

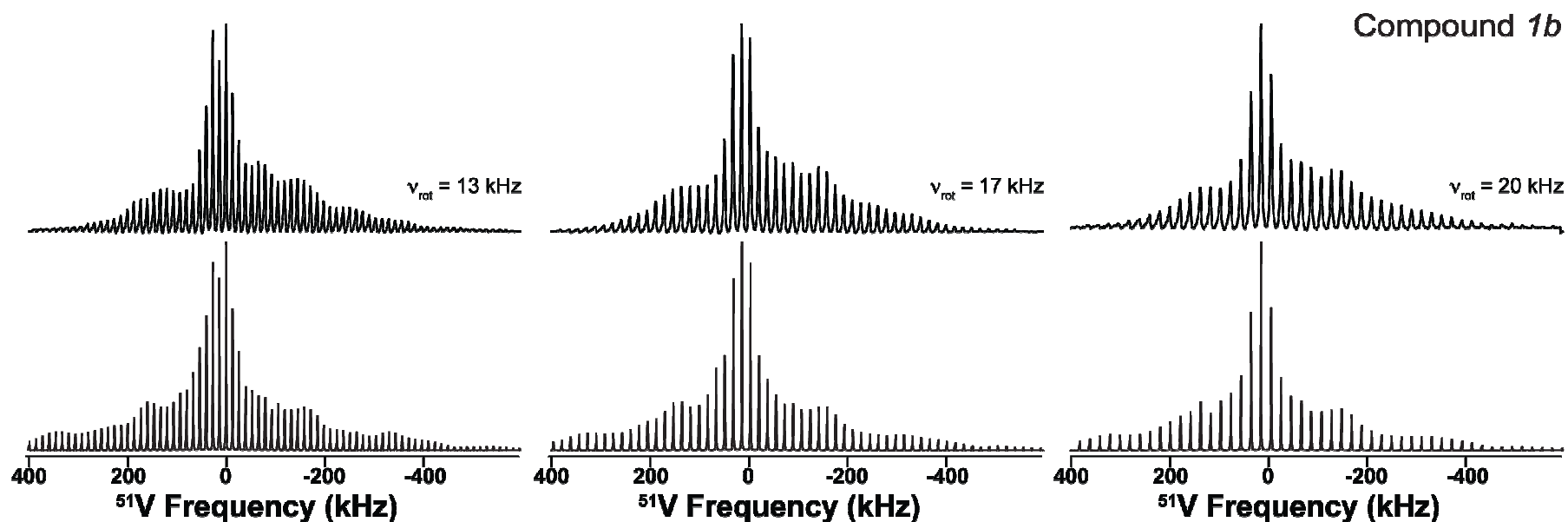
Supporting Information for

**Characterization of Non-Innocent Metal  
Complexes Using Solid-State NMR Spectroscopy:  
*o*-Dioxolene Vanadium Complexes**

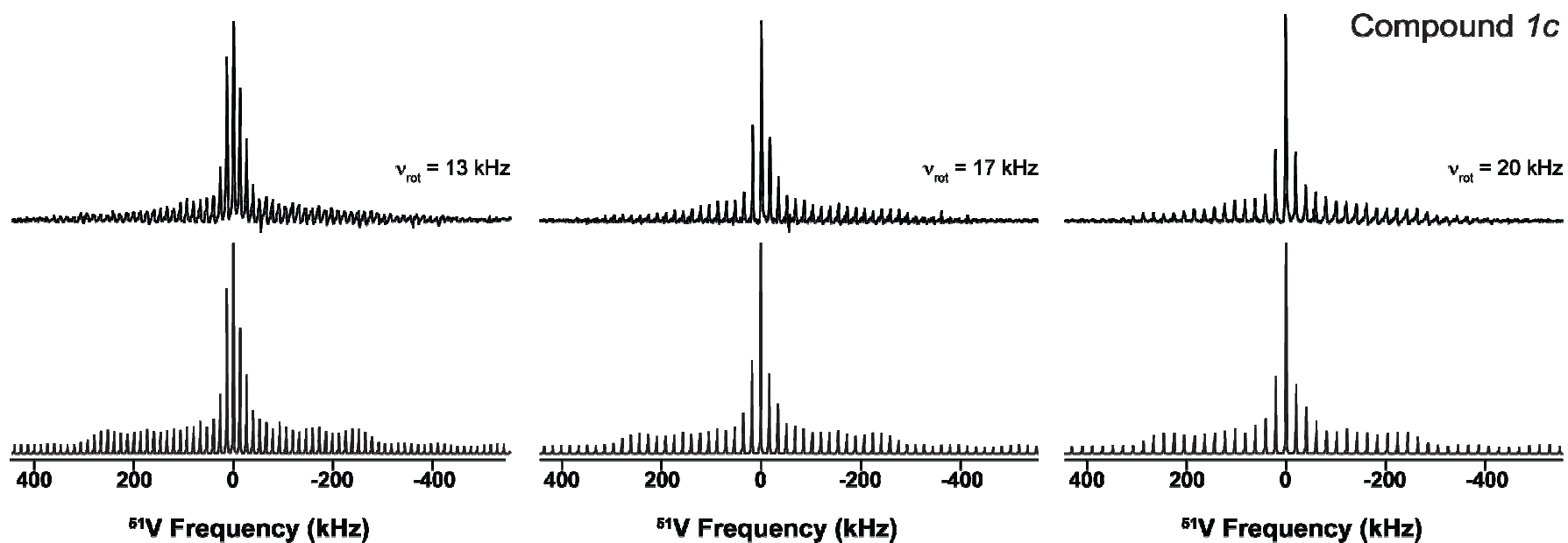
*Pabitra B. Chatterjee,<sup>†</sup> Olga Goncharov-Zapata,<sup>‡</sup> Laurence L.  
Quinn,<sup>†</sup> Guangjin Hou,<sup>‡</sup> Hiyam Hamaed,<sup>§</sup> Robert W. Schurko,<sup>§</sup> Tatyana Polenova,<sup>\*,‡</sup>  
and Debbie C. Crans<sup>\*,†</sup>*

