All data taken at the Pacific Northwest National Laboratory
FTS Operators: Tracy J. Baker
Data Analysis: Tracy J. Baker

Composite spectrum for: 2-Ethylhexyl acrylate (0.002% monomethyl ether hydroquinone as stabilizer)

- First Column: Position in wavenumber (cm^{-1})
- Second column: Real refractive index *n(ῡ)* (dispersion index)
- Third column: Imaginary refractive index, *k(ῡ)* (absorption index per unit length in centimeters)

Where the complex refractive index $\hat{n} = n(\tilde{\upsilon}) + ik(\tilde{\upsilon})$

Following Bertie (in the references below) we define the absorbance as $A = -\log_{10}(I/I_0)$ and the linear absorption coefficient K = A/d, where d is the path length. The connection between the imaginary refractive index and the absorbance coefficient arises from the following: $2.303K = 4\,\pi\,\tilde{\upsilon}\,k$

See the following references for a detailed description of terms and units:

1) Bertie, J. E., Zhang, S. L., Eysel, H. H., Baluja, S., & Ahmed, M. K. (1993). Infrared Intensities of Liquids XI: Infrared Refractive Indices from 8000 to 2 cm^{-1}, Absolute Integrated Intensities, and Dipole Moment Derivatives of Methanol at 25°C. *Appl. Spec.,47*(8), 1100-1114 doi:10.1366/0003702934067973
2) Bertie, J. E., Zhang, S. L., & Keefe, C. D. (1995). Measurement and use of absolute infrared absorption intensities of neat liquids. *Vibrational Spectroscopy, 8*(2), 215-229. doi:10.1016/0924-2031(94)00038-i

Sample:

- Chemical name, formula and CAS number: 2-Ethylhexyl acrylate, $C_{11}H_{20}O_2$, [103-11-7]
- IUPAC name: 2-Ethylhexyl prop-2-enoate
- Synonyms: 2-Ethylhexyl 2-propenoate; (±)-Acrylic acid 2-ethylhexyl ester
- Physical properties: FW = 184.27 g/mole; mp = -90 °C; bp = 215-219 °C; ρ = 0.885 g/cm^3, vp= 0.15 Torr (20 °C)
- Supplier and stated purity: Aldrich, ≥ 99.7% and 0.002% stabilizer (Lot # MKCR8638)
- Temperature of sample: 25 °C (+/-1 °C)
- Individual samples were measured at the following path lengths: MIR: 1.86, 6.49, 10.7, 15.9, 23.4, 36.0, 63.3, 101, 193 and 503 micrometers (μm); NIR: 101, 193, 504, 942 and 2090 μm. Final data are a composite of these spectra.
- Sample cell window material: MIR = potassium bromide (KBr) except potassium chloride (KCl) for the 193 μm cell; NIR = KBr except KCl for the 193, 942 and 2090 μm cells.
- Preparation: None

NIR Instrument Parameters:

- Bruker Vertex 70, purged with UHP nitrogen
- Spectral range: 10,000 to 3,000 cm^{-1} (1.0 to 3.33 microns)
- NIR source: Quartz tungsten bulb
- Beamsplitter: Broadband potassium bromide (KBr)
- Detector: DLTGS at room temperature
- Aperture: 3 mm
- Folding limits: 31597.6 to 0 cm^{-1}

MIR Instrument Parameters:

- Tensor II with stage rotated 6 degrees, purged with UHP nitrogen
- Spectral range: 7,800 to 400 cm^{-1} (1.282 to 25 microns)
- IR source: Silicon carbide glow bar
- Beamsplitter: Potassium bromide (KBr)
- Detector: DTGS at room temperature
- Aperture: 3 mm
- Folding limits: 11664.15 to 0 cm^{-1}

NIR/MIR Instrument Parameters:

- Instrument resolution: 2.0 cm^{-1}
- Number of interferograms averaged per single channel spectrum: 128; 32 (MIR)
- Apodization: Norton-Beer, Medium
- Phase correction: Mertz
- Scanner velocity: 10 kHz; 7.5 kHz (MIR)
- Interferogram zerofill: 4x
- Spectral interval after zerofilling: 0.4822 cm^{-1}

a) b)

