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Identification of Common Polymers using ATR-FTIR and Raman in an Undergraduate Teaching Laboratory

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Introduction

Plastics and polymers are used across a multitude of disciplines including construction, electronics, healthcare, and consumer goods because of their strength, light weight, and versatility. However, plastics pose a threat to the environment in their manufacturing, their end-of-use phases and can contaminate food and drinking water in the form of microplastics. Fast and reliable ways of identifying plastics are becoming increasingly important in analytical science. Vibrational techniques such as Raman and FTIR spectroscopy are well suited for polymer identification because they are nondestructive, require little sample preparation, and their complementary spectra allows for a broad range of chemicals or substances to be identified.

A bond (or vibrational mode) is IR active when absorption of infrared light causes a change in dipole moment. This absorption of light occurs at characteristic frequencies (typically reported in wavenumbers, cm^{-1}) which correspond to the energy required to excite a vibrational mode from a lower to a higher vibrational energy level. Every IR-active functional group gives rise to characteristic absorption bands or peaks within a range of wavenumbers which can be used for identification. Raman spectroscopy, on the other hand, is an inelastic light scattering phenomenon. In this technique, a laser is used to impinge light on a sample, and most of that light is scattered with the same energy as the incident light (this is called Rayleigh scattering). However, a small fraction of this light is scattered at a different energy than the incident radiation due to interactions with molecular vibrations that involve a change in polarizability. The difference in energy between the incident light and scattered light, known as the Raman shift, results in a characteristic spectrum that can be used for compound identification. Using these FTIR and Raman techniques together provides a more complete picture of a sample's identity because the two techniques provide complementary information about molecular vibrations and functional groups.

This application note highlights JASCO's vibrational spectroscopy instruments as integral tools for identifying common polymer types such as polyethylene terephthalate (PET), polyamide (PA), polystyrene (PS), polypropylene (PP), and polyvinyl chloride (PVC) in an undergraduate analytical chemistry laboratory. Raman spectra were collected using the JASCO PR-1w Palmtop Raman spectrometer, and the infrared spectra were collected using the JASCO FT/IR-4X spectrometer with the ATR PRO ONE 4X VIEW accessory. Additionally, this application note highlights the ways in which students can use ATR-FTIR and Raman spectroscopy instruments together to better understand the complementary information obtained from these techniques. The spectral variability between different samples of the same type of polymer, as well as the importance of fluorescence correction in Raman spectra are explored.

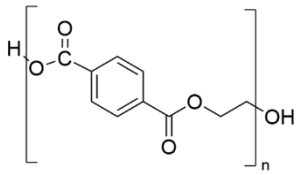
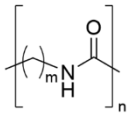
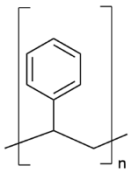
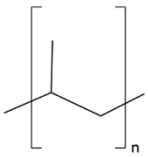
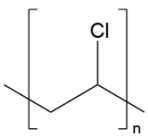
Keywords

Raman spectroscopy, ATR-FT/IR; polymer identification, plastics analysis, undergraduate instruction

Methods

Samples of different plastics were collected from consumer sources. Plastic identification was based on the number inside the chasing arrows (recycling symbol) or though familiarity with the item (sheer tights). These samples are a very small subset of available consumer plastics. Instructors are encouraged to explore different samples or have students bring in their own samples. Table 1 shows the types of plastics, their monomeric structure, and the consumer sources for each sample used in this experiment.

Table 1. *Types of plastics, the structure of the monomer, and sources of samples used in this experiment*

Plastic	Monomer	Sample Sources
Polyethylene terephthalate (PET), recycling symbol 1		Apple juice bottle Clear drinking cup Clear sushi tray Electrolyte drink bottle
Polyamide (PA)		Sheer tights (nylons)
Polystyrene (PS), recycling symbol 6		Yogurt cup
Polypropylene (PP), recycling symbol 5		Centrifuge tube
Polyvinylchloride (PVC), recycling symbol 3		Packaging tray

The PR-1w Palmtop Raman spectrometer equipped with a 785 nm laser and a 200-3000 cm^{-1} spectral range and a spectral resolution of 3 cm^{-1} was used in this experiment to obtain Raman spectra.

