

Ca(ReO₄)₂.Urea.H₂O: triclinic, P $\bar{1}$, $a = 7.322(3)$, $b = 9.088(5)$, $c = 9.175(3)$ Å, $\alpha = 111.67(2)$, $\beta = 91.26(2)$, $\gamma = 104.89(3)^\circ$, $Z = 2$, $D_x = 3.67$ g.cm⁻³, $R = 0.030$ for 2104 3 σ -reflections.

Pb(ReO₄)₂.Urea.H₂O: monoclinic, P 2₁/c, $a = 10.283(2)$, $b = 7.389(6)$, $c = 14.402(8)$ Å, $\beta = 99.72(1)^\circ$, $Z = 4$, $D_x = 4.727$ g.cm⁻³, $R = 0.042$ for 1951 3 σ -reflections.

The CaO₈ environment consists of five apices of the ReO₄ tetrahedra [2.43(1)-2.52(2)Å], O atoms of two urea and one water molecules at distances 2 x 2.41(1) and 2.39(1)Å. Ca atoms are chained by alternating double Ca-O-Ca bridges of urea [Ca...Ca 3.92(1)Å] and fourfold Ca-(OReO)-Ca bridges [Ca...Ca 5.49(3)Å]. Adjacent chains are linked by pairs of Re(1)O₄ tetrahedra [Ca...Ca 6.85(2)Å] and arranged in layers parallel to the *ab*-plane. The layers are packed by H-bonds only.

The Pb atom has a nine-fold coordination of six ReO₄ apices (Pb...O 2.55(2)-2.76(1)Å), two urea molecules [2.61(1), 2.86(1)Å], and a water molecule at 2.47(1)Å. The structure consists of layers parallel to the *bc*-plane in which the Re(1)O₄ tetrahedra form double bridges between Pb atoms (Pb...Pb 4.12(2), 6.64(2), 2 x 6.85(2)Å). The shortening of the first Pb...Pb distance is due to the complementary Pb-O-Pb urea double bridge. The layers are held together by double bridges of Re(2)O₄ tetrahedra [Pb...Pb 7.45(4)Å] and a system of H-bonds.

PS-08.01.34 NEW MEMBERS OF TRIPLE MOLYBDATE FAMILY: COMPOSITIONS, CRYSTAL STRUCTURES, THERMAL STABILITY. By R.F.Klevtsova*, L.A.Glinskaya, N.M.Kozhevnikova, Zh.G.Bazarova, P.V.Klevtsov. Institute of Inorganic Chemistry, Russian Academy of Sciences, Siberian Branch, Russia.

Triple molybdates were synthesized by solid state reactions and the complicated character of chemical interactions in the systems was established. Products of the synthesis were investigated by X-ray and DTA methods. Single crystals were obtained by spontaneous flux crystallization. The compositions are: I-Li₃Ba₂Im₃(MoO₄)₈, Im=Gd,Tm; II-NaMg₃In(MoO₄)₅, III-K₁₀MgZr₃(MoO₄)₁₂. Their crystallographic characteristics are given in the Table.

	Sp.gr.	a, Å	b, Å	c, Å	Z	N	R
I-Gd	C2/c	5.2380	12.758	19.151	2	2453	0.042
		-	91.126	-			
II	P $\bar{1}$	7.0476	17.935	6.9849	2	4503	0.047
		87.650	100.96	92.510			
III	R3c	10.576	-	37.511	3	1166	0.026

The crystal structures were solved using single crystal data (KUMA and CAD-4, Mo K α radiation). The characteristic features of the structures are statistically distributed atoms of bi- and trivalent metals. Structural peculiarities (size, shape, site occupancy and stacking of the coordination polyhedra) are used for interpretation of physical properties. The comparative crystal chemistry analysis of known triple molybdates has been carried out. The compounds studied melt incongruently, polymorphism was not found.

