The phosphates of transition metals form a huge family of compounds. The association of metallic or semi-metallic rows or layers of M\text{O}_6 octahedra with insulating PO_4 rows or layers gives rise to original physical properties. Moreover, it is well known that many phosphates are good ionic conductors. Due to these reasons, a great effort, during the last years, has been devoted to the study of mixed transition-metal phosphates.

Single crystals of the new Co(II) phosphate Na_4Co_3(PO_4)Pz_07 have been isolated and their structure has been determined by X-ray diffraction techniques. The structure of this compound is built up from corner- and edge-sharing between CoO_6 octahedra and PO_4 and PzO_5 groups giving rise to a polyhedral connectivity which produces large tunnels running along the three main crystallographic directions. The structure consists of infinite layers with composition (Co_3Pz_013)\_oo parallel to the bc plane. Interlayer linkages are made via P-O-P bridges of the pyrophosphate groups in such a way that large tunnels extending along the [010] and [001] directions occur between two neighbouring sheets. Besides, there also exist channels along the [100] direction consisting of (Co_3Pz_013)\_oo layers. A complex scheme of tunnel intersections gives rise to the formation of a three-dimensional channel network which hosts the sodium cations.

This compound crystallizes in the orthorhombic noncentrosymmetric space group Pn2_1a with a=18.046(5)\,\text{\AA}, b=6.533(2)\,\text{\AA}, c=10.536(2)\,\text{\AA}, V=1242.1(5)\,\text{\AA}^3 and Z=4. The structure was refined from 1791 reflections with \( \text{R}(\text{ref}) = 0.039 \).

**PS08.00.41 CRYSTALLOGRAPHIC STUDIES OF BROOKITE(TiO_2) WITH SYNCHROTRON RADIATION.**

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X-ray diffraction studies on single crystals of brookite(TiO_2) with synchrotron radiation have been performed. It is found that extra reflections are observed, which is forbidden by the extinction rule for the brookite space group. It is well known brookite crystal have two kinds of crystal habit, one is platy and the other is pyramidal. This is an interesting problem why two kinds of crystal habit are observed from the point of crystal morphology. Hartman suggested that is based on stepwise truncation of low order data. We think it may be possible to observe structural distortion caused by such epitastral growth by using strong X-ray source, so I carry out single crystal X-ray diffraction studies of brookite with synchrotron radiation. A crystal with pyramidal habit from Magnet Cove, Arkansas, USA used for diffraction studies. The diffraction experiments were done with vertical type 4-circle diffractometer at BL-10A, Photon Factory, National Laboratory for High Energy Physics, using radiation of the 0.7\,\text{\AA} wavelength. Intensity data of 1557 reflections were collected from the following range: 2\theta < 65\,\text{degrees}, -17 < h < 17, 0 < k < 16. As the results, some forbidden reflections for brookite space group Pbcn, such as h00, k00, 00h, k, j=2n+1, 0kh=2n+1, b01l=2n+1 or hkt=2n+1, are observed. The intensity of these reflections is lower than that of the fundamental reflection by three orders of magnitude. There is no reflection that suggests existence of super lattice by measurements of diffraction profile along h00. Diffraction studies using a platy habit single crystal are in progress. The reason these extra reflections appear and the relationship between the crystal habit and these extra reflections will be discussed.

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**PS08.00.42 NiAl_3: A STRUCTURE TYPE OF ITS OWN?**

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Generally, the intermetallic compound NiAl_3 is counted as a representative of the Fe3C (cementite) structure type. We grouped the almost 200 different compounds with the cementite structure type according to geometrical and chemical parameters and found that NiAl_3 is not fitting the values typical of this structure type. NiAl_3 was prepared by slowly annealing an Al-rich melt and dissolving excess Al in dilute hydrochloric acid. The crystal structure was redetermined from single-crystal data: \( p\,\text{m} \), \( a=6.613(1)\,\text{\AA}, b=7.367(1)\,\text{\AA}, c=4.811(1)\,\text{\AA}, Z=4 \).

Compared to the parent structure Fe3C, the coordination polyhedron for the minority component changes from a trigonal prism with nearly equal edges to a distorted prism with all quadrilateral faces capped.

We suggest that NiAl_3 is better described by a structure type of its own rather than by the cementite structure.