

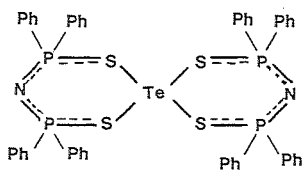
coordination of the Sb atoms is distorted octahedral or  $\psi$ -octahedral.

$2\text{SbCl}_3 \cdot 3(4\text{-phenylpyridine})$ :  $a = 9.622(1)$ ,  $b = 13.363(3)$ ,  $c = 14.445(3)$  Å,  $\alpha = 72.48(2)$ ,  $\beta = 83.28(2)$ ,  $\gamma = 87.70(1)^\circ$ ,  $P\bar{1}$ ,  $Z = 2$ . The most characteristic feature of the structure is the T-shaped geometry of one of the  $\text{SbCl}_3$  molecules, whereas the other is pyramidally shaped as the molecules in  $\text{SbCl}_3$  itself (Lipka, Acta Cryst. (1979) B35, 2020) and in adducts with amines. Another T-shaped  $\text{SbCl}_3$ -fragment has only been found in the complex with 2,2'-bipyridine. Sb-Cl bond lengths vary from 2.36 to 2.74 Å, depending on the strength of bonds in trans position.

$2\text{SbBr}_3 \cdot \text{biphenyl}$ :  $a = 11.301(15)$ ,  $b = 11.638(16)$ ,  $c = 8.194(5)$  Å.  $\beta = 107.54(8)^\circ$ ,  $P2_1/c$ ,  $Z = 2$ .  $\text{SbBr}_3$  molecules and biphenyl molecules form a centrosymmetric complex with coplanar arrangement of the phenyl rings. In agreement with NQR (Grechiskin & Kyuntsel, Opt. Spectry. (1964) 16, 87) and Raman (Perkampus & Schönberger, Chem. Phys. Lett. (1976) 44 (2), 348) results this complex differs significantly from  $2\text{SbCl}_3 \cdot \text{biphenyl}$  (Lipka & Mootz, Z. anorg. allg. Chem. (1978) 440, 217), where the independent  $\text{SbCl}_3$  molecules are differently arranged to the phenyl rings, which are not coplanar but display a dihedral angle of  $40.5^\circ$ .

**09.4-50** THE PREPARATION AND CRYSTAL STRUCTURE OF BIS(LMIDOTETRAPHENYLDITHIODIPHOSPHINO-S,S')-TELLURIUM(II). By S. Bjørnevåg, S. Husebye and K. Maartmann-Moe, Department of Chemistry, University of Bergen, N-5014 Bergen-Univ., Norway.

The title compound,  $[\text{Te}\{\text{N}(\text{Ph}_2\text{PS})_2\}_2]$ , has been prepared from a mixture of  $[\text{Te}(\text{tu})_4]\text{Cl}_2 \cdot \text{H}_2\text{O}$  (tu=thiourea) and  $\text{NH}_4\{\text{N}(\text{Ph}_2\text{PS})_2\}$  dissolved in methanol. Its structure has been determined by means of X-ray crystallographic methods. The yellow crystals are triclinic, space group  $P\bar{1}$ , with  $a = 10.187(1)$  Å,  $b = 12.929(5)$  Å,  $c = 18.282(3)$  Å,  $\alpha = 89.04(2)^\circ$ ,  $\beta = 82.98(1)^\circ$ ,  $\gamma = 77.40(2)^\circ$  and  $Z = 2$ . The crystals are built up of centrosymmetric, square planar  $\text{Te}(\text{II})$  complexes. This is the first such complex where tellurium is bonded to two bidentate ligands. There are two crystallographically independent half molecules in the unit cell with an average Te-S bond length of 2.685(13) Å. Average intraligand S-Te-S angle is  $86.8(1.1)^\circ$ . Ignoring the phenyl groups the complex molecules have an approximate chair configuration.



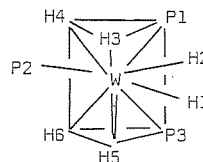
**09.4-51** THE LOW TEMPERATURE NEUTRON DIFFRACTION STUDY OF  $[\text{WH}_6(\text{PPhPr}_2)_3]$ . By David Gregson, Judith A. K. Howard, John L. Spencer, David Turner, Department of Inorganic Chemistry, The University, Bristol BS8 1TS, England, and Sax Mason, Institut Laue Langevin, Grenoble, France.

Hydrogenolysis of hexamethyl tungsten in the presence of  $(\text{PPhPr}_2)_3$  gives the title compound. The structure of these nine co-ordinate species had been a matter of conjecture, until this definitive study was carried out. The molecular symmetry is approximately  $C_{2v}$  rather than  $D_{3h}$ , with two of the phosphine ligands occupying prism vertices rather than the equatorial capping positions of the trigonal prism.

The results of the low temperature neutron experiment are summarised in the Figure (schematic representation).

Full details will be presented, and compared with the low temperature X-ray diffraction study.

Data were collected at the Institut Laue Langevin, Grenoble, the facilities of which are gratefully acknowledged.



W - P(1),P(3)	2.521(2)	P(1) - W - P(3)	107.8(1)°
W - P(2)	2.422(2)	P(2) - W - P(1,3)	126.0(1)
W - H(mean)	1.732(3)	H(1) - W - H(2)	123.8(2)

**09.4-52** THE STRUCTURE OF THE ADDITION COMPOUND OF TRIS-(N,N-DIMETHYLACETAMIDE) HEXAAQUO PRASEODYMIUM TRIFLUOROMETHANE SULPHONATE.

By R.W. Becker, E.E. Castellano, G. Vicentini and T. Tamura, Instituto de Fisica e Quimica de São Carlos, 13560 São Carlos, S.P., Brazil.

The crystal structure of the title compound  $[\text{Pr}(\text{DMA})_3(\text{H}_2\text{O})_6](\text{F}_3\text{CSO}_3)_3$  has been determined by  $^3\text{X}$ -ray diffractometer data and refined anisotropically to an R-factor of 9%. This is part of a series of studies on the coordination of lanthanide adducts and their optical properties (Castellano and Becker, Acta Cryst. (1981), in press). 2299 independent reflections with intensities above background were measured. Space group is  $P2_1$  and unit cell parameters are  $a = 8.63(1)$ ,  $b = 19.89(1)$ ,  $c = 11.44(1)$  Å,  $\beta = 103.6(1)^\circ$ , with  $Z = 2$ . The cation is coordinated to nine oxygen atoms located at the vertices of a tricapped trigonal prism with approximate symmetry  $D_{3h}$ . Six oxygen atoms from equal number of water molecules are located at the vertices of the trigonal prism and three oxygen atoms from DMA groups are at the capping positions. The trifluoromethane sulphonate anions are not coordinated to the cation and are somewhat disordered. The fluorescence spectrum data obtained from the compound doped with  $\text{Eu}^{3+}$ , shows selection rules approximately consistent with the ones expected for point group  $D_{3h}$  on pure symmetry grounds.

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