PS-08.01.35 STRUCTURAL CHEMISTRY OF BaAl₄/ThCr₂Si₂ TYPE COMPOUNDS. By G.Just and P.Paufler, Institute of Crystallography and Solid State Physics, University of Technology, Dresden, Federal Republic of Germany.

Lattice ratios *c/a* and structural parameters x_3 of about 180 representatives of the BaAl₄ type of structure (space group I 4/mmm) have been critically reviewed taking binary and multicomponent phases into account. Leaving out data of minor reliability a considerable scatter of experimental values in a x_3 -*c/a* plane remains, which is classified according to the concepts of equal interatomic spacing and coordination polyhedra. Lines of equal spacings *d* were calculated for all atom positions in the unit cell up to $d = 1.9$ Å. Moreover, the coordination of all atom positions is given. Several points in the x_3 -*c/a* plane were found to represent special structural features like coordination polyhedra with coordination numbers of 16, 18 or 20 for the barium position with characteristic packing of distorted tetrahedra. Individual space requirements of atoms are met with different spacing conditions between Al(1) and Al(2) positions. Subdivision of representatives into families behaving similarly from a point of view of crystal chemistry is discussed.

PS-08.01.36 NON-STOICHIOMETRIC V-Mo OXIDE COMPOUNDS WITH V₂O₅ STRUCTURE TYPE. By L. M. Plyasova, L. P. Solovyeva*, G. N. Kryukova, S. V. Tsybulya and T. V. Andrushkevich.

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The V-Mo oxide system was studied by X-ray analysis because such oxides serve as catalysts for oxidation of acrolein to acrylic acid. Thermal treatment of products from reaction between para-molybdate and meta-vanadate resulted in the formation of V-Mo compounds of varying composition (V₆Mo₄O₂₅, (V⁵⁺, V⁴⁺)MoO₅, VMo₃O₁₁, etc.). The structures of these compounds appear to depend on the degree of vanadium reduction and on V/Mo ratio. V_{0.95}Mo_{0.97}O₅ (V⁴⁺/V=0.6) was selected for structural investigations. Crystal structure analysis and refinement using Rietveld method ($R_1 = 0.072$, $R_p = 0.11$) were carried out using X-ray powder diffraction data. Layers of edge-sharing octahedra of two types (Mo+V⁴⁺) and (Mo+V⁵⁺) are connected by corners. Structurally the compound is similar to the V₂O₅ type. Our results indicate that for compounds of V₂O₅ structure

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type the increase of Mo and V⁴⁺ content gives rise to the uniformity of Me-O bonds. Connections between octahedron layers tend to release. Disorder becomes most pronounced when the compound possesses a VM₃O₁₁ composition. The peculiarities of the VM₃O₁₁ structure are assumed to provide its best catalytic properties among V-Mo oxide compounds in acrolein oxidation to acrylic acid.

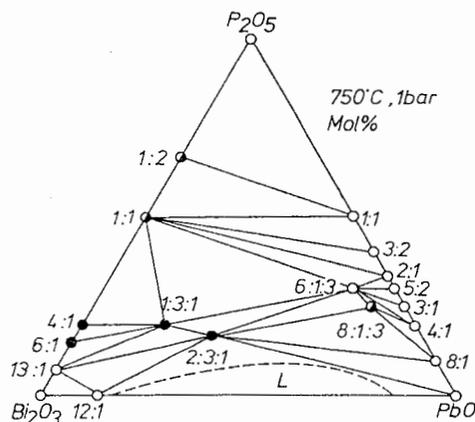
*Alternative cell: a = 9.814Å, b = 11.440Å, c = 6.550Å, β = 94.27°

layer structures containing [Bi₂O₂] layers. Structure determinations are in preparation. Corresponding phosphates, arsenates and vanadates form solid solutions.

The authors thank the ICDD for financial support.

