C2 diffractometer demonstration

Structural transitions in metal hexaflurophosphates [MPF_6]

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The basics of a neutron powder diffractometer will be discussed including the choice of spectrometer parameter to measure magnetic vs nuclear reflections. The effects of choosing neutron wavelength on the observed diffraction pattern will be reviewed. We will also demonstrate how to analyse the observed pattern in order to identify the structure of the material under study by the Rietveld method.

At room temperature, most univalent metal hexafluorophosphates have a cubic structure based on the NaCl structure, where the PF_6 molecular anion replaces Cl in the lattice. These simple structures undergo a variety of transitions on cooling and under pressure that have still not been systematically studied. These transitions are defined by a tilt (rotation) of the PF_6^- anion and a coupled spontaneous strain (i.e. distortion of the lattice with respect to the high-temperature parent phase).

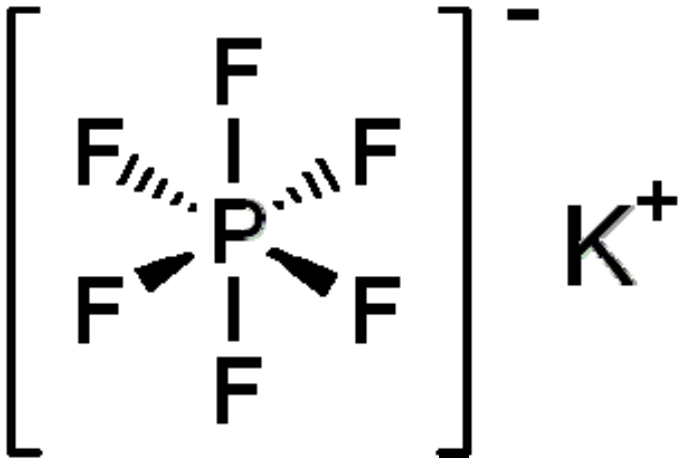

Figure 1: Potassium hexafluorophosphate.

KPF_6 undergoes two transitions on cooling at ambient pressure, one at ca. 273 K to a form that is reportedly rhombohedral, and another below 258 K to another structure [neither structure has been published].

NH_4PF_6 also undergoes a couple of transitions. The upper transition was understood to be rhombohedral for a long time, but was eventually shown to be a slight monoclinic distortion of this. The lowest-temperature phase is also monoclinic, and it appears to be of the same space group and basis as the intermediate phase; i.e., the transition is isosymmetric.

We will start of quickly with an overview of the neutron powder diffractometer, the ancillary equipment quickly measure the diffraction patterns of KPF_6. Time permitting, we will discuss the problems of approaching an unknown phase, indexing, and refining the crystal structure by the Rietveld method (we may use some previously taken data from ND_4PF_6 for comparison), and some ideas of symmetry changes associated with phase transitions.