Laser power was toggled between low (5 mW), medium (25 mW), and high (50 mW) as necessary to obtain high-quality spectra and avoid sample destruction. A 1 second exposure time and 10 accumulations were utilized. Fluorescence correction was toggled on and off in the software as described below.

The FT/IR-4X spectrometer with the ATR PRO ONE 4X VIEW accessory was employed to obtain IR spectra. The spectral range used was 400-4000 cm^{-1} with a 4 cm^{-1} resolution. Sixteen accumulations were acquired for each spectrum and were collected in absorbance mode for easy comparison to the Raman spectra.

Raman and FTIR spectra were normalized to 100 for easy comparison. When multiple spectra are shown together in the figures below, a small vertical offset from the baseline is used to ease interpretation.

Experimental Suggestions for Undergraduate Teaching Lab

Exploration of the complementary nature of Raman and FTIR

Students should collect spectra from each type of plastic provided on both the FTIR and Raman spectrometers and determine the functional groups present based on the observed spectral peaks. A chart that correlates functional groups with spectral frequency can aid in identification. Students should identify which vibrational modes are Raman active, which are IR active, and which are both Raman and IR active.

Observation of the effects of fluorescence correction on Raman spectra

The effect of background interference from fluorescence in the Raman spectra can be explored using different plastic samples. Some plastic samples (such as PA) have stronger fluorescence background when excited at 785 nm than other plastics (such as PET). The ability to easily toggle the fluorescence correction on and off in the software allows for observation of the importance and effects of the correction. Students should obtain Raman spectra of various plastic samples with and without fluorescence correction and compare the resulting spectra.

Observation of the variability of Raman and FTIR spectra for different samples

Different sources of a single type of plastic (such as PET) must be obtained. Students collect spectra of each sample on both the FTIR and Raman spectrometers. Students should analyze the variability of the Raman spectra and the FTIR spectra for each sample by overlaying the spectra in the provided software.

Results

Exploration of the complementary nature of Raman and FTIR

To highlight how Raman and IR spectra can complement each other, data was collected for five different polymer types and overlaid for comparison. Figures 1-5 show the overlay of Raman and IR spectra for PET, PA, PS, PP, and PVC, respectively.

In each figure, the strongest peaks are highlighted.

In Figure 1, the PET sample has characteristic C=O ester stretches at the 1730 cm^{-1} and 1712 cm^{-1} in the Raman and FTIR spectra, respectively. The C=C aromatic stretch appears at 1617 cm^{-1} in the Raman spectrum. The FTIR spectrum shows strong peaks at 1241 cm^{-1}, 1094 cm^{-1}, and 723 cm^{-1} corresponding to the C-C-O ester stretch, O-C-C ester stretch, and out-of-plane IR peak likely from the linking ester backbone bonds along with a Raman active aromatic band at 1292 cm^{-1}.