PS-08.01.37 NEW COMPOUNDS IN THE SYSTEMS Bi₂O₃-PbO/SrO-P₂O₅/As₂O₅/V₂O₅. By Y.C. Jie and W. Eysel, Mineralogisch-Petrographisches Institut, Universität Heidelberg, Germany

The system Bi₂O₃-PbO-P₂O₅ was investigated between 600°C and 1000°C in air. The new phosphates PbBi₃PO₈, PbBi₆P₂O₁₅ and Bi₁₂P₂O₂₃ (full dots in the figure) were characterized by X-ray diffraction. For some other compounds (half



dots) high precision powder data were determined. For the new phosphates isostructural arsenates and vanadates as well as Sr compounds were synthesized (Table 1). According to the lattice parameters the various tetragonal, orthorhombic and monoclinic structures are closely related. The (pseudo-)tetragonal lattice parameter a ≈ 11.7 = 3 x 3.9 Å indicates

Table 1 Structural data of new compounds. T = tetragonal, O = orthorhombic, M = monoclinic

Compounds	Sym.	a(Å)	b(Å)	c(Å)	β(°)
PbBi ₃ PO ₈	T, I	11.756		15.604	
PbBi ₃ AsO ₈		11.816		16.054	
PbBi ₃ VO ₈		11.838		16.066	
SrBi ₃ PO ₈	T, I	11.620		16.973	
SrBi ₃ AsO ₈		11.769		16.439	
SrBi ₃ VO ₈	O, I	11.981	11.678	16.266	
PbBi ₆ P ₂ O ₁₅	O, P	11.764	10.845	17.015	
PbBi ₆ As ₂ O ₁₅		11.943	11.007	17.267	
PbBi ₆ V ₂ O ₁₅		11.970	11.023	17.324	
Bi ₁₂ P ₂ O ₂₃ *	M, P	12.210	11.440	15.767	92.28
Bi ₁₂ As ₂ O ₂₃		12.208	11.551	16.104	91.45
Bi ₁₂ V ₂ O ₂₃		12.193	11.579	16.163	91.13

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THE RESULTS OF MASS EMPLOYMENT OF THE UNIFICATION SYSTEM FOR THE QUANTITATIVE PHASE ANALYSIS PERFORMANCE BY X-RAY DIFFRACTION (QPAXRD) OF ROCK, ORE AND CONCENTRATES. By A.A. Brovkin, Sci.-Ind. Union "GRANT", Geological Committee, Russia.

In early eighties was developed and widely introduced in the geological service of the FSU unification system (QPAXRD). Later on this system was always improved to fit computing technology progress. QPAXRD system is based on: 1) inner standard method, i.e. α-Al₂O₃ powder certified according to the intensity ratio of diffraction reflections; 2) grinding unification, trial and standard homogenization, texture factor consideration; 3) diffraction spectra survey (DS) in the discrete scanning regime; 4) evaluation of the analysis results by the intermethodical control data and intralaboratory 20% reproducibility control. Within the framework of the unification system there are widely approved in practice Chung calibration coefficients (1974) for over 100 minerals. In one test up to 10-15 minerals content is determined. Peaks superposition factor was taken via the strip-tease method for DS stage removal of DS discrete minerals whose spectra were earlier recorded in DS processing program. Under one-stage samples surveying (2 sec. exposition in the dispersion scanning step (δ) of the analysis results in the content interval of 5-10%, 10-30%, and 30-50% was equal to 12%, 5% and 5%, respectively). At two-stage surveying c^o reduces by 2-3 times. However, a total time of DS surveying increases by 1.5 time.

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THE CRYSTAL STRUCTURE CLASSIFICATION ON CATION TYPE NETS FOR THE FLUORIDES WITH HEAVY METALS.

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Complex fluorides including those of Zr, U, Th La-Lu have been analysed crystallochemically proceeding from the assumption that a main factor determining crystal structures of compounds with heavy atoms is a mutual arrangement of these atoms. The crystal chemistry analysis involved: 1) determination of close packed crystallographic layers (filled with cations); 2) construction of the cation nets in the layers; 3) formulation of the rules