

All data taken at Pacific Northwest National Laboratory (PNNL)

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Composite spectrum for TMB4_50T

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

Equivalent concentration x path-length of composite spectrum: 5.5259×10^{-6} grams/liter-meter

Although vendor claims purity of sample is only 90%, comparison of spectra with the Aldrich Library of FT-IR Spectra does not indicate significant contamination.

Sample Conditions-

- Chemical name and CAS number: 1,2,3,4-Tetramethylbenzene, (C₆H₂)(CH₃)₄ : [488-23-3]
- Physical properties: MW=134.2206 g/mole, mp=-6° C, bp=205° C, Density (20 C) 0.905 g/cm³
- Supplier and stated purity: Pfaltz & Bauer, 90%
- Sample class: I (PNNL scale).
- Temperature of White cell (796.0 cm optical path length) 50 ± 2 C
- Diluent (high purity nitrogen) flowed at 24.2 liter/min (21.1° C), ambient atmospheric pressure 760 ± 5 Torr.
- Samples flowed at 3.000, 6.000, 2.000, 4.000, 5.000, 10.000, 15.000, 25.000, 35.000, 45.000 and 55.000 microliters/minute
- Individual samples at equivalent pressures of 0.015169, 0.030338, 0.010113, 0.020215, 0.025261, 0.050516, 0.075754, 0.126240, 0.176713, 0.227202 and 0.277654 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 ($\alpha=0.90$, $\epsilon=500$)
- Composite spectrum created from 11 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 = 0.25%, Type B $\leq 7\%$

- Frequency correction (already applied): $V(\text{corrected})=V(\text{instrument})\cdot 0.99999959-3.45278\times 10^{-4}$
- Axis units: X=wavenumbers (cm^{-1}), Y=Absorbance (base-10)
- Baseline correction via 7th order polynomial subtraction