

How to analyze polymer samples via FTIR specular reflectance spectroscopy

While single bounce ATR spectroscopy is one of the most popular techniques for material identification, there are still cases where the specular reflectance sampling technique may be necessary. In a specular reflectance measurement, the infrared light from the spectrometer is reflected directly off the sample, much like light hitting a mirror. Because it can be a useful sampling technique several ATR accessories can be configured to acquire specular reflectance spectra. Figure 1 shows the Thermo Scientific™ Nicolet™ Summit FTIR Spectrometer with the Everest ATR Accessory, which can be configured with the specular reflection attachment.



Figure 1. Thermo Scientific Nicolet Summit FTIR Spectrometer with the Everest ATR accessory configured for Specular Reflectance.

Other dedicated reflectance accessories make it possible to measure very large samples, like paintings, without any contact between the instrument and the art work. One of the drawbacks of the specular reflectance sampling technique are the distorted spectra that can result from anomalous dispersion, or a scattering effect as the light interacts with the sample. The distorted spectra make it difficult to get reasonable matches with most spectral libraries, which are usually collected in transmission.

Figure 2 shows the specular reflectance spectrum acquired with the Everest accessory from a white piece of smooth, white plastic. You can see that the collected spectrum (red) doesn't look much like our best match value (blue). In fact, the Thermo Scientific™ OMNIC™ Paradigm Software calculates a match value of only 52.5, which is low and suggest that the two spectra are from different materials.

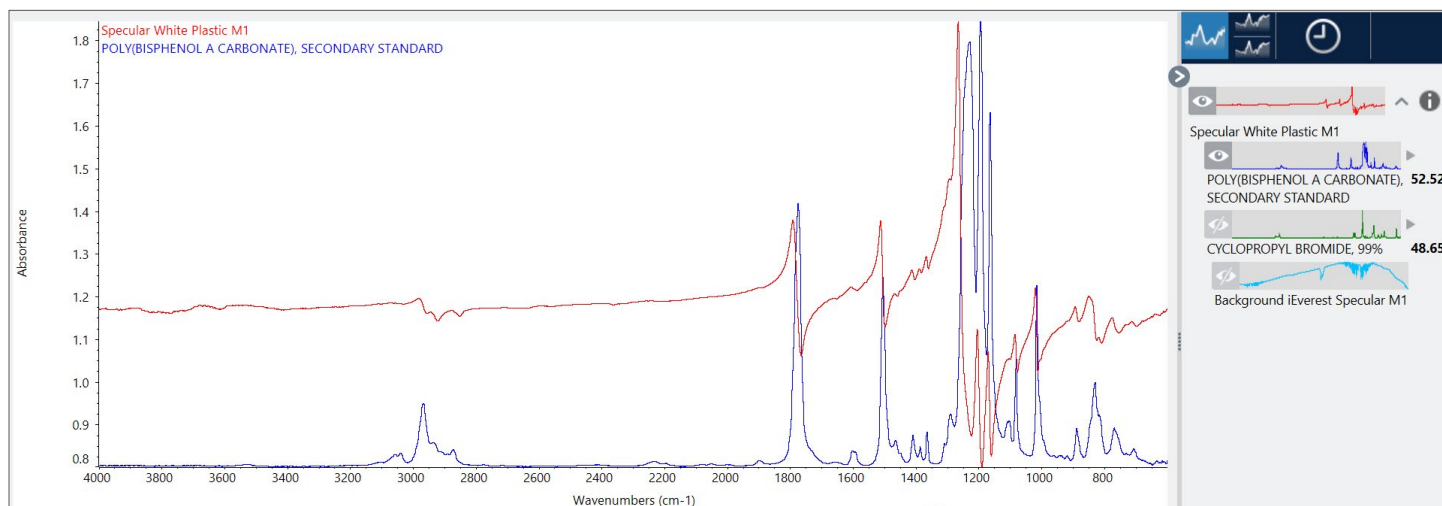


Figure 2. Specular reflectance spectrum (red) from a white piece of plastic and the library spectrum from the best search result.

The Kramers-Kronig correction in the OMNIC Paradigm Software mathematically eliminates much of the spectral distortion resulting from the specular reflectance measurement and creates a spectrum that looks as if it were collected in transmission.

Figure 3 shows the corrected spectrum (Red) overlaid with the top match value (Purple). Once the Kramers-Kronig is applied, the match value increases dramatically to 93, indicating that the sample is most likely a polycarbonate plastic.

