Analysis of a Mixture With the Select-a-Path™

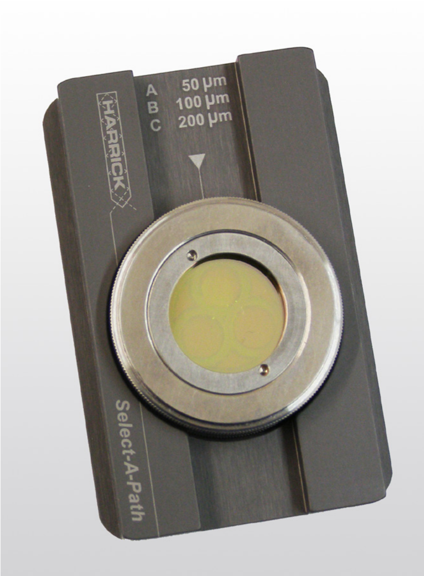

Figure 1. The Select-a-Path™.

INTRODUCTION

Transmission spectroscopy is widely used in the mid-infrared for analysis of liquids. One of the limitations of transmission is the pathlength of the cell. Most of the samples investigated are mixtures, where measurement of the higher concentration component may need to be done with a shorter pathlength than is optimal for the lower concentration species. In some cases, it may be necessary to use several different pathlengths for the analysis of all the desired components. This is likely to introduce additional errors using a traditional demountable liquid cell where only one pathlength is available at a time.

This work demonstrates the potential of one cell with several different pathlengths using a simple mixture of two oils.

EXPERIMENTAL

The FTIR measurements were carried out using the Select-a-Path™ and a commercial FTIR spectrometer equipped with KBr beam-splitter

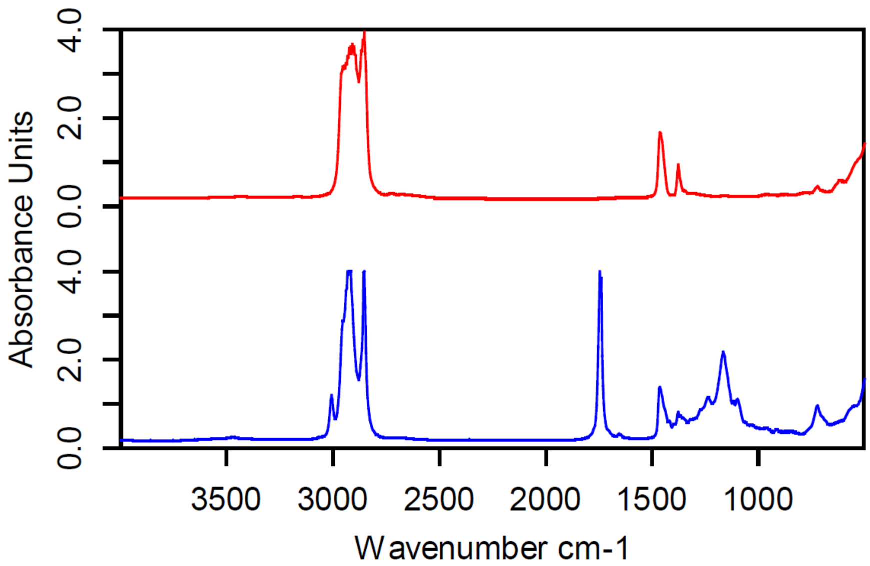

Figure 2. The spectra of paraffin oil (red) and corn oil (blue) measured at a 54.9 µm pathlength.

and DTGS detector. The spectro-meter parameters were as follows: the spectral range was set from 4000 cm^{-1} to 500 cm^{-1}, the aperture was set to 10% with a gain of 2; the optical velocity was 0.6329; the number of scans was 32 and the measurements were taken with a resolution of 4cm^{-1}.

A mixture of Paraffin Oil (MC/B, L549) and Mazola Corn Oil was prepared by pipetting 50 drops of paraffin oil into 5 drops of corn oil, generating a solution ~9% by volume.