<sup>†</sup>Department of Chemistry, Colorado State University, Fort Collins, CO 80523-1872, USA. <sup>‡</sup>Department of Chemistry and Biochemistry, University of Delaware, Newark, Delaware 19716, USA. <sup>§</sup>Department of Chemistry, University of Windsor, Windsor, Ontario, Canada N9B 3P4

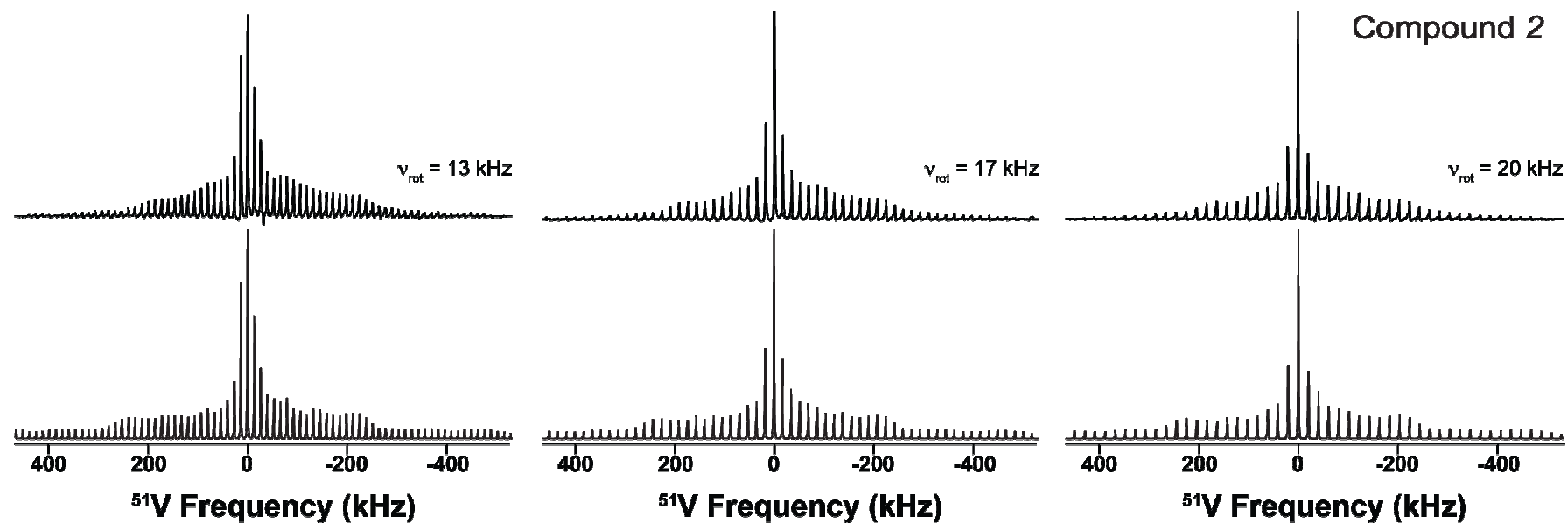
E-mail: [crans@lamar.colostate.edu](mailto:crans@lamar.colostate.edu); [tpolenov@mail.chem.udel.edu](mailto:tpolenov@mail.chem.udel.edu)



**Figure S1.**  $^{51}\text{V}$  experimental (top) and simulated (bottom) 9.4 T solid-state NMR spectra of the vanadium(V)-*o*-dioxolene compound  $\text{V}^{\text{V}}\text{O}(\text{hshed})(\text{DTBCat})$ - **1b**, acquired with MAS frequencies of 13 kHz (left), 17 kHz (middle) and 20 kHz (right). 8192 scans were accumulated for each spectrum, and the pulse delay was 1.0 s. The spectra were simulated using the parameters listed in Table 1.



**Figure S2.**  $^{51}\text{V}$  experimental (top) and simulated (bottom) 9.4 T solid-state NMR spectra of the vanadium(V)-*o*-dioxolene compound  $\text{V}^{\text{V}}\text{O}(\text{hshed})(\text{TBCat}) - \mathbf{1c}$  acquired with MAS frequencies of 13 kHz (left), 17 kHz (middle) and 20 kHz (right). 8192 scans were accumulated for each spectrum, and the pulse delay was 1.0 s. The spectra were simulated using the parameters listed in Table 1.



**Figure S3.**  $^{51}\text{V}$  experimental (top) and simulated (bottom) 9.4 T solid-state NMR spectra of the vanadium(V)-*o*-dioxolene compound VO(acac)(TCCat)-**2** acquired with MAS frequencies of 13 kHz (left), 17 kHz (middle) and 20 kHz (right). 8192 scans were accumulated for each spectrum, and the pulse delay was 1.0 s. The spectra were simulated using the parameters listed in Table 1.