

Supplementary Information for

Slow Scan Cyclic Voltammetry of Li-ion Insertion in T-Nb₂O₅ Reveals Hidden Peaks and Multi- Electron Redox

*Luke D. Salzer, Cami Christensen, Claire Gervais, Jacob D. Steeley, James R. Neilson, and
Justin B. Sambur**

*jsambur@colostate.edu

Department of Chemistry, Colorado State University, Fort Collins, CO 80523, USA

Table S1. Rietveld refinement results of the PXRD data in Figure 1 of the main text.

Refinement model	Mass fraction of T- Nb ₂ O ₅	Mass fraction of B- Nb ₂ O ₅	R _{wp} (%)
T-Nb ₂ O ₅	100%	n/a	10.739
T-Nb ₂ O ₅ & B-Nb ₂ O ₅	98.2%	1.8%	10.127

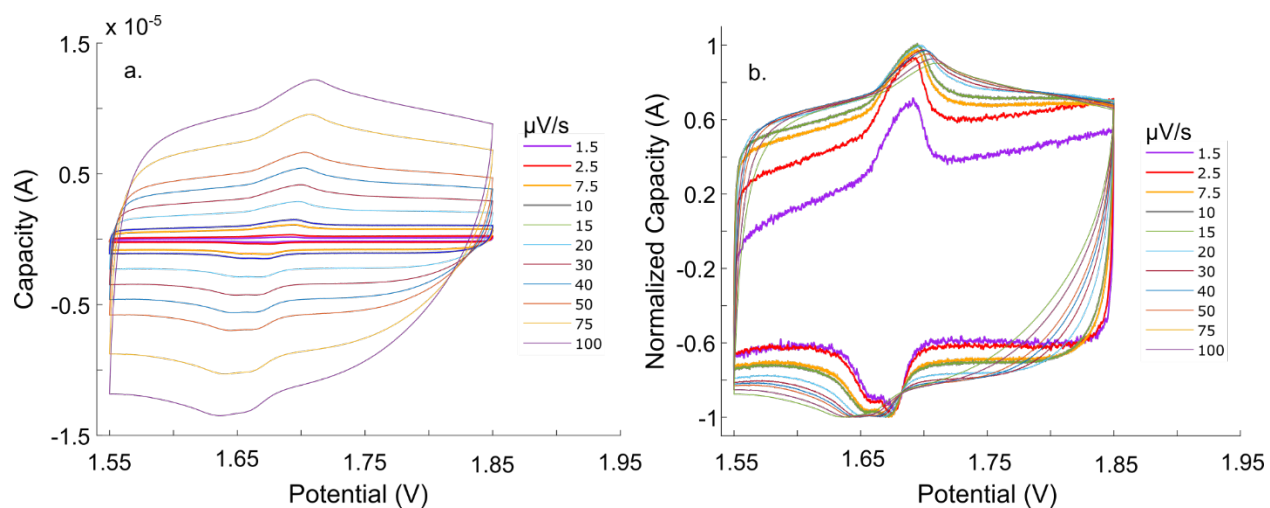


Figure S1. (a) SSCV of a T-Nb₂O₅ electrode that underwent 20 charge-discharge cycles from 3.0 to 1.2 V (b) Same data as in panel (a) normalized with respect to the maximum cathodic current.

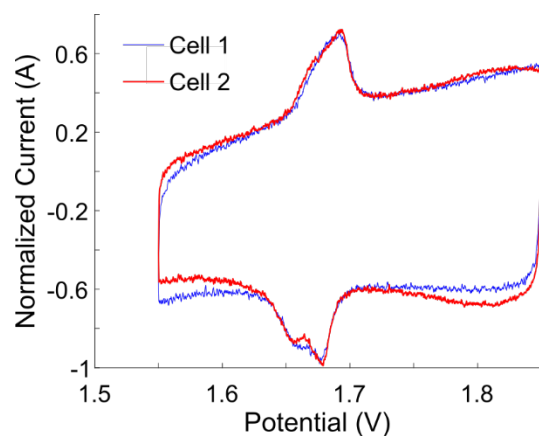


Figure S2. Comparison of SSCV data at at 1.5 μV/s for two different T-Nb₂O₅ electrodes that underwent 20 charge/discharge cycles from 3.0 V to 1.2 V in 1 M LiClO₄ in PC.

Figure S3. Estimated number of electrons stored in the T-Nb₂O₅ sample associated with $i_{p,c1}$ and $i_{p,c2}$ per number of Nb surface atoms. To calculate that charge-to-surface atom ration, we estimated the fraction of surface Nb atoms involved in charge storage by relating the total charge associated with the sharp peaks in Figure 3b (1.66×10^8 moles electrons or 9.99×10^{15} electrons) to the estimated total number of surface atoms in our T-Nb₂O₅ samples. The total solid volume was calculated from the sample mass (1.45×10^{-4} g) and the density of T-Nb₂O₅ (4.6 g cm^{-3}). Assuming the particles are monodisperse, pseudospheres with diameters between 100 nm and 5000 nm, the number of particles was obtained by dividing the total solid volume by the volume of a single spherical particle. The total surface area was then determined by multiplying the number of

particles by the surface area of a single sphere. The total number of surface Nb atoms was estimated by assuming a surface Nb areal density of 7 atoms nm^{-2} , consistent with the atomic packing density of Nb_2O_5 surfaces. Finally, the ratio of stored electrons to surface Nb atoms was calculated for each assumed particle size.

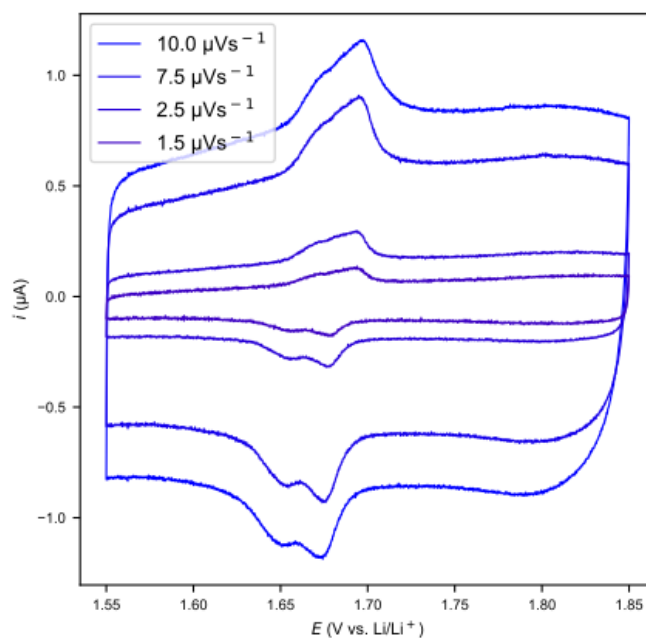


Figure S4. Selected SSCV data from Figure 3a of the main text, obtained using a T- Nb_2O_5 electrode that underwent 20 charge-discharge cycles from 3.0 to 1.2 V in 1M LiClO_4 in PC.