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Figure S1. Two-pulse ESEEM spectra and their simulations of $2 \mathrm{mM} \mathrm{VO}(\mathrm{Himac})_{2}$ in $\mathrm{H}_{2} \mathrm{O}$ /ethylene glycol 2:1 v/v glass. Conditions: $v=8.84 \mathrm{GHz}, T=77 \mathrm{~K}$; (a) $B=260.5 \mathrm{mT}$; (b) $B=305.5 \mathrm{mT}$; (c) $B=313.0 \mathrm{mT}$; (d) $B=318.5 \mathrm{mT}$. The peaks with $v_{\mathrm{H}}$ are due to matrix ${ }^{1} \mathrm{H}$ nuclei.


Figure $\mathbf{S 2}$. Three-pulse ESEEM spectra and their simulations of $2 \mathrm{mM} \mathrm{VO}(\mathrm{Himac})_{2}$ in $\mathrm{H}_{2} \mathrm{O}$ / ethylene glycol $2: 1 \mathrm{v} / \mathrm{v}$ glass. The $-1 / 2 \| \perp$ line was selected for measurements. Conditions: $v=8.84$ $\mathrm{GHz}, T=77 \mathrm{~K}, B=313.0 \mathrm{mT}$; (a) $\tau=300 \mathrm{~ns}$; (b) $\tau=380 \mathrm{~ns}$. The peak with $v_{\mathrm{H}}$ is due to matrix ${ }^{1} \mathrm{H}$ nuclei.


Figure S3. (a) Three-pulse ESEEM spectrum of 5 mM VO(salhisH)(acac) in $1: 1 \mathrm{v} / \mathrm{v}$ DMF:toluene glass. Conditions: $v=8.83 \mathrm{GHz}, T=77 \mathrm{~K}, B=305.6 \mathrm{mT}, \tau=310 \mathrm{~ns}$. (b) Spectrum after addition of one equiv. of aqueous HCl . Conditions: $v=8.84 \mathrm{GHz}, T=77 \mathrm{~K}, B=305.5 \mathrm{mT}, \tau=310 \mathrm{~ns}$. The peaks with $v_{\mathrm{H}}$ are due to matrix ${ }^{1} \mathrm{H}$ nuclei.