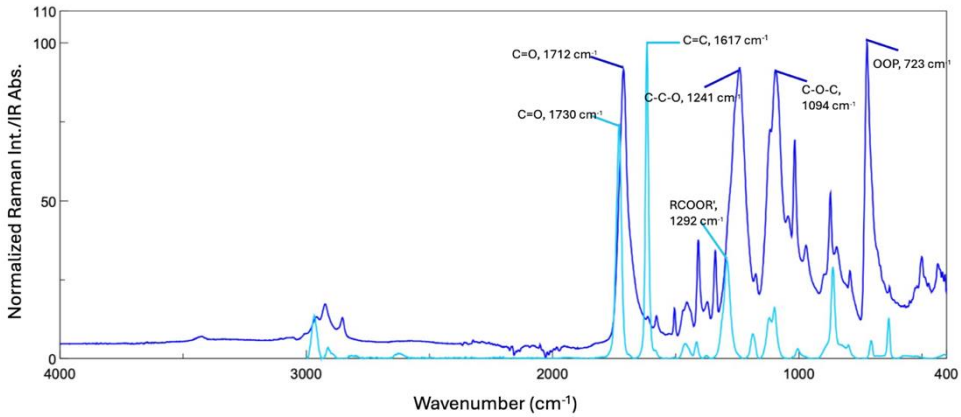


Fig. 1 *PET polymer from apple juice bottle, light blue = Raman, dark blue peaks = IR, small vertical offset for ease of interpretation*

Figure 2 shows the spectra of PA obtained from a sample of sheer tights. PA shows a strong band at 3296 cm^{-1} in the IR spectrum which arises from the N-H bond stretch. Both Raman and FTIR spectra show C=O carbonyl (amide) peaks at 1637 cm^{-1} and 1631 cm^{-1}, respectively. The FTIR spectrum also shows the N-H amide stretch at 1531 cm^{-1} which is not present in the Raman spectrum. A stronger band appears at 2903 cm^{-1} in the Raman spectrum, corresponding to aliphatic methylene group. The Palmtop spectral range extends from 3000 to 200 cm^{-1} which does not allow for observation of the N-H bond stretch in PA, however, characteristic peaks in the fingerprint region allows for easy identification of this polymer.

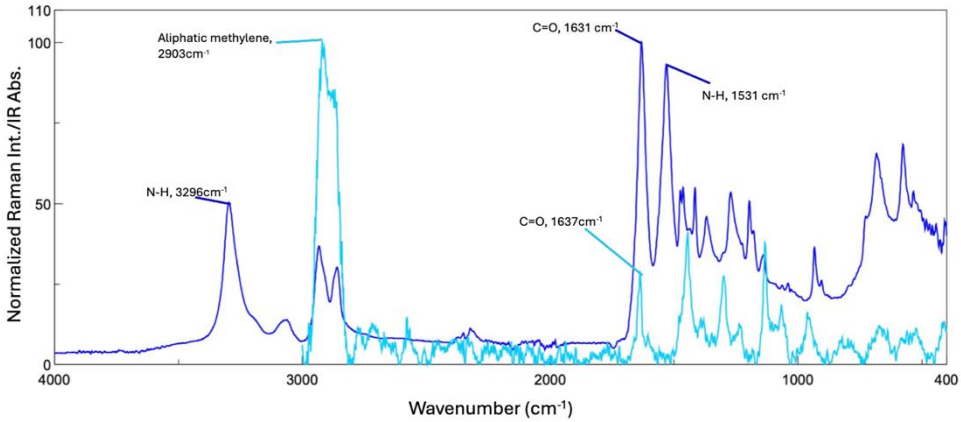


Fig. 2 *PA polymer from sheer tights, light blue = Raman, dark blue peaks = IR, small vertical offset for ease of interpretation*

Figure 3 shows the FTIR and Raman spectra from the yogurt cup comprised of PS. In both the Raman and FTIR spectra, there is a peak around 1600 cm^{-1} that arises from C=C stretching of the benzene ring. In the Raman spectrum, a peak is present at 1004 cm^{-1} and corresponds to the breathing mode of the benzene ring; this peak is not present in the FTIR spectrum. Bands resulting from out-of-plane bending modes of the benzene ring are present at 695 cm^{-1} and 749 cm^{-1} in the FTIR spectrum.

