

08.2-44 THE CRYSTAL STRUCTURE OF IRON(II)-DI-HYDROGEN PHOSPHATE DIHYDRATE ($\text{Fe}(\text{H}_2\text{PO}_4)_2 \cdot 2\text{H}_2\text{O}$). By W. Guse, K.-H. Klaska, H. Saalfeld, G. Adiwidjaja. Mineralogisch-Petrographisches Institut der Universität Hamburg, 2 Hamburg 13, W.-Germany.

Single crystals of the title compound were grown from the solution. The crystal structure was determined by X-ray diffraction. $\text{Fe}(\text{H}_2\text{PO}_4)_2 \cdot 2\text{H}_2\text{O}$ crystallizes in the monoclinic space group $P2_1/m$ with two formula units per unit cell, of which dimensions are: $a=7,297 \text{ \AA}$, $b=9,925 \text{ \AA}$, $c=5,337 \text{ \AA}$, $\beta=95,13^\circ$; $d_m=2,43 \text{ g/cm}^3$, $d_{\text{cal}}=2,46 \text{ g/cm}^3$. The structure was refined with 1034 observed reflections to $R=0,018$. Corrections were made for absorption and extinction. Atomic coordinates, anisotropic thermal parameters, bond lengths and bond angles will be presented. The structure of $\text{Fe}(\text{H}_2\text{PO}_4)_2 \cdot 2\text{H}_2\text{O}$ contains layers parallel (101), which consist of distorted FeO_6 -octahedra ($\text{Fe}(40+2\text{O}_w)$) and PO_4 -tetrahedra ($\text{P}(\text{O}_2+2\text{O}_H)$). The connection between the corner sharing octahedra and tetrahedra is given by the two tetrahedra oxygen atoms (O_2). A report upon structural details of the isotype compound $\text{Cd}(\text{H}_2\text{PO}_4)_2 \cdot 2\text{H}_2\text{O}$ was given by M.T. Averbuch-Pouchot et al. (Bull. Soc. fr. Minéral. Cristallogr. (1973), 96, 278) without the consideration of the hydrogen atoms. The results of this paper include the arrangement of the hydrogen atoms solved by a Fourier-difference synthesis.

08.2-45 DODECAHEDRAL AND SQUARE ANTIPRISMATIC 8-COORDINATION IN THREE NEW OCTACYANOMOLYBDATES (IV). By W. Meske and D. Babel, Sonderforschungsbereich 127 and Fachbereich Chemie der Philipps-Universität, Marburg, Germany

The crystal structures of the following compounds were determined:

$(\text{NMe}_4)_3\text{LiMo}(\text{CN})_8 \cdot 3,5\text{H}_2\text{O}$, $P\bar{4}2_1m$, $Z=4$, $R_g=0,046$ (1862 hkl)
 $a=17,075 \text{ \AA}$, $c=10,548 \text{ \AA}$, $V=3075,3 \text{ \AA}^3$
 $\text{Cs}_7\text{Na}(\text{Mo}(\text{CN})_8)_2 \cdot 4,17\text{H}_2\text{O}$, $I4_1/a$, $Z=8$, $R_g=0,036$ (3100 hkl)
 $a=15,479 \text{ \AA}$, $c=32,546 \text{ \AA}$, $V=7798,0 \text{ \AA}^3$
 $\text{Ti}_4\text{Mo}(\text{CN})_8$, $C2/c$, $Z=8$, $R_g=0,049$ (1749 hkl)
 $a=30,877$, $b=7,348$, $c=16,126 \text{ \AA}$, $\beta=121,0^\circ$, $V=3136,1 \text{ \AA}^3$

Approximate dodecahedral geometry of the $\text{Mo}(\text{CN})_8^{4-}$ group, as reported for most octacyanomolybdates(IV) (P.M. Kierman and W.P. Griffith, Inorg. Nucl. Chem. Letters (1976) 12, 377, S.S. Basson et al., Acta Cryst. (1980) B36, 2025 and Inorg. Chim. Acta (1980) 44, L99), is observed also in $(\text{NMe}_4)_3\text{LiMo}(\text{CN})_8 \cdot 3,5\text{H}_2\text{O}$.

