

All data taken at Pacific Northwest National Laboratory (PNNL)

Operators: Steven W. Sharpe, Timothy J. Johnson and Robert L. Sams : sw.sharpe@pnl.gov

Version 1.0, December, 05

Composite spectrum for MOTHBAL_25T

Effective burden of composite spectrum: 1 part-per-million-meter (ppm-meter) at 296 K

Equivalent concentration x path-length of composite spectrum: 5.277×10^{-6} grams/liter-meter

Sample prepared by volumetrically dissolving naphthalene in carbon disulfide. Solvent spectra (CS₂) removed by spectral subtraction.

Sample Conditions-

- Chemical name and CAS number: Naphthalene, camphor tar, mothballs, mighty rd1, C₁₀H₈ : [91-20-3]
- Physical properties: MW=128.1732 g/mole, mp=80.6° C, bp=218° C, Density (20 C) 0.997 g/cm³
- Supplier and stated purity: Aldrich, 99+%
- Sample class: I (PNNL scale).
- Temperature of White cell (796.0 cm optical path length) 25 ± 2 C
- Diluent (high purity nitrogen) flowed at 24.2 liter/min (21.1° C), ambient atmospheric pressure 760 ± 5 Torr.
- Sample was prepared as a solution in carbon disulfide: 3.78 grams up to 10 ml, in a volumetric flask. A pseudo sample density of 0.378 g/cm³ was used in the flow calculations. Samples flowed at 1.000, 3.000, 5.000, 7.000, 10.000, 15.000, 20.000, 2.000, 0.500, 4.000 and 30.000 microliters/minute.
- Individual samples at equivalent pressures of 0.002227, 0.006677, 0.011127, 0.015576, 0.022251, 0.033341, 0.044437, 0.004443, 0.001110, 0.008880 and 0.066585 Torr. Final data is a composite spectrum.
- Preparation: None

Instrument Parameters-

- Bruker-66V FTIR, evacuated optics bench.
- Modified to include second aperture, between interferometer output and White cell. This substantially reduces both “ghosting” and warm aperture effects.
- Spectral range: 6,500 to 600 cm⁻¹ (1.538 to 16.667 microns)
- Instrumental resolution based on maximum interferometer displacement is 0.112 cm⁻¹
- Spectral interval after 2X zero-filling interferogram and FFT: 0.06 cm⁻¹
- Interferogram zero-fill: 2X
- Apodization: Boxcar
- Phase correction: Mertz
- Beam splitter: Potassium bromide (KBr)
- IR source: Carbide glowbar (22 V)
- Scanner velocity: 60KHz (HeNe crossing frequency)
- Number of interferograms averaged per single channel spectra: 256
- Detector: Mid-band HgCdTe, photoconductive, 77K operation
- Folding limits: 15798 to 0 cm⁻¹

Post Processing and Related Parameters-

- Non-linearity detector correction (Bruker proprietary) applied to interferogram ($\alpha=0.90$, $\epsilon=500$)

- Composite spectrum created from 11 individual absorbance (base-10) spectra via classical least squares fit: Intercept=0, slope is fitted, individual absorbance values weighted by T^2 (transmission squared), all absorbance values ≥ 1.6 are given zero weight
- Calculated and estimated errors: Type A = 1.74%, Type B $\leq 7\%$
- Frequency correction (already applied): $V(\text{corrected})=V(\text{instrument})*0.99999959-3.45278 \times 10^{-4}$
- Axis units: X=wavenumbers (cm^{-1}), Y=Absorbance (base-10)
- Baseline correction via 7th order polynomial subtraction