Supporting Information for: Molecular Motion of the Bis(maleonitriledithiolato)nickel Trianion in Solution

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Preparation of the Trianion, $[Ni(mnt)_2]^{3-}$, in Diglyme and DME by Reduction with Potassium Metal

The same procedure was used for both diglyme and DME. The sample cell, shown in Figure S1, was attached to the vacuum line. Stoppers were securely positioned in the left chamber, which contained $(Bu_4N)_2[Ni(mnt)_2]$, and in the right arm, which held several small pieces of potassium metal. The stopcock connecting the sample cell to the vacuum line was opened and the left chamber and right arm were sealed with a torch.

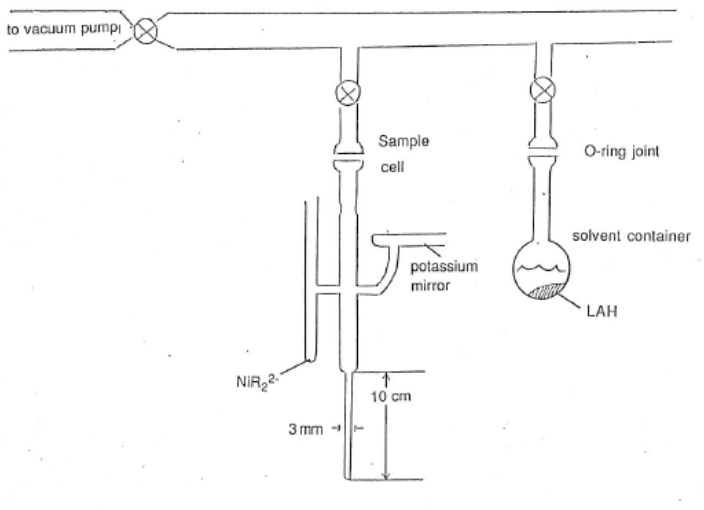

Figure S1. Vacuum line, ESR sample cell, and sample tube.

The stopcock connecting the sample tube to the vacuum was closed and solvent was distilled from the storage vessel into the chamber containing $(n\text{-}Bu_4)_2[Ni(mnt)_2]$, which was immersed in liquid nitrogen. While keeping the solvent frozen, the stopcock to the vacuum was opened; the metal was heated and formed a mirror as it moved along the tube. The portion of the tube from which the metal had been distilled was then sealed

and removed. The mirror was improved by being moved again, after which the portion of the tube in which the first mirror was formed was sealed and removed.

With the solvent still frozen, the stopcock connecting the sample cell to the vacuum was opened and the sample tube was sealed off from the vacuum line. The frozen solvent was allowed to melt; the trianion was formed when the solution was passed over the mirror while cooling the tube with glass wool dipped in a dry ice-acetone bath. After the reduction was complete, the solution was poured into the 3 mm o.d. sample tube, which was placed in the ESR spectrometer's pre-cooled cavity. The glassy spectrum of $[Ni(mnt)_2]^{3-}$ in diglyme was recorded at −98.0 °C (Figure 2). The temperature was then raised and motionally averaged spectra were taken in the temperature range $-81.0 \leq T \leq -15.5$ °C; they are shown in Figure S2 with the magnetic field increasing left-to-right. The singlet at high field has been assigned to the solvated electron.

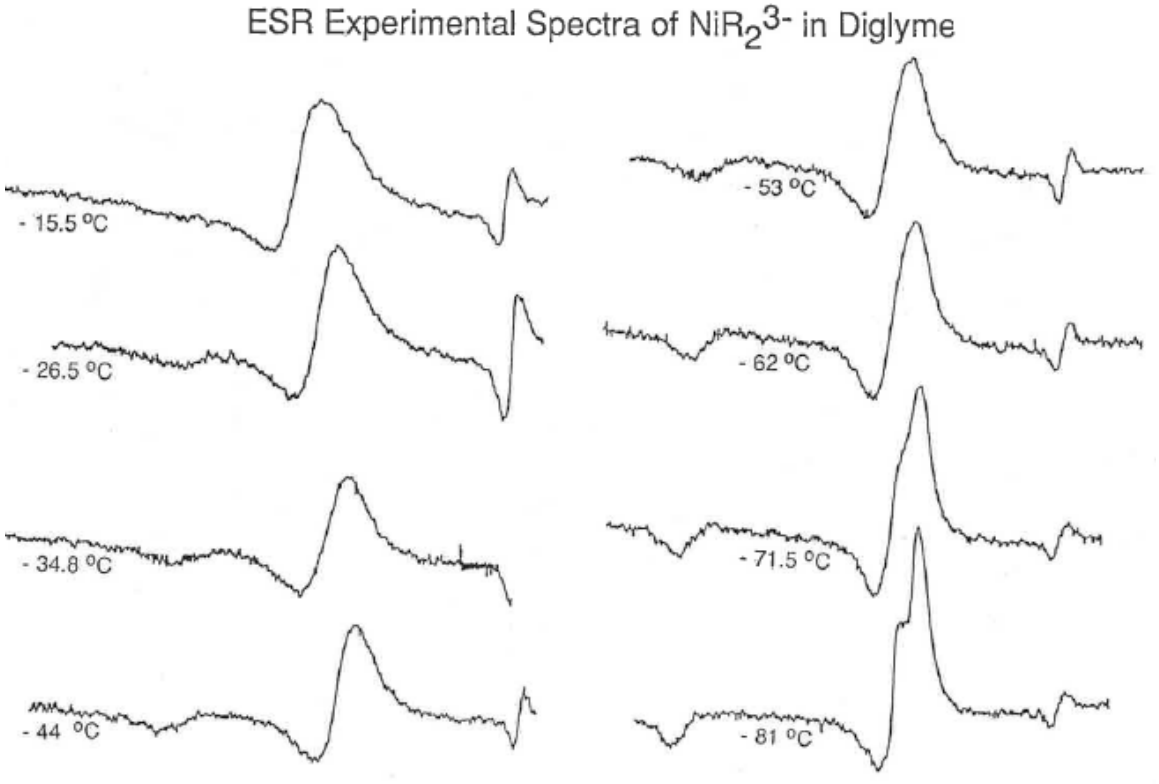

Figure S2. Temperature-dependent ESR spectra of $[Ni(mnt)_2]^{3-}$ in diglyme.