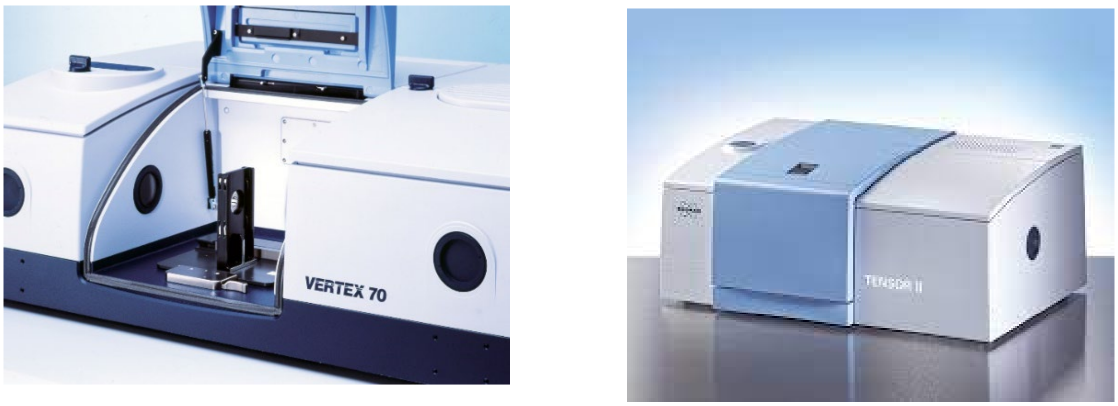

Figure 1: The Bruker Vertex 70 FTIR (a) and Tensor II (b).

Measured Refractive Index:

The refractive index for 2-Ethylhexyl acrylate was measured at 25 °C using an Atago model DR-M2/1550 Abbe refractometer. Notch filters were employed in front of a white light source to make measurements at multiple wavelengths. An infrared viewer from Atago was used to detect signal at 1550 nm. The temperature was controlled to match that in the sample compartment of the FTIR using a heated circulating bath.

480 nm: n = 1.4389	486 nm: n = 1.4386	546 nm: n = 1.4346
589 nm: n = 1.4324	644 nm: n = 1.4304	656 nm: n = 1.4301
1550 nm: n = 1.4255		

The refractive index, n, vs. wavelength in microns, λ, was fit to an equation similar to that of Sellmeier:

$$n(\lambda) = \{a + b/(\lambda^2 - c)\}^{1/2}$$

The resulting best-fit equation was used to find the refractive index at the highest energy data points in our experimental spectra. For 2-Ethylhexyl acrylate, the results were

n(7,800 cm^{-1}) = 1.4258 at 25 °C for MIR data and
n(10,000 cm^{-1}) = 1.4267 at 25 °C for NIR and merged data.

Post Processing and Related Parameters:
For the MIR, a composite spectrum was created from 10 absorbance spectra (base-10) taken at 10 path lengths: 1.86, 6.49, 10.7, 15.9, 23.4, 36.0, 63.3, 101, 193 and 503 micrometers (µm). These data were collected with the sample stage rotated by 6° to minimize artifacts resulting from back-reflection into spectrometer (see Johnson et al., Appl. Spectrosc., 76(5) 620-624, 2021). For the NIR, a composite spectrum was created from 5 absorbance spectra (base-10) taken at 5 path lengths: 101, 193, 504, 942 and 2090 µm. The same cells and liquid fills for the ~100, 200 and 500 µm path lengths were used for both spectral ranges. At each path length several spectra were measured and the results averaged for better signal to noise. The measured cell lengths were adjusted using Beer's law plots in which the NIR and MIR data were analyzed independently.

1) The imaginary part of the refractive index, or *k* vector, was determined for each absorbance file as per Bertie's program "RNJ46A" (see reference above). This takes into account the reflective losses due to the KBr and/or KCl windows.
2) A composite *k* vector is created via a classical, weighted, linear, least squares fit using the output files of program "RNJ46A": Intercept=0, slope is fitted, individual absorbance values weighted by T^2 (transmission squared), all absorbance values ≥ 2.5 are given zero weight. For the MIR, eight composite vectors were created and merged by hand.
    a) The first *k* vector used the results from the 193 and 503 µm cells. This *k* vector determined the final values for the range from 7800 to 3000 cm^{-1} and 800 to 660 cm^{-1}.
    b) The second *k* vector used the results from the 15.9 through 36.0 µm cells. This *k* vector determined the final values for the range from 3000 to 2920 cm^{-1}, 1670 to 1410 cm^{-1} and 1400 to 1200 cm^{-1}.
    c) The third *k* vector used the results from the 23.4 through 63.3 µm cells. This *k* vector determined the final values for the range from 2920 to 2800 cm^{-1} and 1175 to 800 cm^{-1}.
    d) The fourth *k* vector used the results from the 503 µm cell. This *k* vector determined the final values for the range from 2800 to 1800 cm^{-1} and 660 to 370 cm^{-1}.
    e) The fifth *k* vector used the results from the 1.86 through 10.7 µm cells. This *k* vector determined the final values for the range from 1800 to 1720 cm^{-1}.
    f) The sixth *k* vector used the results from the 36.0 and 63.3 µm cells. This *k* vector determined the final values for the range from 1720 to 1670 cm^{-1}.
    g) The seventh *k* vector used the results from the 10.7 and 15.9 µm cells. This *k* vector determined the final values for the range from 1410 to 1400 cm^{-1}.
    h) The eighth *k* vector used the results from the 6.49 µm cell. This *k* vector determined the final values for the range from 1200 to 1175 cm^{-1}.
3) A frequency correction was applied to the resulting composite MIR *k* vector.
    a) Frequency correction (already applied): $\tilde{\upsilon}$(corrected) = [$\tilde{\upsilon}$(instrument) * 0.99988 + 0.025] as determined by comparing measured atmospheric spectral lines (H_2O and CO_2) to values from the Northwest Infrared Spectral Library Database.
4) For the NIR, five composite vectors were created and merged by hand.
    a) The first *k* vector used the results from the 2090 µm cell. This *k* vector determined the final values for the range from 10,000 to 4450 cm^{-1} and 4000 to 3470 cm^{-1}.
    b) The second *k* vector used the results from the 504 through 2090 µm cells. This *k* vector determined the final values for the range from 4450 to 4000 cm^{-1} and 3470 to 3400 cm^{-1}.
    c) The third *k* vector used the results from the 942 and 2090 µm cells. This *k* vector determined the final values for the range from 3400 to 3120 cm^{-1}.
    d) The fourth *k* vector used the results from the 193 through 942 µm cells. This *k* vector determined the final values for the range from 3120 to 3000 cm^{-1}.
    e) The fifth *k* vector used the results from all the cells. This *k* vector determined the final values for the range from 3000 to 370 cm^{-1}.
5) The resulting composite NIR *k* vector and the refractive index at 10,000 cm^{-1} were used to create the real or *n* vector using the Kramers-Kronig relation, as per Bertie's program "LZZKTB."
    a) Frequency correction (already applied): $\tilde{\upsilon}$(corrected) = [$\tilde{\upsilon}$(instrument) * 0.99998 - 0.0005] as determined by comparing measured atmospheric spectral lines (H_2O and CO_2) to values from the Northwest Infrared Spectral Library Database.
6) The MIR data were mapped onto the NIR x-axis using an interpolation routine, i.e. the Make Compatible

