approximately 1734 wavenumbers. This is the stretching region for carbonyl groups and may indicate some modifications to the polymer, or perhaps an additional component.

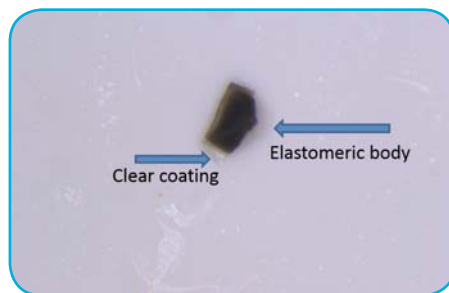
So now that we know what our clear coating is, let's consider our elastomeric core material. Once again a quick visual assessment shows that this is a good quality spectrum. We have a little scatter evident from the lowered baseline at the higher frequencies. Scatter might be caused by the sample itself, or it may be an indicator that our sample was uneven or perhaps a little thick. That can be as common problem with transmission spectroscopy. Certain fillers, like carbon black can also cause scatter, and this would be inherent to the sample itself, and not the preparation. We have no indication of atmospheric water or carbon dioxide, and we have minimal noise. We could re-prepare the sample, or correct the baseline to even things out. And while severe scattering can interfere with spectral matching, what we're seeing here is mild scattering, and that doesn't interfere with the match. So we will go ahead and see what a database search tells us. Automated database searches will provide a list of possibilities. The highest ranked match shares the

most features in common with the questioned spectrum. It may not have every feature present. If we did a quick library search of the gray elastomeric core material we might find our best match is poly(isobutene). Bands due to poly(isobutene) are all present in the spectrum of our unknown, but we also have additional peaks that need to be identified.

Our initial search tells us that isobutene is present, and that makes sense, this is an elastomeric particle—we saw that when we prepared it, and isobutene is a common rubber. But that does not explain that broad peak at 1092 wavenumbers. This is where a good understanding of spectral interpretation becomes essential. This band may indicate the presence of an inorganic component, like silica, or perhaps a phosphate. We might not be able to make that determination solely from the IR, and complementary tests may be necessary to fully identify the material. We may send this on for SEM or EDS to get an elementary profile of what's present.

The best approach, of course, is to build a library of your own, based on those materials you most commonly see and work with. This will provide the most accurate information. And you can see, when we compared this sample to a library McCrone built of rubbers with common fillers, we had a beautiful match—isobutene rubber with a silica filler.

Just to wrap things up, we have determined that our particle (image below) is an isobutene rubber with silica filler and a fluorinated polymer coating. This gives the client a specific type of rubber to try and locate. They can now look at their manufacturing and shipping processes to see where their liquid sample may have come into contact with this coated rubber.



This has been a really simplified version of the application of infrared microspectroscopy and the approach to sample identification using database searches. We were unable, really, to discuss spectral interpretation, elucidating chemical information from the spectrum rather than using a database comparison. Spectral interpretation can't really be addressed in a short webinar, it is much better suited to a classroom setting.

I'm so glad you were able to join us today. Mary, Kate, Kristen, and I will open the floor for any questions.

Questions & Answers

CZ:

Thanks, Andrea. If anyone has any questions, please type them into the question box there, and we'll begin to answer them. Okay, it looks like we have some coming in here. First question: "Why did you use transmission IR rather than ATR?"

AC:

I'm very glad you asked this question. I didn't get a chance to talk about it too much in the webinar. ATR is another technique for obtaining your spectra, and it's very useful for deformable materials, like rubber; although it isn't so great when you have carbon-filled rubbers. Since this particle consisted of two phases, the coating and the core rubber, we still might need to make sure we have good separation. We'd either have to separate them and then run ATR, or make sure that when we pressed it out on ATR we had good clarity between those, so it was just as straightforward if we need to do it in transmission. Either technique would be applicable.

CZ:

What other tests would you use to confirm the presence of silica?

AC:

To confirm silica, I would send it on for EDS to confirm we had silica; silicon, the element, present. My colleagues are welcome to chime in with anything they might...

CZ:

They're giving us the thumbs up.

CZ:

Okay, it looks like we have kind of a two-part question here from Ken. "Is Hooke College's database available for sale?" Well, I don't think we...we don't have a Hooke College database, it's the McCrone Associates library, correct? And it's not for sale. And the other part to his question is, "if not, do you recommend a database that is for sale?"

AC:

There are a lot of commercial libraries, and really, some of them are sort of class-based: there are polymer libraries, there are inorganic libraries... I would recommend one that is in keeping with the sorts of samples that you see the most. That's going to be the most useful.

CZ:

Here's a question from James. "Can you speak to the use of ATR diamond compression cell and KBr, where you would use one or the other."

AC:

I'm going to defer this one to Kate, actually, we spent a lot of time talking about this and she gives a much more detailed answer.

KM:

I'm not sure if you're referring to KBr windows in a compression cell. Diamond, of course, is a much harder material—it's a lot more expensive than KBr, but you can exert a lot more pressure with diamond than you can with KBr. KBr is a crystal that will crack very easily under fairly modest pressure. We sometimes use barium fluoride windows with a compression cell, which are going to be sturdier than the KBr, but still not as sturdy as a diamond.

CZ:

Okay, thanks Kate. Let's see, what else do we have. From Kimberly, "Do you have a comparable ATR database?"

KM:

I believe there are some commercial ATR databases, and we have among our in-house libraries, we do have an ATR library as well. A lot of it is... check with your instrument vendor, and they can supply you with the commercial libraries that are that are available that work with your software.

