All data taken at the Pacific Northwest National Laboratory
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Composite spectrum for: Iron pentacarbonyl

- 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. *Applied Spectroscopy, 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: Iron pentacarbonyl, C_5FeO_5, [13463-40-6]
- IUPAC name: Pentacarbonyliron
- Synonyms: Iron(0) pentacarbonyl; Iron(0) carbonyl; Pentacarbonyliron(0)
- Physical properties: FW = 195.90 g/mole; mp = −20 °C; bp = 103 °C; $\rho = 1.49$ g/cm^3
- Supplier and stated purity: Aldrich, > 99.99% (Lot # SHBD9102V)
- Temperature of sample: 26 °C (+/-1 °C)
- Individual samples were measured at the following path lengths: 0.865, 3.67, 6.58, 15.3, 30.5, 55.8, 101 and 205 micrometers (μm). Final data are a composite of these spectra.
- Sample cell window material is potassium bromide (KBr).
- Preparation: None.

Instrument Parameters:

- Bruker Tensor 27 FTIR, purged with UHP nitrogen
- Spectral range: 7800 to 400 cm^{-1} (1.282 to 25 microns)
- 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
- Folding limits: 15802 to 0 cm^{-1}
- Interferogram zerofill: 4x
- Spectral interval after zerofilling: 0.4823 cm^{-1}
- IR source: Silicon carbide glow bar
- Beamsplitter: Broadband potassium bromide (KBr)
- Detector: DLTGS at room temperature
- Aperture: 3 mm

Measured Refractive Index:
The refractive index for Iron pentacarbonyl was measured at 27 °C using an Atago model DR-M2/1550 Abbe refractometer at only one wavelength due to the toxicity of the liquid. A notch filter was employed in front of a white light source to make measurements and an InGaAs camera 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 = –	486 nm: n = –	546 nm: n = –
589 nm: n = –	644 nm: n = –	656 nm: n = –
1550 nm: n = 1.4773		

In general, the refractive index, n, vs. wavelength in microns, λ, is fit to an equation similar to that of Sellmeier:

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

Then, the resulting best-fit equation is used to find the refractive index at the highest energy data point in our experimental spectra. For Iron pentacarbonyl, however, only one measurement was obtained so that an estimate of the change in refractive index between 1550 nm and 7800 cm^{-1} was calculated from prior measurements of 53 other liquids. The change in refractive index from these other liquids was calculated to be .0013 ± .0006. Thus, for Iron pentacarbonyl, the result was

$$n(7800\ cm^{-1}) = 1.4786 \text{ at } 27\ °C.$$

a) b)

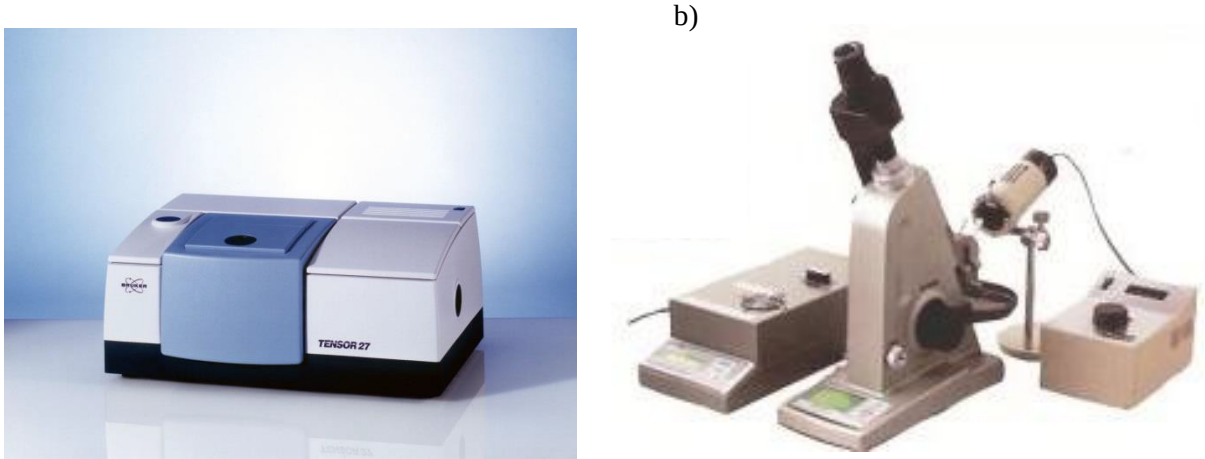

Figure 1: The Bruker Tensor 27 FTIR (a) and Abbe refractometer (b).

Post Processing and Related Parameters:
A composite spectrum was created from 8 absorbance spectra (base-10) taken at 8 path lengths: 0.865, 3.67, 6.58, 15.3, 30.5, 55.8, 101 and 205 micrometers (μm). 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.

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.
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. Four composite vectors were created and merged by hand.
   a) The first k vector used the results from the 205 and 101 μm cells. This k vector determined the final values for the range from 7800 to 2246 cm^{-1}.

b) The second *k* vector used the results from the 55.8 and 30.5 μm cells. This *k* vector determined the final values for the range from 2246 to 2062 cm^{-1}, 1832 to 686 cm^{-1} and 549 to 400 cm^{-1}.
c) The third *k* vector used the results from the 15.3 and 6.58 μm cells. This *k* vector determined the final values for the range from 1926 to 1832 cm^{-1}, 686 to 651 cm^{-1} and 607 to 549 cm^{-1}.
d) The fourth *k* vector used the results from the 3.67 and 0.865 μm cells. This *k* vector determined the final values for the range from 2062 to 1926 cm^{-1} and 651 to 607 cm^{-1}.

3) The resulting composite *k* vector and the refractive index at 7800 cm^{-1} were used to create the real or *n* vector using the Kramers-Kronig relation, as per Bertie's program "LZZKTB."
a) Calculated and estimated errors: Type A = 0.7%.
b) Frequency correction (already applied): $\tilde{\nu}$(corrected) = [$\tilde{\nu}$(instrument) * .99977 - .01872] as determined by comparing measured atmospheric spectral lines (H_2O and CO_2) to values from the Northwest Infrared Spectral Library Database.
c) Axis units: X = Wavenumbers (cm^{-1}); Y = Absorbance (base 10).

Photograph of Sample Iron pentacarbonyl:

Figure 2: Iron pentacarbonyl in Aldrich container.