

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, November 25, 2008

Composite spectrum for CROTAL_50T

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

Equivalent concentration x path-length of composite spectrum: 2.8856×10^{-6} grams/liter-meter

Sample Conditions-

- Chemical name and CAS number: Crotonaldehyde; 2-Butenal, (E)-; Crotonaldehyde, (E)-; (E)-Crotonaldehyde; trans-Crotonaldehyde; trans-2-Butenal; Topanel CA; Crotonic aldehyde; Crotylaldehyde; Crotonal; 2-Butenal; β -Methyl acrolein; Propylene aldehyde; Topanel; (2E)-2-Butenal; but-(E)-2-enal; (E)-2-butenal; (E)-but-2-enal; t-2-Butenal C₄H₆O: [4170-30-3] OR [123-73-9]
- Physical properties: MW=70.0898 g/mole, mp= -75° C, bp=134° C, Density (25 C) = 0.853 g/cm³
- Supplier and stated purity: Aldrich, mixture of cis and trans >99.5 %
- Sample class: I (PNNL scale).
- Temperature of sample: 49.94 \pm 0.02 C
- Diluent: Sample back filled with ultra high purity nitrogen to 760 \pm 5 Torr.

Individual samples at 1.06400, 2.6800, 0.65300, 28.20, 12.55 and 6.0850 Torr. Path length= 19.96 cm. Final data is a composite spectrum.

- Preparation: Multiple freeze-thaw cycles at 77K to remove air.

Instrument Parameters-

- Bruker-66V FTIR, 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 cm⁻¹ (1.538 to 20.00 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 6 individual absorbance (base-10) spectra via classical least squares fit: Intercept=0, slope is fitted, individual absorbance values weighted by T² (transmission squared), all absorbance values ≥ 1.6 are given zero weight
- Calculated and estimated errors: Type A =0.75%, Type B \leq 3%

- Frequency correction (already applied): $V(\text{corrected})=V(\text{instrument})*1.00000135+8.5741\times 10^{-4}$
- Axis units: X=wavenumbers (cm^{-1}), Y=Absorbance (base-10)
- Trace water features were removed via spectral subtraction.
- Baseline correction via 7th order polynomial subtraction