CZ:

Thanks Kate. This one's from Tom: "Does the McCrone Atlas have this information in it that Ken requested?" I believe there is some of the IR information in some of the characterizations, but go ahead Kate.

KM:

The McCrone Atlas (of Microscopic Particles) does include some IR spectra, and some interpretation of those spectra, so the spectra are available for viewing within the Atlas. I don't believe that they can be downloaded from the Atlas, but I'm not sure of that. *Editor's Note: Particle Characterizations can be printed from the McCrone Atlas of Microscopic Particles by clicking the PRINT icon.*

CZ:

Question from Seema: "Can ATR be used for quantitative applications? How do you compare it to KBr techniques for the purpose?"

KM:

Well, IR, of course, is not generally the best for quantitative purposes unless you're using something like a fixed path link cell, like you would with some liquids. It can be good for some mixtures, where you've got... where you're comparing two components that are part of a mixture. An ATR can be used for quantitation, but you have to be very careful because, for example, if you change the refractive index of a material, for example, if you're changing the depth of penetration, or something of that kind. Or if you're changing the material in any way, you can change the intensity of the bands. So it really depends on the circumstances—you have to think carefully about whether the particular material will be apt for that.

MS:

I think it's also important to point out about the ATR technique, is that it's basically recording a spectrum of the material that's in contact with the crystal, and because of that, it's primarily obtaining data from the surface of your sample. And so, if your sample, for example, is like the one Andrea talked about with a coating, you want to be sure that you're looking at your sample to be sure that you're obtain-

ing spectra from each layer, if you have a multi-layered sample. So that's one important thing to keep in mind when you're doing ATR work.

CZ:

Thanks Mary. Question from Shelby: "What are the keys to interpretation of the peaks that do not show up with the best possible match?"

AC:

We're all kind of looking at each other for a minute... if this is in respect to the clear coated polymer sample, I would let Mary handle that answer.

MS:

Could you repeat the question Chuck?

CZ:

Sure. "What are the keys to interpretation of the peaks that do not show up with the best possible match?"

MS:

I assume we're talking about the extra carbonyl band in the spectrum of the rubber that Andrea showed, perhaps? Sometimes you can look at the frequencies of those bands and get an idea of what kind of compound they might be from. For example, the carbonyl band that Andrea saw in her sample could be due to perhaps an oil or some other...maybe an ester-based additive in the material. You can think about the environment that the sample was removed from and what other types of material the sample might have been in contact with.

You can also try maybe doing some solvent extractions with the particle to see if you can isolate that unidentified component and get an infrared spectrum of that material, as well, and that may help you to identify it.

CZ:

Thanks Mary. Okay, a question from Herant: "How would you handle spectra that contain tertiary or quaternary components, for example, if the liquid was a viscous medium that contained the samples?"

KM:

That's a good question, because quaternary and tertiary amines do not always give robust IR spectra that lets you identify them as quaternary and tertiary amines. So sometimes you have to use other information you know about the sample. That's one of the reasons we use the stereomicroscope first, because it helps us a lot. In terms of a viscous liquid, if you want to do your best matching, make sure you get a very good quality spectrum so that some subtleties in the spectra can also be matched up, you need to make sure that your library covers all of those possibilities that you might be interested in.

And sometimes, of course, you want to employ other techniques. You might want to do a pyrolysis measurement. Pyrolysis IR, pyrolysis GC, HPLC—or something like that, or some other mass spectrometry.

CZ:

Thanks Kate. A question from James: "If you were to examine a powder for homogeneity, would you consider using a pellet press to make it flat, or ATR; and if ATR, how do you address the limited sample size?"

AC:

If you're looking at a powder for homogeneity, obviously, looking at it under the 'scope first, you're going to be able to pick out the stuff that has some visual differences. ATR, pressed down on an inhomogeneous powder, you're going to see a lot of issues with consistent contact throughout the sample, and really, the only way to compensate for the fact that the ATR is only looking at a small part of that is to take multiple ATR scans from multiple places.

KM:

Yes, and I think furthermore, again, I like ATR in a lot of ways, but it has some limits, and one of them is that not all components in a mixture will make contact equally with the ATR crystal, and so you can sometimes get some self-selection in that regard. We encounter that on a few occasions with mixtures.

CZ:

Great. We have one from Jan: "I always start with micro-Raman. Is micro-IR your starting technique?"

KM:

For general identification, we have found that IR is better as a general identifier. Raman spectroscopy is less applicable to just generic unknowns; it's great for certain classes of materials such as pigments, many minerals, carbon compounds such as diamond and graphite, but as a very general identification tool, we like IR.

CZ:

Thanks Kate. Okay, I think that's it for the questions. Thank you very much for all of your questions, very interesting.

Conclusion

CZ:

I'd just like to talk about our next upcoming webinar *Thermal Characterization as Part of an Empirical Process for Developing Optimized Formulations and Lyophilization Cycles*. We're going into the freeze dry realm on that one. The presenters will be Dr. Jeff Schwegman from AB Biotech, and Ruben Nieblas from McCrone Microscopes & Accessories. Jeff and Ruben will also be teaching a course on that topic, it's a three-day course on *Lyophilization: Practical Applications Utilizing the Latest Equipment*. The course takes place November 3rd through the 5th at Hooke College of Applied Sciences, and for more information please visit our website.

And with that, I guess we'll wrap it up here and thank everybody again for attending, and hope to see you again for another webinar. Thank you.



Kate Martin uses Fourier transform infrared spectroscopy (FTIR) to analyze a sample.