# Thermal Decomposition of Neptunyl Ammonium Nitrate: Mechanistic Insights and Structural Characterization of the $Np_2O_5$ Intermediate Phase

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SUPPLEMENTARY INFORMATION

**Single Crystal XRD**

Feed solutions from the MDD production line were dried and crystals were placed in microscopy oil on a glass slide. From this material, a crystal suitable for scXRD was isolated using an optical microscope and mounted onto a Mitegen cryoloop. Once selected, this crystal was coated in 1-minute bonding epoxy after binding agent was added and was allowed to cure. These cryoloops were decontaminated and moved from the radiological fume hood to the laboratory with the scXRD instrument, where the samples were mounted and exposed to the X-rays for 4 seconds per 0.5° 2θ frame for a full hemisphere collection on the Bruker D8 Venture diffractometer, equipped with an Iμs 3.0 molybdenum X-ray source (λ = 0.71073 Å). The structure was solved using OLEX2 for structural parameter refinement and atom assignment.[1]

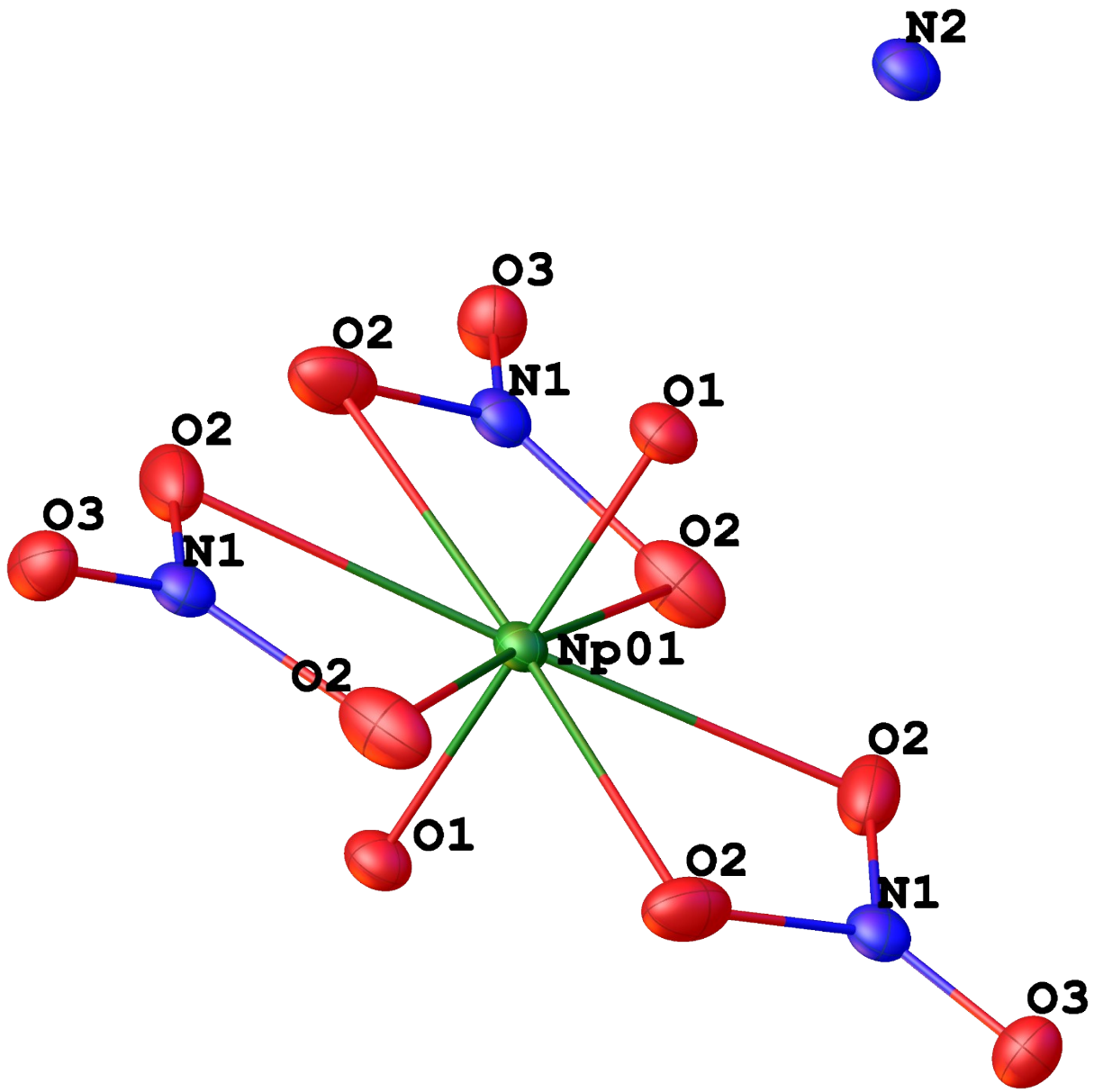


**Figure S1.** Crystal structure for $(NH_4)(NpO_2)(NO3)_3$ crystallized from feed solution. Major differences from the Autillo (2022) structure are a contracted unit cell with higher symmetry and the coordination of N2 along the neptunyl axis instead of off-axis nearer to O3. Hydrogen atoms are excluded from the ammonium atom (N2), as X-ray diffraction cannot reliably identify H in a structure.

## References

(1) Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H. OLEX2: a complete structure solution, refinement and analysis program. *Journal of Applied Crystallography* **2009**, *42* (2), 339-341. DOI: doi:10.1107/S0021889808042726.