

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, 03

Composite spectrum for HCCPD_50T

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

Equivalent concentration x path-length of composite spectrum: 1.123×10^{-5} grams/liter-meter

Sample Conditions-

- Chemical name and CAS number: Hexachlorocyclopentadiene, 1,2,3,4,5-hexachloro-1,3-cyclopentadiene, HCCPG, C56, grapholox, hrs-1655, C_5Cl_6 : [77-47-4]
- Physical properties: M.W. 72.773 amu, F.P. $-9.9^\circ C$, B.P. $239^\circ C$, Density (20 C) 1.702 g/cm^3
- Supplier and stated purity: Aldrich, 98+%
- Sample class: I (PNNL scale).
- Temperature of White cell (815.76 cm optical path length) $50 \pm 2 C$
- Diluent (high purity nitrogen) flowed at 25.2 liter/min ($21.1^\circ C$), ambient atmospheric pressure 760 ± 5 Torr.
- Samples flowed at 3.000, 2.000, 0.800, 1.200, 1.500, 1.000, 0.600, 2.000, 0.700, 1.700, 0.500, 1.300 and 2.500 microliters/minute
- Individual samples at equivalent pressures of 0.013509, 0.009005, 0.003607, 0.005411, 0.006765, 0.004510, 0.002705, 0.009011, 0.003152, 0.007657, 0.002252, 0.005852 and 0.011252 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 575 cm^{-1} (1.538 to 17.391 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 13 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 = 0.42%, Type B $\leq 7\%$
- Frequency correction (already applied): $V(\text{corrected}) = V(\text{instrument}) * 0.999998 + 1.566836e-04$

- Axis units: X=wavenumbers (cm^{-1}), Y=Absorbance (base-10)
- Baseline correction via 5th order polynomial subtraction
- Trace carbon monoxide and carbon dioxide features removed from composite spectrum via spectral subtraction.