

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 2.0, February, 05

Composite spectrum for DMSO_25T

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

Equivalent concentration x path-length of composite spectrum: 3.2166×10^{-6} grams/liter-meter

Contaminated with formaldehyde [0.44%] and water [0.13%] and corrected by rescaling and spectral subtraction. DMSO appears to absorb onto windows and mirrors at higher concentration, causing negative going features in baseline.

Sample Conditions-

- Chemical name and CAS number: Dimethylsulfoxide, Methyl sulfoxide, DMSO, M-176, dimethyl sulfur oxide, dimexide, dromisol, DMS-70, $\text{OS}(\text{CH}_3)_2$: [67-68-5]
- Physical properties: MW=78.1288 g/mole, mp=18.5° C, bp=189° C, Density (20 C) 1.101 g/cm³
- Supplier and stated purity: Aldrich, 99.7%
- Sample class: I (PNNL scale).
- Temperature of White cell (815.76 cm optical path length) 25 ± 2 C
- Diluent (high purity nitrogen) flowed at 25.20 liter/min (21.1° C), ambient atmospheric pressure 760 ± 5 Torr.
- Samples flowed at 3.000, 1.000, 5.000, 2.000, 10.000, 7.000, 20.000, 30.000, 25.000, 15.000 and 22.500 microliters/minute
- Individual samples at equivalent pressures of 0.030652, 0.010216, 0.051079, 0.020426, 0.102118, 0.071407, 0.203967, 0.305869, 0.254655, 0.152752 and 0.229037 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 575 cm⁻¹ (1.538 to 17.391 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 T^2 (transmission squared), all absorbance values ≥ 1.6 are given zero weight
- Calculated and estimated errors: Type A = 0.29%, Type B $\leq 7\%$
- Frequency correction (already applied): $V(\text{corrected})=V(\text{instrument})*0.9999987-4.24224 \times 10^{-4}$
- Axis units: X=wavenumbers (cm^{-1}), Y=Absorbance (base-10)
- Trace water and CO_2 removed via spectral subtraction
- Baseline correction via 7th order polynomial subtraction