All data taken at the Pacific Northwest National Laboratory
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Composite spectrum for: Butyl stearate

- First Column: Position in wavenumber (cm^{-1})
- Second column: Real refractive index $n(\tilde{\upsilon})$ (dispersion index)
- Third column: Imaginary refractive index, $k(\tilde{\upsilon})$ (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: Butyl stearate, $C_{22}H_{44}O_2$, [123-95-5]
- IUPAC name: Butyl octadecanoate
- Synonyms: Stearic acid, butyl ester; n-Butyl octadecanoate; n-Butyl stearate; Emerest 2325
- Physical properties: FW = 340.58 g/mole; mp = 27 °C; bp = 343 °C; $\rho = 0.855\ g/cm^3$
- Supplier and stated purity: Sigma-Aldrich, ≥ 95% (Lot # BCBQ0290)
- Temperature of sample: 35 °C (+/-1 °C)
- Individual samples were measured at the following path lengths: MIR: 1.94, 4.03, 11.9, 17.4, 18.9, 30.2, 63.6, 118, 204 and 506 µm; NIR: 118, 203, 505 and 990 µm. Final data are a composite of these spectra.
- Sample cell window material is potassium bromide (KBr) except the 30.2 (MIR), 204 (MIR), 506 (MIR), 203 (NIR) 505 (NIR), and 989 (NIR) cell windows were potassium chloride (KCl).
- 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: 31601 to 0 cm^{-1}

MIR Instrument Parameters (11.9 through 506 µm data):

- Tensor 27, purged with UHP nitrogen
- Spectral range: 7800 to 400 cm^{-1} (1.282 to 25 microns)
- IR source: Silicon carbide glow bar
- Beamsplitter: Broadband Potassium bromide (KBr)
- Detector: DLTGS at room temperature
- Aperture: 3 mm
- Folding limits: 15802 to 0 cm^{-1}

MIR Instrument Parameters (1.94 and 4.03 µm data):

- Bruker Vertex 70, purged with UHP nitrogen
- Spectral range: 7800 to 400 cm^{-1} (1.33 to 25 microns)
- IR source: Silicon carbide glow bar
- Beamsplitter: Broadband Potassium bromide (KBr)
- Detector: DLTGS at room temperature
- Aperture: 3 mm
- Folding limits: 15802 to 0 cm^{-1}

NIR/MIR Instrument Parameters:

- Instrument resolution: 2.0 cm^{-1}
- Number of interferograms averaged per single channel spectrum: 128
- Apodization: Norton-Beer, Medium
- Phase correction: Mertz
- Scanner velocity: 10 kHz
- Interferogram zerofill: 4x
- Spectral interval after zerofilling: 0.4823 cm^{-1}

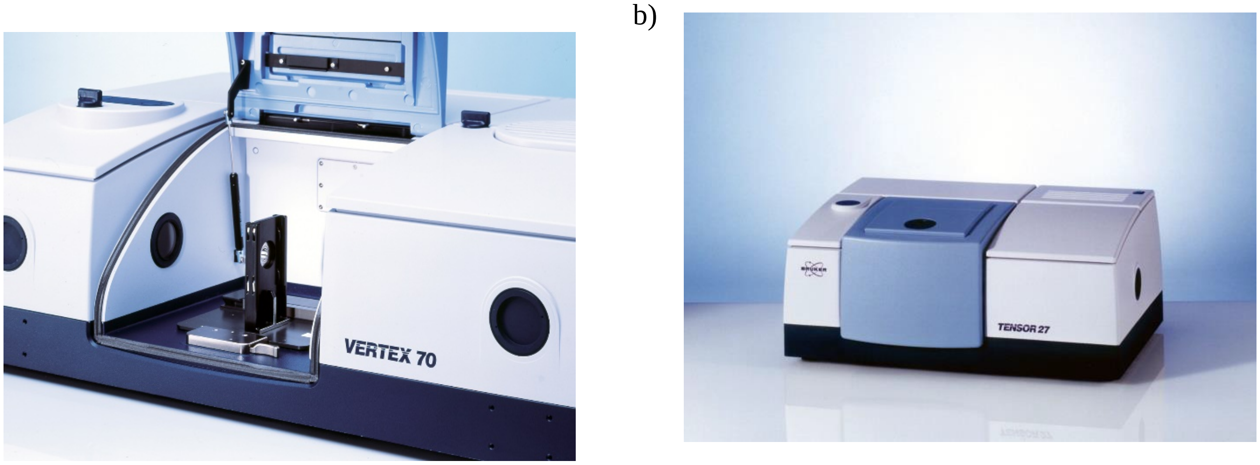

Figure 1: The Bruker Vertex 70 FTIR (a) and Tensor 27 FTIR (b).

