**09.4-02**   THE X-RAY INVESTIGATION OF POTASSIUM XANTHATES. By A.A. Kashaev, N.A. Frolova, S.B. Leonov, M.S. Metsik, O.N. Belkova, Problem Laboratory, Irkutsk Polytechnical Institute, Irkutsk, USSR.

Potassium xanthates are crystallized in the space group $P2_1/c$ and have the following lattice parameters:

		$a(Å)$	$b(Å)$	$c(Å)$	β	Z
I.	$KS_2COC_2H_5$	4.40	16.46	18.89	$92°7'$	8
II.	$KS_2COC_3H_7$	7.23	12.39	21.26	$96°$	8
III.	$KS_2COC_4H_9$	4.37	19.60	9.60	$93°$	4
IV.	$KS_2COC_5H_{11}$	7.88	9.60	24.78	$95°$	8

The structure I has been determined by F. Mazzi, C. Tadini (Zeitschr. f. Krist. 3, 1963). The authors of this work interpreted the structure III and determined the lattice parameters of the structures II and IV. The investigation has been carried out by X-ray photomethod (MoKα). 625 independent reflections were obtained. The structure has been solved by the Patterson method and refined by the least-squares method with R=0.107. In the investigated structure S atoms are located in the corners of the prism in the centre of which potassium is located. Oxygen adjoins a prism face at 1/4 of its height. Therefore the potassium polyhedron has seven corners. Such potassium polyhedra are bonded by triangle bases S_1-S_2-S_1 in the direction of axis a into infinite columns connected to each other by prism edges S_1-S_1 and carbon triangles to an infinite layer in the plane ac. Zigzag chains CH_2-CH_2-CH_2-CH_3 directed along [001] join these layers by means of oxygen atoms. The structure III has a layer character in contrast to the frame structure I.

09.4-03   CRYSTAL STRUCTURES OF A MACROBICYCLIC POLYETHER AND OF ITS COMPLEX WITH RUBIDIUM PICRATE. By I.R.Hanson, J.D.Owen and Mary R.Truter, Molecular Structures Department, Rothamsted Experimental Station, Harpenden, Herts. AL5 2JQ, U.K.

I   II
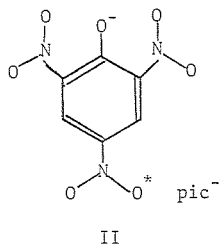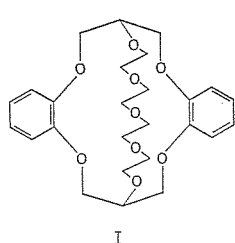
Crystals of the uncomplexed ether, I, are monoclinic with four molecules in a unit cell having a = 8.859(4), b = 11.555(10), c = 25.325(11) Å, and β = 102.49(4)° and space group $P2_1/c$. The structure was solved by direct methods and refined by full matrix least squares to R = 0.068 for 3285 observed reflections.

The molecules take up a folded conformation, with the aliphatic bridge much nearer one benzene ring than the other. There are several unexpected values for the torsion angles.

The rubidium picrate complex is triclinic, a =16.280(4), b = 16.660(5), c = 16.077 Å, α =111.62(2), β =116.29(2), γ = 91.32(2)°. Assuming the space group to be $P\bar{1}$, there are two crystallographically independent rubidium ions, ligand molecules, I, picrate ions and water molecules. Rubidium, Rb(1) is nine-coordinated by the oxygen atoms of I(1) with Rb - O ranging from 2.952(7) to 3.368(11) Å, giving a complex cation $[Rb(1)I(1)]^+$.

The second rubidium Rb(2) is eleven-coordinated by the nine oxygen atoms of I(2) (Rb - O 2.922(11) to 3.277(15) Å) by a nitro oxygen atom, O*, from pic⁻ (2), Rb - O 2.933(16) Å and by a water molecule on a centre of symmetry, Rb - O 3.077(2) Å. This water molecule bridges two complexed ion pairs so that the entity is $\{[Rb(2)I(2)pic^-(2)]_2H_2O\}^0$. There may be hydrogen bonding contacts between the second water molecule, the coordinating pic⁻(2) and the uncoordinated pic⁻(1). Disorder is evident in the aliphatic chains of each ligand and in the pic⁻(1). Least-squares refinement in two blocks of full matrices gave R = 0.105 for 4746 observed reflections.

09.4-04   MOLECULAR AND CRYSTAL STRUCTURES OF URATE SALTS. By E.Dubler, Z.Kopajtic, A.Reller and G.B.Jameson, Institute of Inorganic Chemistry, University of Zürich, Winterthurerstr.190, 8057 Zürich, Switzerland.

Uric acid and urate salts occur as deposits in gouty arthritis as well as in urinary calculi. Since epitaxy is believed to be a growth factor for these deposits (K.Lonsdale, Nature 217,56 (1968)), a detailed knowledge of the structures of the crystals involved is of importance. We have grown single crystals of magnesium bis-(urate) octahydrate, $Mg(C_5H_3N_4O_3)_2 \cdot 8H_2O$, and of monosodium urate monohydrate, $NaC_5H_3N_4O_3 \cdot 1H_2O$, and solved their crystal structures (Mg: R_W=0.053 Na: R_W=0.047). The structure of the magnesium urate contains $Mg(OH_2)_6$-octahedra, two urate anions with deprotonated N(3)-nitrogen atoms (fig. 1) and two non-coordinating water molecules. Medium-strong hydrogen bonding between these units is observed. For the sodium urate we confirmed the general features of the structure determined by N.S. and G.S.Mandel(JACS 98,2319(1976)). In addition we located the hydrogen atoms from fourier maps and refined them successfully.
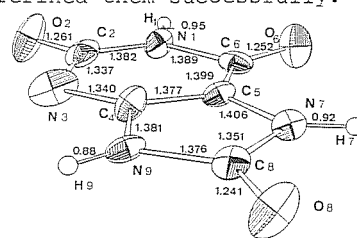

Fig. 1

Geometry of the urate anion in $Mg(C_5H_3N_4O_3)_2 \cdot 8H_2O$