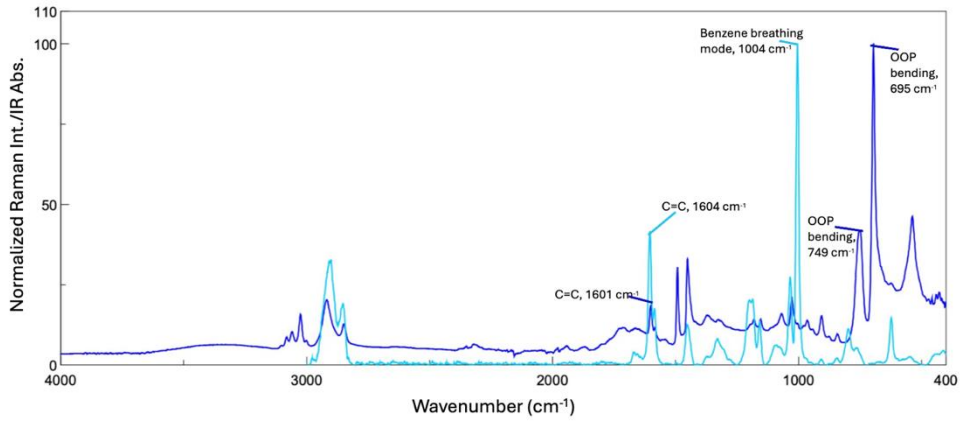


Fig. 3 *PS polymer from yogurt cup, light blue = Raman, dark blue peaks = IR, small vertical offset for ease of interpretation*

In Figure 4, characteristic C-H stretching bands are present in both the FTIR and Raman spectra around 2750-3000 cm^{-1} for the PP centrifuge tube. While there are bands present in both the Raman and FTIR fingerprint region, the strongest bands arise from $(CH_3)_2$ antisymmetric deformation at 1456 cm^{-1} and $(CH_3)_2$ symmetric deformation at 1376 cm^{-1} in the FTIR spectrum.

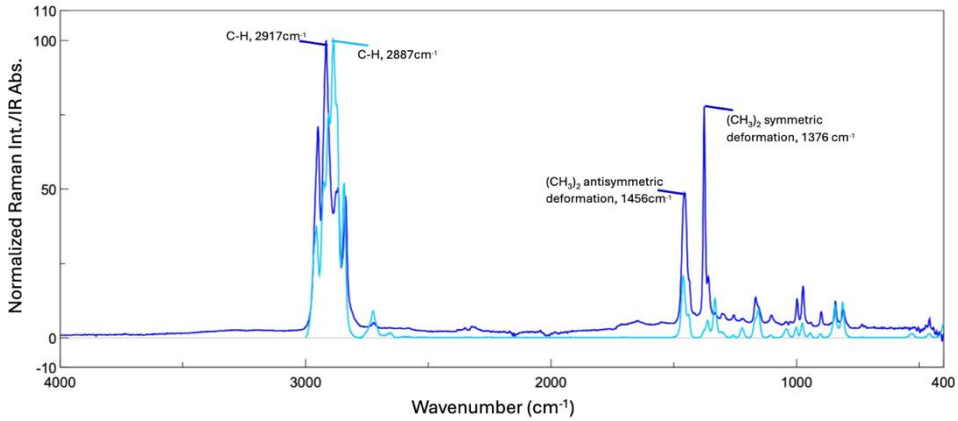


Fig. 4 *PP polymer from centrifuge tube, light blue = Raman, dark blue peaks = IR, small vertical offset for ease of interpretation*

For PVC, a strong Raman peak occurs at 2916 cm^{-1} resulting from the C-H stretching and is nearly absent in the FTIR spectrum in Figure 5. The FTIR band at 612 cm^{-1} is indicative of the characteristic C-Cl stretching vibration. In the Raman Spectrum, the band at 645 cm^{-1} results from the C-Cl stretch. An FTIR peak at 1731 cm^{-1} highlights a carbonyl functional group that does not show up in the Raman spectrum and is likely the result of an additive as pure PVC does not contain a carbonyl group.

