

All data taken at Pacific Northwest National Laboratory (PNNL)

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Composite spectrum for DMF_50T

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

Equivalent concentration x path-length of composite spectrum: 3.009×10^{-6} grams/liter-meter

Sample Conditions-

- Chemical name and CAS number: N,N-Dimethylformamide, DMF, N,N-dimethylmethanamide, N-formyldimethylamine, DMFA, formyldimethylamine, $\text{CH}_3(\text{CH}_2)_2\text{NO}_2$: [68-12-2]
- Physical properties: fw=73.09 g/mole, fp=-61° C, bp=153° C
- Supplier and stated purity: Aldrich, 99.9%, HPLC grade
- Sample class: III (PNNL scale). Extremely sticky, forms salts with KCl windows.
- Temperature of sample: 49.97 ± 0.02 C
- Diluent: Sample back filled with ultra high purity nitrogen to 760 ± 5 Torr
- Individual samples at 1.4320, 1.0350, 0.62400, 2.0700, 0.94900 and 3.2822 Torr. Path length = 19.94 cm. Final data is a composite spectrum.
- Preparation: Multiple freeze-thaw cycles at 0 C 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 600 cm^{-1} (1.534 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 ($\alpha = 0.85$, $\beta = 530$)
- 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 T^2 (transmission squared), all absorbance values > 1.6 are given zero weight
- Calculated and estimated errors: Type A = 1.36%, Type B = 10%
- Frequency correction (already applied): $V(\text{corrected}) = V(\text{instrument}) * 0.999997 + 5.18 \times 10^{-4}$
- Axis units: X=wavenumbers (cm^{-1}), Y=Absorbance (base-10)

- Trace water vapor features removed via spectral subtraction. Trace water vapor and carbon dioxide features removed via spectral subtraction. Features observed at ~ 1635 and below 855 cm^{-1} are thought to be due to formation of salts with windows.
- Baseline correction via 7th order polynomial subtraction