command in OPUS 5.5. Then the composite MIR and NIR k vectors were merged around 3000 cm^{-1} to generate a final composite k vector across the entire spectral range. The resulting composite k vector and the refractive index at 10,000 cm^{-1} were used to create the final n vector using the Kramers-Kronig relation, as per Bertie's program "LZZKTB."

Photograph of Sample 2-Ethylhexyl acrylate:

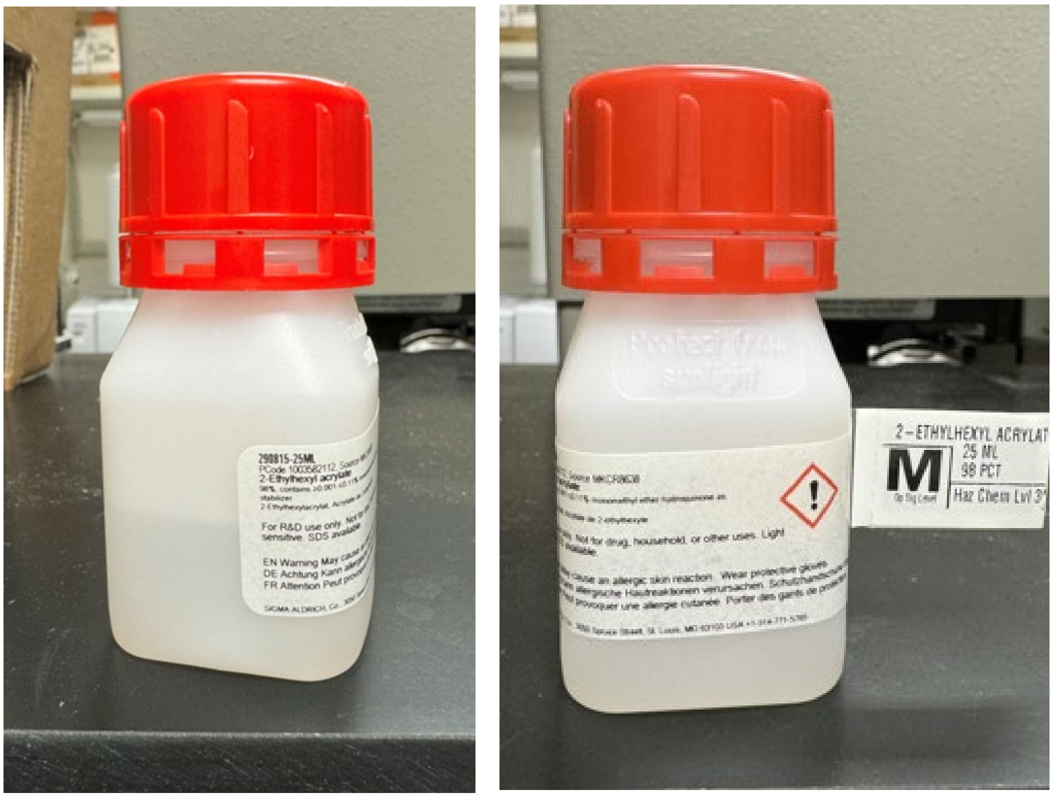

Figure 2: 2-Ethylhexyl acrylate in Aldrich container for NIR and MIR measurements.