Four drops of solution was applied onto the Select-a-Path™ cell, one on each of the three pathlength 'islands' and one in the center. The single beam background measurement was recorded with the Select-a-Path slide plate installed in the slide plate holder of the spectrometer. Then the Select-a-Path™ was installed in its mount. The cell was then rotated to one of the indicated pathlengths and the sample spectrum collected. This procedure was repeated for the other two pathlengths.

RESULTS AND DISCUSSION

Figure 2 shows the difference between the two oils. While the C-H stretching region around 3000 cm^{-1} are clearly saturated, the fingerprint region is not and there are clearly distinct bands in the two oils.

Figure 3 shows the spectra at all three pathlengths. In the fingerprint region, the band at around 1490 cm^{-1} from corn oil is close to saturation at the longest pathlength so it would be best examined at one of the shorter pathlengths. The corn oil band between 1750 cm^{-1} can be seen clearly at all pathlengths (see Figure 4).

To demonstrate the performance of the cell, the area under the 1750 cm^{-1} band, as calculated using spectrometer software, was graphed as function of pathlength (see Figure 5). A trend line was drawn through the points, with the trend line at zero pathlength set to zero. The resulting R2 value of 0.9946 shows that the absorbance versus pathlength line exhibits good linearity, as required by the Lambert Law.

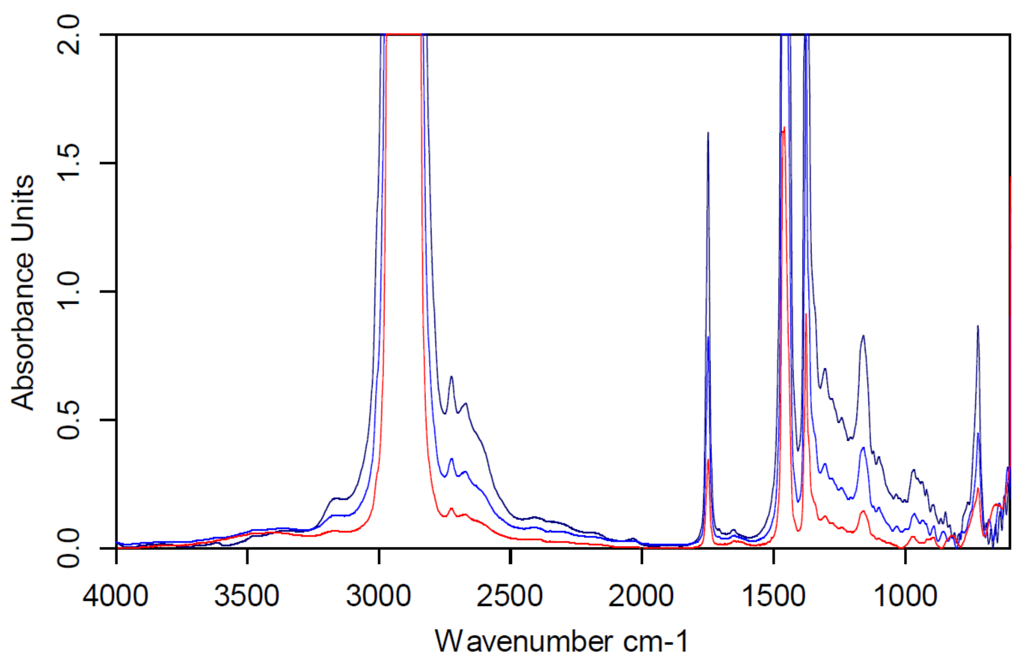

Figure 3. The spectrum of paraffin oil in corn oil, measured with pathlengths of 54.9 µm (red), 102.9 µm (blue) and 201.6 µm (black).