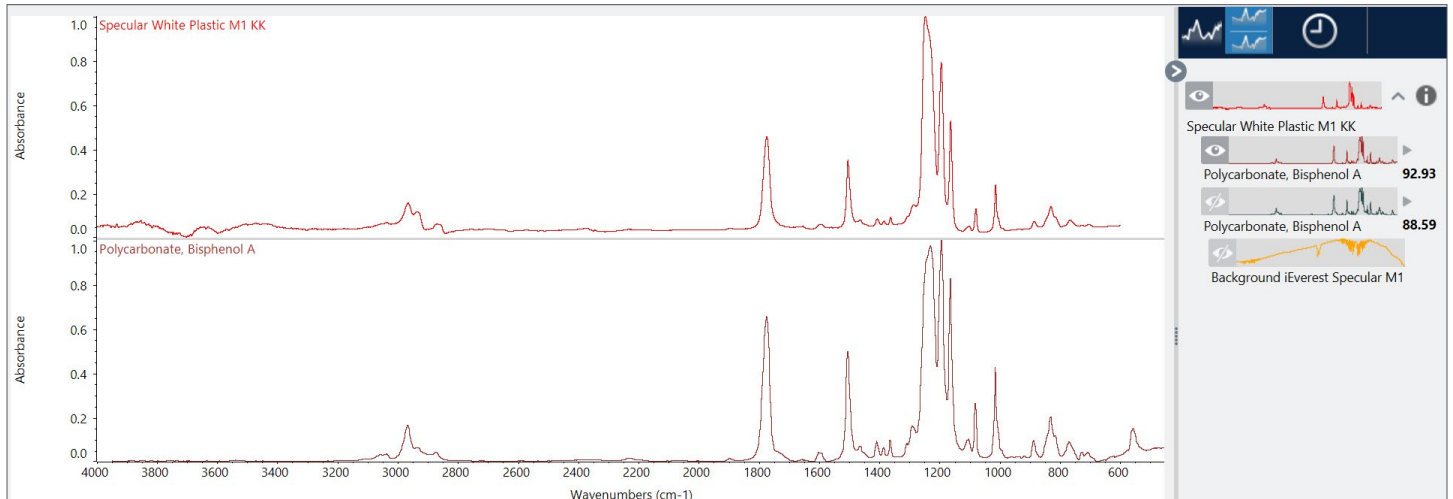


Figure 3. Spectrum after Kramers-Kronig corrections and library spectrum from the best search result.

To speed up the analysis in the future, the OMNIC Paradigm Software can automatically create a workflow, similar to a *Standard Operating Procedure*, to ensure consistent results from one analyst to the next. On the right hand of Figure 4, you can see the actual processing

steps completed for the spectral reflectance spectrum, including the Kramers-Kronig correction. Clicking on the Create Workflow button at the bottom right hand side of Figure 4 will automatically create the workflow for future use (Figure 5).