Measured Refractive Index:

The refractive index for Butyl stearate was measured at 35 °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 = 1.4456	486 nm: n = 1.4439	546 nm: n = 1.4399
589 nm: n = 1.4364	644 nm: n = 1.4359	656 nm: n = 1.4339
1550 nm: n = 1.4177		

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 Butyl stearate, the results were

$$n(7800\ cm^{-1}) = 1.4198 \text{ at } 35\ °C \text{ for MIR data and}$$
$$n(10{,}000\ cm^{-1}) = 1.4237 \text{ at } 35\ °C \text{ for NIR and merged data.}$$

Post Processing and Related Parameters:
For the MIR, a composite spectrum was created from 10 absorbance spectra (base-10) taken at 10 path lengths: 1.94, 4.03, 11.9, 17.4, 18.9, 30.2, 63.6, 118, 204 and 506 micrometers (µm). For the NIR, a composite spectrum was created from 4 absorbance spectra (base-10) taken at 4 path lengths: 118, 203, 505 and 990 µm. The same cells 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 windows and the measured refractive index at 7800 cm^{-1} for the MIR data and 10,000 cm^{-1} for the NIR data.
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, six composite vectors were created and merged by hand.
   a) The first *k* vector used the results from the 118 through 506 µm cells. This *k* vector determined the final values for the range from 7800 to 3400 cm^{-1} and 2700 to 1850 cm^{-1}.
   b) The second *k* vector used the results from the 1.94 through 17.4 µm cells. This *k* vector determined the final values for the range from 3400 to 2700 cm^{-1}.
   c) The third *k* vector used the results from the 1.94 through 18.9 µm cells. This *k* vector determined the final values for the range from 1850 to 1650 cm^{-1}.
   d) The fourth *k* vector used the results from the 11.9 through 118 µm cells. This *k* vector determined the final values for the range from 1650 to 650 cm^{-1}.
   e) The fifth *k* vector used the results from the 63.6, 118 and 506 µm cells. This *k* vector determined the final values for the range from 650 to 400 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) * .999848+ .010722] for data collected on the Tensor 27 and $\tilde{\upsilon}$(corrected) = [$\tilde{\upsilon}$(instrument) * .999869 + .0158818] for data collected on the Vertex70 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, three composite vectors were created and merged by hand.
   a) The first *k* vector used the results from the 505 and 990 µm cells. This *k* vector determined the final values for the range from 10000 to 7450 cm^{-1}.
   b) The second *k* vector used the results from the 203 through 990 µm cells. This *k* vector determined the final values for the range from 7450 to 6200 cm^{-1}.
   c) The third *k* vector used the results from the 118 through 505 µm cells. This *k* vector determined the final values for the range from 6200 to 400 cm^{-1}.
5) A frequency correction was applied to the resulting composite NIR *k* vector.
   a) Frequency correction (already applied): $\tilde{\upsilon}$(corrected) = [$\tilde{\upsilon}$(instrument) * .999869 + .0158818] as determined by comparing measured atmospheric spectral lines (H_2O and CO_2) to values from the Northwest Infrared Spectral Library Database.
6) Finally, 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 to generate a final composite *k* vector across the entire spectral range. The NIR data were used exclusively above 6750 cm^{-1}, and only the MIR data were used below 6000 cm^{-1}. A weighted average, with the weight of the MIR vector increasing linearly from 0 to 100% between 6750 and 6000 cm^{-1} was used in the

overlapping spectral region. 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 Butyl stearate:

Figure 2: Butyl stearate in Sigma-Aldrich container.