08.2-44 THE CRYSTAL STRUCTURE OF IRON(III)-DIHYDROGEN PHOSPHATE DIHYDRATE Fe(H₂PO₄)₂·2H₂O. By W.Guse, K.-H.Klaaska, R.Saalfeld, G.Adolf, A. Münch, and H. Münch. 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. Fe(H₂PO₄)₂·2H₂O crystallizes in the monoclinic space group P2₁/n with two formula units per unit cell, of which dimensions are: a=7.297 Å, b=9.925 Å, c=5.337 Å, β=95.13°. The structure was refined with 1034 observed reflections to R=0.018. 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 OCTACYANOMOLYBDATE(VI). By W. Maske and D. Babel, Sonderforschungsbereich 127 and Fachbereich Chemie der Philipps-Universität, Marburg, Germany.

The crystal structures of the following compounds were determined:
- (NM₄)₂[N₆(CN)₂]·3.5H₂O, P₂₁/n, Z=4, R=0.046 (1982 hkl)
- Cs₄Na[Mo(CN)₈]·1.7H₂O, I₄/a, Z=8, R=0.035 (3100 hkl)
- Ti₄[N₆(CN)₂]₂·C₁₂H₂O, P₄/nm, Z=4, R=0.036 (1749 hkl)


The two other compounds provide new examples of approximately square antiprismatic coordination, only found in Cd₄[N₆(CN)₂]·4H₂O hitherto (J. Chojecki 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 Å, which is the same within ± 0.005 Å in all three compounds. The structural relations and cation coordinations of octacyanomolybdates are discussed.

08.2-46 THE CRYSTAL STRUCTURES OF BaMgF₂₇ AND BaCaF₂₇, SHOWING THE INFLUENCE OF INCREASING M(I)ION SIZE IN COMPOUNDS BaMgF₂₇, BaCaF₂₇, AND BaGaF₂₇. By H. Holler and D. Babel, Sonderforschungsbereich 127 and Fachbereich Chemie der Philipps-Universität, Marburg, Germany.

Two new structure types of compounds BaMgF₂₇ have been determined and refined to R~0.03 (using about 1800 reflections):
- S.G. Z (cm³) b(pm) c(pm) α(°)
  BaMgF₂₇ C2/c 8 1389.6 535.4 1495.0 91.56
  BaCaF₂₇ P2/n 4 539.0 541.0 1897.8 92.33

Contrary to the framework structures of BaMgF₂₇ (H. Holler et al., J. Solid State Chem. (1981) 43, 345) and HT-BaZnF₂₇ (2, anorg. allg. Chem. (1982) 191, 127), in which all M(I) and M(II) ions are octahedrally coordinated, the above compounds have layer structures and exhibit distorted square antiprismatic 8-coordination for half the M(I) ions in BaCaF₂₇ (H. Holler et al., Rev. Chim. Miner. (1984), in press) and all of them in BaMgF₂₇. The resulting average distances (pm) are:
- C.N. in (): Ba-S(12) 280(6) M-N(6) 237.2
- BaCaF₂₇ 289.4 187.8 - 236.3

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

08.2-47 ORTHORHOMBIC KCuPO₄: SYNTHESIS AND CRYSTAL STRUCTURE. By H. Effenberger, Institut für Mineralogie und Kristallographie, Universität Wien, Vienna, Austria.

Crystals of the orthorhombic phase of KCuPO₄ were synthesized from KH₂PO₄ + KN₃ + Cu in 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 Pbcn with a = 17.242(2) Å, b = 6.742(6) Å and c = 6.795(6) Å by R = 8. The Cu atom is coordinated by four O atoms with a mean Cu-O distance of 1.950 Å, a fifth O atom with Cu-O = 2.506 Å completes the coordination.

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 [CuPO₄]₄⁴⁻ sheets parallel (100) as known from KCuPO₄·H₂O (Bräunel-Lalotte & Torjdman (1976), Acta Cryst. B32, 801).

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 KCuPO₄ (monoclinic) represents a network type structure (Rashkarev, Kozhukhov & Anderson (1980), Z. Kristallogr. 161, 371).