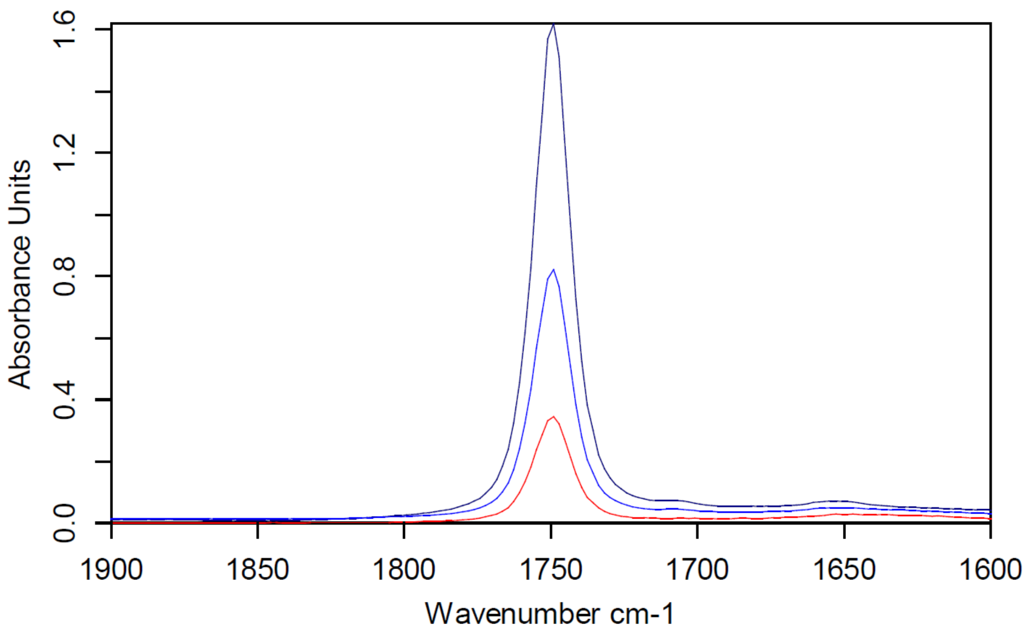

Figure 4. Expanded spectra of paraffin oil in corn oil measured with pathlengths of 54.9 µm (red), 102.9 µm (blue) and 201.6 µm (black).

Presumably the 1750 cm^{-1} band could be used with a proper calibration curve to determine the concentration of corn oil in the mix. To keep well away from potential saturation effects in this mixture, one of the two shorter pathlengths would be the best choice. To examine the concentration of paraffin oil at the same time would be challenging because most of the bands overlap with those of corn oil. But there is a small unique band at 1303 cm^{-1}. Because of the low intensity of this band, it would best be analyzed at the longest pathlength.

CONCLUSION

This experiment shows that measuring absorbance at three different path lengths can be done quickly and efficiently with the Select-A-Path™ cell. If a traditional liquid cell were used for this experiment, every time a measurement at a different path length needed to be taken the cell would need to be disassembled, cleaned, and a new sample aliquot would have to be used. This adds considerable amount of time to an experiment, especially if it includes a plethora of samples needed for reproducibility. With the Select-A-Path™, it is simple to change pathlengths for different measurements, as the cell is simply rotated to align with the desired pathlength for data collection.

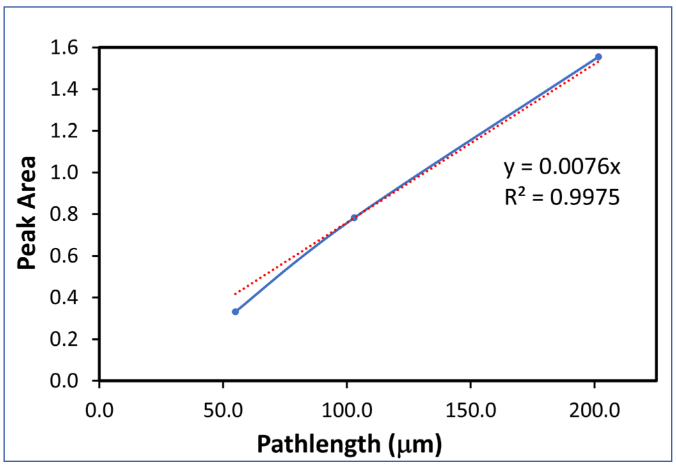

Figure 5. Absorbance vs. pathlength at 1750 cm^{-1}.