Adventures in Solving Structures of Inorganic Compounds Using Powder Diffraction Data

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Anhydrous hygroscopic $Pd(NO_3)_2$ can be prepared by evaporating a commercial palladium nitrate solution to dryness in a vacuum oven at 50-60°C. It crystallizes in space group $P2_1/a$, with a = 10.0886(10), b = 5.395(6), c = 5.7484(5) Å, β = 97.377(7)°, V = 310.28(5) Å^3, and Z = 2. The Pd was placed at the origin, and the N and O atoms located by difference Fourier techniques. The structure consists of discrete planar $Pd(NO_3)_2$ molecules. The molecular solid is an insulator with a bandgap of ~2.3 eV.

An attempt to prepare a magnesium vanadate using hydrothermal techniques yielded a hygroscopic new compound. The pattern could be indexed in space group *Cmcm* with a = 6.3727(7), b = 13.5715(8), c = 6.3657(4) Å, and V = 550.56(8) Å^3. The structure was solved by direct methods and difference Fourier techniques. The Rietveld refinement clearly indicated that the compound $(Mg_{0.37}V_{0.63})O_{0.63}(SO_4)(H_2O)_{1.5}$ has a layered structure.

The powder pattern of the potential thermoelectric material $NaGe_4$ could be indexed on a primitive hexagonal cell. The structure was solved and refined in *P6/m* with a = 15.05399(5), c = 3.96845(2) Å, and V = 778.852(4) Å^3. It consists of a zeolite-like Ge framework, with partially-occupied sites in a large 24-ring channel. Progress on the crystal structure of the mineral charoite, $K(Ca,Na)_2Si_4O_{10}(OH,F)(H_2O)$, will also be discussed.

Keywords: palladium nitrate, vanadium sulfate hydrate, sodium germanium