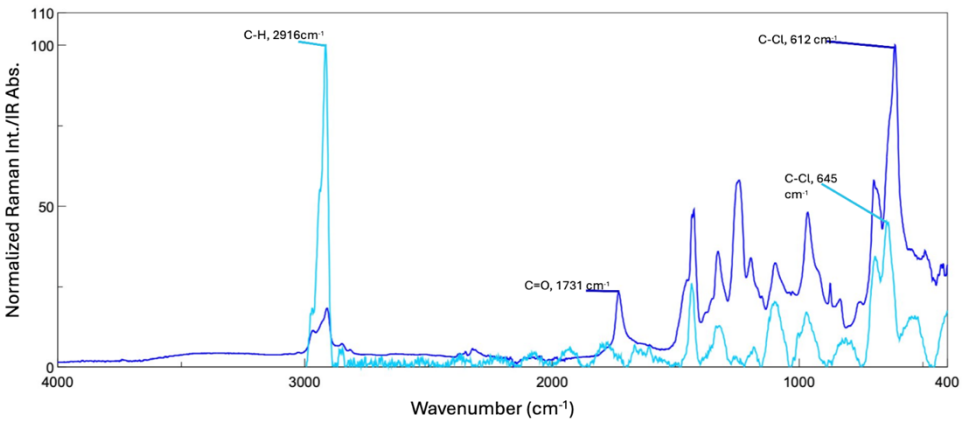


Fig. 5 *PVC polymer from large tray, light blue = Raman, dark blue peaks = IR, small vertical offset for ease of interpretation*

As shown in figures 1-5, some vibrational modes are clearly active in both Raman and FTIR. However, other bands may exist only on the Raman spectrum and not on the FTIR and vice versa. The ability to use unique Raman or FTIR peaks simplifies polymer identification and provides a higher level of confidence.

Observation of the effects of fluorescence correction on Raman spectra

Background interference from fluorescence can be a hinderance to obtaining quality Raman spectra.

A key feature of the JASCO PR-1w Raman Spectrometer is the ability to adjust for fluorescence by toggling the fluorescence correction (FC) option on or off in the instrument parameters in the software. This easy-to-use feature significantly improves spectral quality by mathematically filtering out background fluorescence for highly fluorescent samples. Figure 6 highlights the spectrometers FC feature when analyzing a PET apple juice bottle. The uncorrected light blue spectrum has broad and intense fluorescence background and can hide important peaks needed for identification. When FC is applied, the dark blue spectrum illustrates how the emission background is removed. The baseline of the spectrum is approximately zero intensity. This allows for observation of distinct peaks and easier interpretation. Fluorescence correction can be applied to any sample that has a high fluorescence background to assist in optimizing spectral quality.

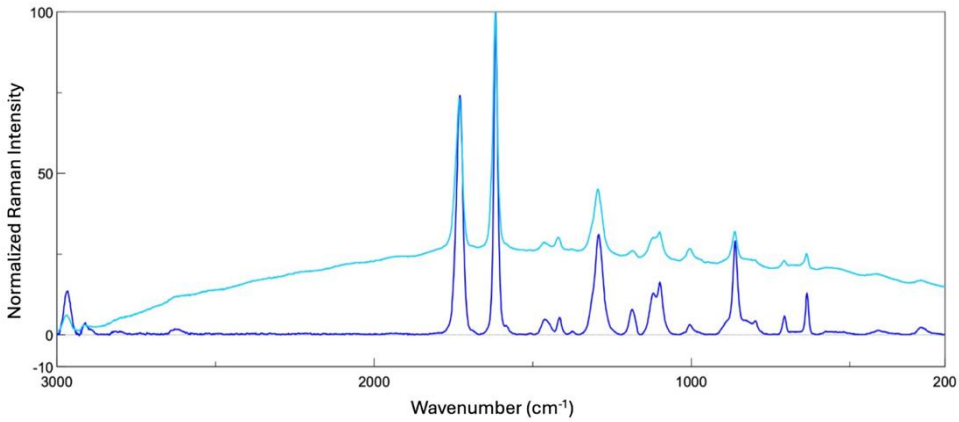


Fig. 6 *Positive effect of fluorescence correction in Raman spectroscopy for the PET apple juice bottle; dark blue = fluorescence correction, light blue = no fluorescence correction*