The two other compounds provide new examples of approximate square antiprismatic coordination, only found in $\text{Cd}_2\text{Mo}(\text{CN})_8 \cdot 2\text{N}_2\text{H}_4 \cdot 4\text{H}_2\text{O}$ hitherto (J. Chojnacki et al., Roczniki Chem. (1969) 43, 273).

In the three new structures the Mo-C distances vary but slightly around the average of $2,165 \text{ \AA}$, which is the same within $\pm 0,005 \text{ \AA}$ in all three compounds. The structural relations and cation coordinations of octacyanomolybdates are discussed.

08.2-46 THE CRYSTAL STRUCTURES OF BaCdGaF_7 AND BaCaGaF_7 , SHOWING THE INFLUENCE OF INCREASING M(II) ION SIZE IN COMPOUNDS $\text{BaM}^{\text{II}}\text{M}^{\text{III}}\text{F}_7$. By H. Holler and D. Babel, Sonderforschungsbereich 127 and Fachbereich Chemie der Philipps-Universität Marburg, Germany

Two new structure types of compounds $\text{BaM}^{\text{II}}\text{M}^{\text{III}}\text{F}_7$ have been determined and refined to $R \approx 0,03$ (using about 1800 reflections):

	S.G.	Z	c(pm)	b(pm)	c(pm)	$\beta(^{\circ})$
BaCdGaF_7	C2/c	8	1389.6	535.4	1495.0	91.55
BaCaGaF_7	P2/n	4	539.0	541.0	1897.8	92.33

Contrary to the framework structures of BaMnFeF_7 (H. Holler et al., J. Solid State Chem. (1981) 39, 345) and HT-BaZnFeF_7 (Z. anorg. allg. Chem. (1982) 491, 137), in which all M(II) and M(III) ions are octahedrally coordinated, the above compounds have layer structures and exhibit distorted square antiprismatic 8-coordination for half the M(II) ions in BaCdGaF_7 (H. Holler et al., Rev. Chim. Miner. (1984), in press), and all of them in BaCaGaF_7 . The resulting average distances (pm) are:

C.N. in ():	Ba-F(12)	Ga-F(6)	M-F(6)	M-F(8)
BaCdGaF_7	290.7	188.8	222.4	237.2
BaCaGaF_7	289.4	187.8	-	236.3

The lattice constants and results from additional investigations (structure determinations, high pressure work, EPR and Mössbauer spectra) on isostructural compounds are given. The structural relations among heptafluorodimetalates are discussed.

08.2-47 ORTHORHOMBIC KCuPO_4 : SYNTHESIS AND CRYSTAL STRUCTURE. By H. Effenberger, Institut für Mineralogie und Kristallographie, Universität Wien, Vienna, Austria.

Crystals of the orthorhombic phase of KCuPO_4 were synthesized from $\text{KH}_2\text{PO}_4 + \text{KNO}_3 + \text{Cu}$ under hydrothermal conditions at 493 K. The crystal structure was determined from 1285 unique X-ray data (two circle diffractometer) and refined to $R = 0,031$ in space group Pbca with $a = 17,94(2) \text{ \AA}$, $b = 6,742(6) \text{ \AA}$ and $c = 6,795(6) \text{ \AA}$ by $Z = 8$.

The Cu atom is coordinated by four O atoms with a mean Cu-O distance of $1,950 \text{ \AA}$, a fifth O atom with Cu-O = $2,506 \text{ \AA}$ completes the coordination figure. The phosphate tetrahedron is characterized by distinct P-O distances of $1,503$, $1,546$, $1,555$ and $1,591 \text{ \AA}$ due to the different coordination of the oxygen atoms.

The Cu polyhedron shares one O-O edge with a phosphate tetrahedron. The connection of these structure units over common oxygen atom corners results in $\infty [\text{CuPO}_4]^{1-}$ sheets parallel (100) as known from $\text{KCuPO}_4 \cdot \text{H}_2\text{O}$ [Brunel-Laügt & Tordjman (1976), Acta Cryst. B32, 203].

The K atom is irregularly coordinated by seven O atoms: six K-O bonds lie within one sheet; the seventh K-O bond is the only conjunction of adjacent sheets. This is in agreement with the good cleavage parallel (100). Contrary the high temperature phase of KCuPO_4 (monoclinic) represents a network type structure [Shoemaker, Kostiner & Anderson (1980), Z. Krist. 152, 371].