

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, April, 01

Composite spectrum for ClPicrin\_5T

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

Equivalent concentration x path-length of composite spectrum:  $6.768 \times 10^{-6}$  grams/liter-meter

### Sample Conditions-

- Chemical name and CAS number: Trichloronitromethane, chloropicrin, nitrotrichloromethane, nitrochloroform,  $\text{CCl}_3\text{NO}_2$  : [76-06-2]
- Physical properties: F.W. 164.38 amu, F.P.  $-69.2$  C, B.P. 112 C
- Supplier and stated purity: Aldrich, 98%
- Sample class: I (PNNL scale).
- Temperature of sample:  $5.11 \pm 0.02$  C
- Diluent: Sample back filled with ultra high purity nitrogen to  $760 \pm 5$  Torr
- Individual samples at 1.03580, 2.0619, 3.2961, 4.2300, 5.1913 and 0.75793 Torr. Path length = 19.94 cm. Final data is a composite spectrum.
- Preparation: Multiple freeze-thaw cycles at  $-50$  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 600  $\text{cm}^{-1}$  (1.534 to 16.667 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 ( $\tau = 0.85$ ,  $\nu = 530$ )
- Composite spectrum created from 6 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 = 0.51%, Type B = 3%
- Frequency correction (already applied):  $V(\text{corrected}) = V(\text{instrument}) * 0.999997 + 5.18 \times 10^{-4}$
- Axis units: X=wavenumbers ( $\text{cm}^{-1}$ ), Y=Absorbance (base-10)
- Trace water vapor features removed via spectral subtraction.
- Baseline correction via 7<sup>th</sup> order polynomial subtraction