Although, fluorescence correction may be used as needed, there still exist limitations. If a sample fluoresces strongly, that signal can overwhelm the detector and drown out weaker Raman scattering before the instrument can record useful data. After FC, Raman peaks may stay lost in the noise and reducing the fluorescence baseline may not help to resolve those peaks and offer little additional information. With samples that fluoresce less, FC may still improve spectral data even if the correction is not perfect. Figure 7 contains the corrected and uncorrected spectrum of PA (sheer tights). In Figure 7, the correction is uneven across the spectral range and improves one side more than the other. This is especially evident in the 400-1000 cm^{-1} region where the bands become more prominent after correction. especially when combined with FTIR to aid in the identification of an unknown sample.

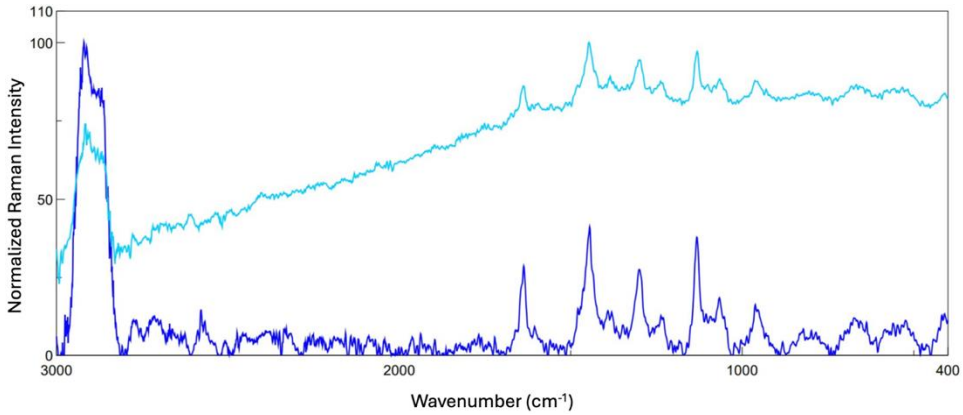


Fig. 7 *(above): Limitations of fluorescence correction in Raman spectroscopy for the PA sheer tights; dark blue = fluorescence correction, light blue = no fluorescence correction*

Observation of the variability of Raman and FTIR spectra for different samples

Three PET samples were obtained from different sources including a clear drinking cup, an electrolyte drink bottle, and a clear tray from a sushi container. Each sample was analyzed using Raman spectroscopy (Figure 8) and ATR-FTIR spectroscopy (Figure 9) to examine the variability among different PET samples due to manufacturing differences such as chemical additives.

Both the Raman and FTIR spectra for the three samples of PET from different sources have good general agreement and show the characteristic ester and aromatic stretches as described above. In both the Raman spectrum in Figure 8 and the FTIR spectrum in Figure 9, a noticeable difference in intensity occurs at the band near 1000 cm^{-1}, where the electrolyte drink bottle sample has a more intense peak with a slightly different peak shape than the other samples. This band likely corresponds to a C-O stretch. The variability in the FTIR and Raman spectra may be indicative of the presence of chemical additives to the polymer as a result of different manufacturers. These results highlight the ability of the JASCO Raman and FTIR systems to confirm identity while being sensitive enough to detect subtle differences in composition.

