

All data taken at Pacific Northwest National Laboratory (PNNL)

Operators: Steven W. Sharpe, Timothy J. Johnson and Robert L. Sams : [sw.sharpe@pnl.gov](mailto:sw.sharpe@pnl.gov)

Version 1.0, July, 02

Composite spectrum for DCLP13\_25T

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

Equivalent concentration x path-length of composite spectrum:  $4.6517 \times 10^{-6}$  grams/liter-meter

### Sample Conditions-

- Chemical name and CAS number: 1,3-Dichloropropane, trimethyl dichloride,  $\text{CH}_2\text{ClCH}_2\text{CH}_2\text{Cl}$ : [142-28-9]
- Physical properties: fw=112.9864 g/mole, fp=-99° C, bp=120.4° C
- Supplier and stated purity: Aldrich, 99%
- Sample class: I (PNNL scale).
- Temperature of sample:  $25.01 \pm 0.02$  C
- Diluent: Sample back filled with ultra high purity nitrogen to  $760 \pm 5$  Torr
- Individual samples at 1.1212, 4.0084, 8.0542, 14.09, 0.66200, 6.0333, 3.0687, 12.12, 5.2767, 10.0810, 2.0444 and 13.17 Torr. Path length = 19.96 cm. Final data is a composite spectrum.
- Preparation: Multiple freeze-thaw cycles at -60 C to remove air.

### Instrument Parameters-

- Bruker-66V FTIR, temperature controlled environment, evacuated optics bench
- Modified to include second aperture, between interferometer output and sample cell. This substantially reduces both “ghosting” and warm aperture effects.
- Spectral range:  $6,500$  to  $570 \text{ cm}^{-1}$  (1.534 to 17.544 microns)
- Instrumental resolution based on maximum interferometer displacement is  $0.112 \text{ cm}^{-1}$
- Spectral interval after 2X zero-filling interferogram and FFT:  $0.06 \text{ cm}^{-1}$
- 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  $0 \text{ cm}^{-1}$

### Post Processing and Related Parameters-

- Non-linearity detector correction (Bruker proprietary) applied to interferogram ( $\tau = 0.90$ ,  $\nu = 500$ )
- Composite spectrum created from 12 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 = 0.15%, Type B = 3%
- Frequency correction (already applied):  $V(\text{corrected}) = V(\text{instrument}) * 0.999998 - 2.75000e-6$
- Axis units: X=wavenumbers ( $\text{cm}^{-1}$ ), Y=Absorbance (base-10)
- Trace carbon dioxide vapor features removed via spectral subtraction
- Baseline correction via 3<sup>rd</sup> order polynomial subtraction