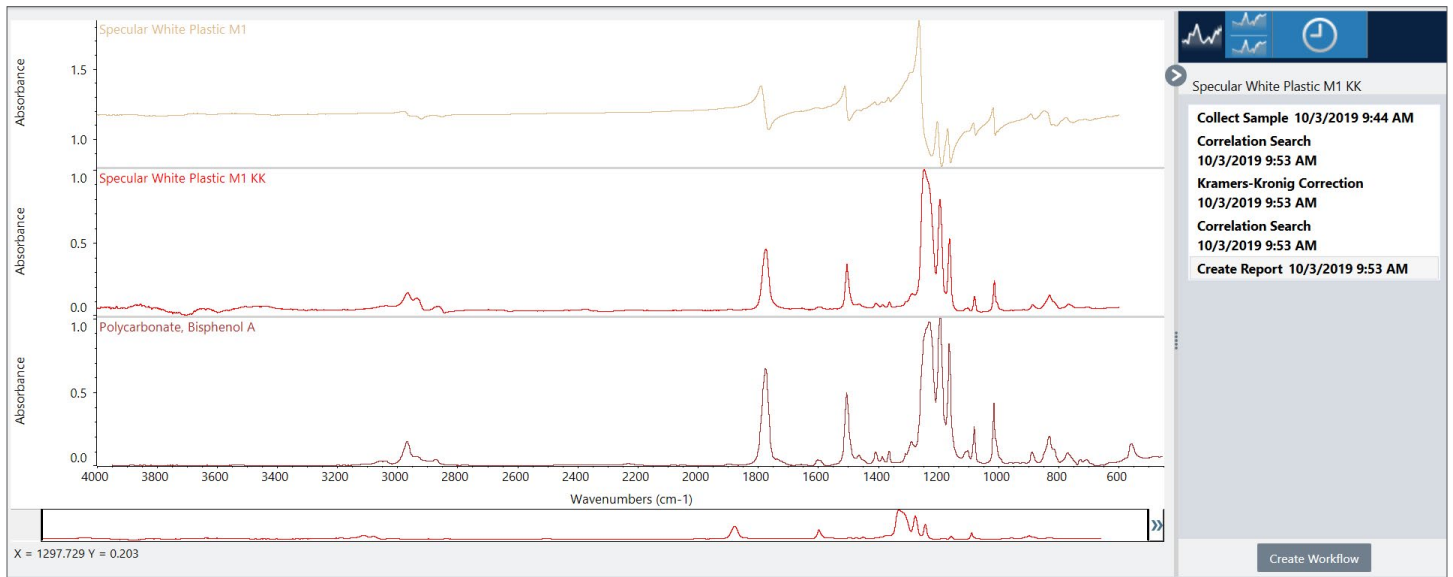


Figure 4. Comparison of the original spectrum from the white plastic, the KK corrected spectrum and the best library match.

The OMNIC Paradigm software automatically creates a fully functional workflow shown below.

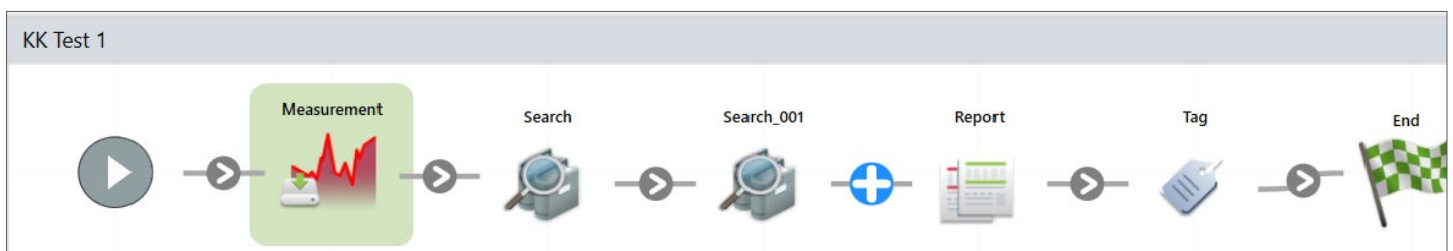


Figure 5. The workflow resulting from clicking on "Create Workflow" button.

In many cases the lab manager may want to add additional functionality to the workflow to make it better. Figure 6 shows a more robust workflow with a few features, or Tiles, added:

1. **Instruction tile** – Operator is asked to enter sample information, such as the name and batch number
2. **Save Results tile** – Archives the spectra and results to a specific file location
3. **Repeat tile** – Adds a loop so that multiple samples can be analyzed at once

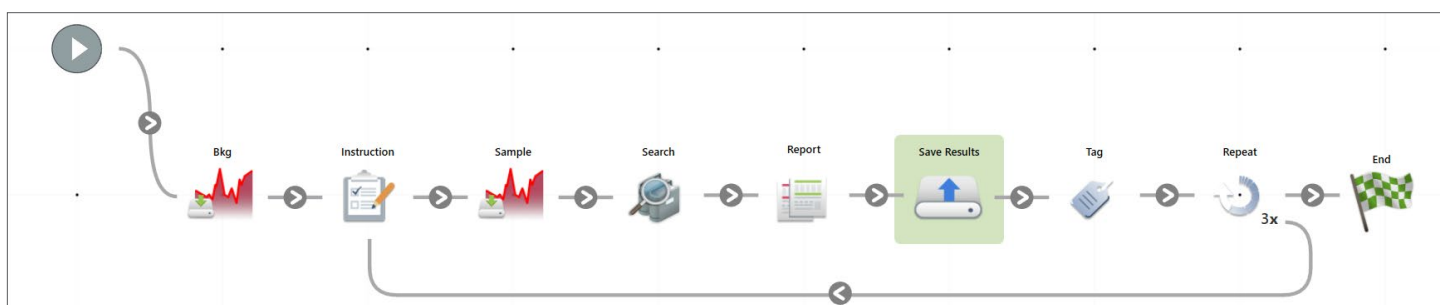


Figure 6. Final Workflow with enhancements.

OMNIC Paradigm Software includes several useful features to make analysis easy. The Kramers-Kronig correction is used to remove spectral distortions from diffuse reflectance measurements. Applying the Kramers-Kronig correction significantly improves the library match value from around 50 to greater than 90. The process of collecting a spectrum, applying the Kramers-Kronig correction, and successfully searching a library can be automated into a Workflow, which makes the steps easy for any scientist or technician in the lab.