

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, May, 03

Composite spectrum for TEXANOL_25T

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

Equivalent concentration x path-length of composite spectrum: 8.906×10^{-6} grams/liter-meter

Sample Conditions-

- Chemical name and CAS number: Texanol, 2,2,4-Trimethyl-1,3-pentanediol monoisobutyrate, 2,2,4-Trimethyl-1,3-pentanediol isobutyrate, $(\text{CH}_3)_2\text{CHCH}[\text{OHR}]\text{C}(\text{CH}_3)_2\text{CH}_2\text{OR}(\text{H})$ R=-COCHCH₃)₂ : [25265-77-4]
- Physical properties: M.W. 216.3198 amu, F.P. -50° C, B.P. 244° C, Density (20 C) 0.950 g/cm³
- Supplier and stated purity: Aldrich, 99%
- Sample class: I (PNNL scale).
- Temperature of White cell (792.0 cm optical path length) 25 ± 2 C
- Diluent (high purity nitrogen) flowed at 24.90 liter/min (296 K), ambient atmospheric pressure 770 ± 5 Torr.
- Samples flowed at 2.000, 1.000, 0.800, 0.500, 1.700, 0.700, 1.500, 0.350, 1.350 and 1.20 microliters/minute
- Individual samples at equivalent pressures of 0.006391, 0.003196, 0.002557, 0.001598, 0.005432, 0.002236, 0.004792, 0.001118, 0.004313 and 0.003834 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 600 cm⁻¹ (1.538 to 16.667 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 ($\eta=0.90$, $\xi=500$)
- 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 T² (transmission squared), all absorbance values ≥ 1.6 are given zero weight
- Calculated and estimated errors: Type A = 0.71%, 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 7th order polynomial subtraction