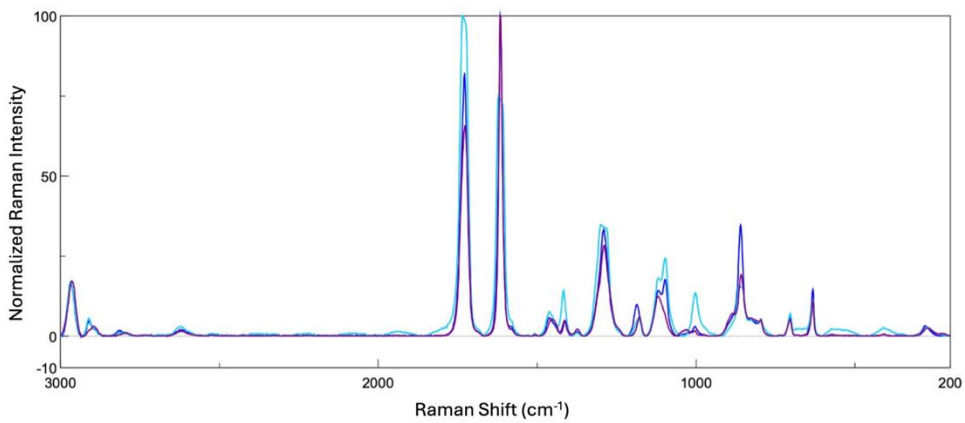


Fig. 8 *Raman spectra of PET samples from three sources; dark blue = clear drinking cup, light blue = Electrolyte drink bottle, maroon = clear tray*

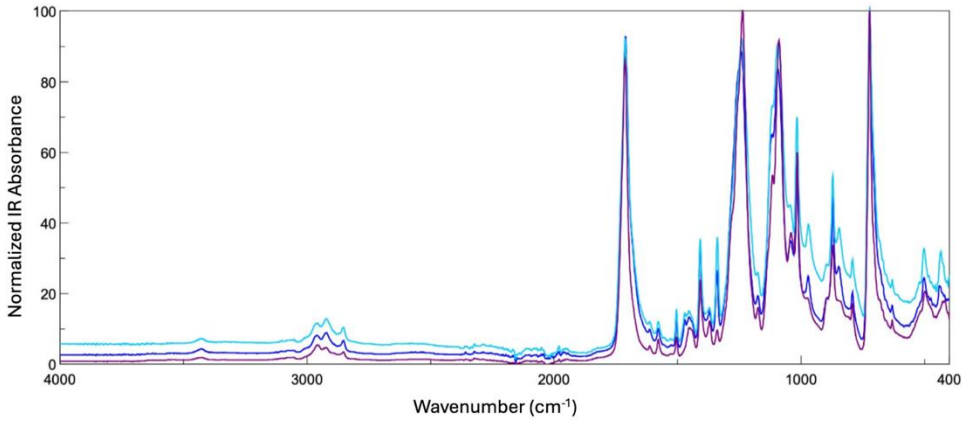


Fig. 9 *ATR-FTIR spectra of PET samples from three sources; dark blue = clear drinking cup, light blue = Electrolyte drink bottle, maroon = clear tray, small vertical offset for ease of interpretation*

Conclusion

This application note demonstrates the way in which undergraduate chemistry laboratory students can explore the complementary nature of Raman and ATR-FT/IR instrumentation and identify polymers using these two spectroscopic techniques. The sensitivity of the instruments can highlight the variability due to different chemical additives in plastics. The easy to turn on and off fluorescence correction feature on the Raman PR-1w Palmtop spectrometer is a great teaching tool to illustrate the importance of the properly using and understanding data processing features to allow the user to obtain high-quality spectra of highly fluorescent compounds.

About the Authors

Matthias Sanow is a current senior in the Department of Chemistry and Biochemistry at The Ohio State University. He is working towards his Bachelor of Science in chemistry with a minor in pharmaceutical sciences. His research focuses on FT-IR and Raman Spectroscopy. He is excited to continue working with JASCO.

Nicole Karn received her Ph.D. in analytical chemistry from Ohio State University. She is currently a Professor of Teaching at Ohio State University in the Department of Chemistry and Biochemistry. Her research centers on understanding the student experience in general chemistry and developing innovative analytical chemistry laboratory exercises.

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