

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, May, 03

Composite spectrum for TCE111\_25T

Effective burden of composite spectrum: 1 part-per-million-meter  $5.492410^{-6}$  grams/liter-meter

[Composite spectrum corrected for contamination of 1,1-Dichloroethene \[0.40%\] by rescaling.](#)

### Sample Conditions-

- Chemical name and CAS number: 1,1,1-Trichloroethane, 1,1,1-tce, aethane tt, alpha-trichloroethane, solvent 111, methyl chloroform,  $\text{CH}_3\text{CCl}_3$ ; [71-55-6]
- Physical properties: fw=133.4047 g/mole, fp=-32.6° C, bp=74.1° C
- Supplier and stated purity: Aldrich, 99.5%
- 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 1.08421, 2.0541, 4.2813, 1.5105, 0.81030, 3.1673, 67.75, 32.44, 8.2520 and 16.38 Torr. Path length = 19.96 cm. Final data is a composite spectrum.
- Preparation: Multiple freeze-thaw cycles at 77 K 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 500  $\text{cm}^{-1}$  (1.534 to 20.000 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 ( $\alpha=0.85$ ,  $\beta=530$ )
- Composite spectrum created from 10 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  $\geq 1.6$  are given zero weight
- Calculated and estimated errors: Type A = 0.42%, Type B  $\leq 3\%$
- Frequency correction (already applied):  $V(\text{corrected}) = V(\text{instrument}) * 0.99999896 + 8.812 \times 10^{-4}$
- Axis units: X=wavenumbers ( $\text{cm}^{-1}$ ), Y=Absorbance (base-10)
- Trace carbon dioxide and 1,1-dichloroethene features removed via spectral subtraction
- Baseline correction via 7<sup>th</sup> order polynomial subtraction