

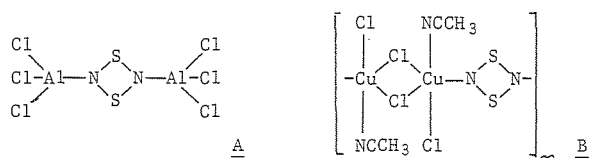
**09.4-08** THE STRUCTURES OF FOUR COMPLEXES OF MERCURY(II)TRIFLUOROACETATE WITH 2,2'-BIPYRIDYL. By Joan Halfpenny, Chemistry Department, The University, Durham, England and R.W.H. Small, Chemistry Department, The University, Lancaster, England.

Four distinct crystalline complexes of mercury(II)trifluoroacetate (A) and 2,2'-bipyridyl (B) have been prepared and their crystal structures determined from Mo K $\alpha$  data using standard heavy atom techniques. They are all triclinic, space group P1, with asymmetric units having the compositions A<sub>1.5</sub>B, A<sub>2</sub>B<sub>2</sub>, A<sub>2</sub>B<sub>3</sub>, AB<sub>2</sub> respectively. Although seven different environments are provided for the mercury atoms within the series, there are some common features between these structural units. There are some quite unusual features arising from the disposition of the trifluoroacetate groups, leading in some cases to an apparent disproportionation of charge between adjacent non equivalent mercury atoms. The wide range of Hg-O contact distances (2.1-3.0Å) has been interpreted in terms of the variable role of the trifluoroacetate groups. The extent to which any of these compounds can be described as ionic will be discussed.

**09.4-09** STRUCTURAL CHEMISTRY OF SOME COORDINATION COMPOUNDS WITH SULFUR-NITROGEN LIGANDS. By U. Thewalt and M. Burger, Sektion für Röntgen- und Elektronenbeugung, Universität Ulm, Oberer Eselsberg, D-7900 Ulm, F.R.G.

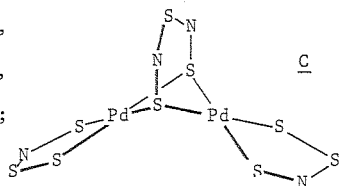
The reactions of S<sub>4</sub>N<sub>4</sub> with metal halogen compounds often are unpredictable and unexpected reaction products can be obtained. Besides ionic compounds we have isolated and characterized by X-ray crystallography some non-ionic complexes with S<sub>n</sub>N<sub>m</sub> rings or fragments covalently bonded to the metal atom(s).

In the formation of (AlCl<sub>3</sub>)<sub>2</sub>S<sub>2</sub>N<sub>2</sub> (A) and [CuCl<sub>2</sub>(CH<sub>3</sub>CN)]<sub>2</sub>-S<sub>2</sub>N<sub>2</sub> (B), S<sub>4</sub>N<sub>4</sub> undergoes a symmetrical scission yielding neutral S<sub>2</sub>N<sub>2</sub> rings.



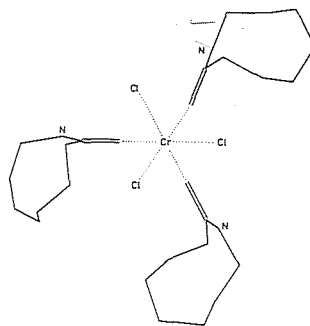
Pd<sub>2</sub>(S<sub>3</sub>N)<sub>2</sub>(S<sub>3</sub>N<sub>2</sub>) (C), obtained from PdCl<sub>2</sub> and S<sub>4</sub>N<sub>4</sub>, contains the new ligand S<sub>3</sub>N<sub>2</sub>. In  $\beta$ -FeCl<sub>3</sub>S<sub>4</sub>N<sub>4</sub> (D) S<sub>4</sub>N<sub>4</sub> acts as a neutral N-bonded ligand, which completes the coordination tetrahedron around the Fe center.

A: P2<sub>1</sub>/n, a 8.96, b 9.64, c 7.31 Å,  $\beta$  110.4°;  
B: P2<sub>1</sub>/c, a 9.63, b 7.27, c 10.01 Å,  $\beta$  93.2°;  
C: R3, a 32.76, c 6.52 Å;  
D: P2<sub>1</sub>/c, a 6.80, b 11.31, c 13.78 Å,  $\beta$  95.0°.



**09.4-10** STRUCTURE OF A TRIS LACTAM CHROMIUM(III) TRICHLORIDE. By J. Hašek, J. Ječný and K. Huml, Institute of Macromolecular Chemistry, Czechoslovak Academy of Sciences, 162 06 Praha 6, Czechoslovakia.

CrCl<sub>3</sub>·3(C<sub>8</sub>H<sub>15</sub>ON)<sub>3</sub>·(C<sub>7</sub>H<sub>8</sub>)<sub>0.8</sub>·(C<sub>6</sub>H<sub>6</sub>)<sub>0.7</sub> ranks among the most efficient initiators of lactam polymerization leading to linear polyamides (Puffr R., US.Pat. 4,111869). Mass spectroscopy showed that the crystalline complex is solvated with benzene and toluene molecules. The crystals deteriorate in air due to hydrolysis. Solvating molecules giving high "temperature factors" and fractional occupancy factors are supposed to be disordered. Also high "temperature factors" and difficulties in the localization of three C atoms in one of the ligands can be explained by the presence of different conformations allowed for the octanelactam ring.



Monoclinic, P2<sub>1</sub>/c

a = 18.536 (8) Å  
b = 15.228 (8)  
c = 14.849 (8)  
 $\beta$  = 96.56 (4)°

Z = 4  
R = 0.092

Cr-Cl: 2.32, 2.33, 2.44 Å

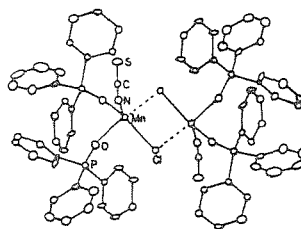
Cr-O: 2.00, 2.00, 2.08

O...O: 2.64, 2.75, 2.75

Plane Cl...Cr 1.30

**09.4-11** THE CRYSTAL STRUCTURE OF BIS(TRIPHENYLPHOSPHINE OXIDE) MANGANESE(II) CHLORIDE ISOTHIOCYANATE. By K. Tomita, Instituto de Quimica de Araraquara, 14800 - Araraquara, Sao Paulo, Brasil.

This complex, synthesized formerly as bis(isothiocyanate)bis(triphenylphosphine oxide) manganese(II) by M.R. Davolos (Private communication, Instituto de Quimica da Universidade de Sao Paulo, Brasil, 1978) shows fluorescence at 640 nm which suggests a hexacoordinated complex (Orgel, J. Chem. Phys. (1955) 23, 1958). The crystals are orthorhombic, Pbca, a=10.992 (3), b=22.331(3), c=27.722(4) Å, V=6805 Å<sup>3</sup>, Z=8, d<sub>o</sub>=1.41, d<sub>c</sub>=1.420g/cm<sup>3</sup>. Of the 5333 independent reflections measured on a CAD-4 diffractometer using MoK $\alpha$  radiation, 2486 had intensities greater than  $\alpha$ <sub>3</sub>(I). The structure was solved by direct methods and refined by least-squares techniques to an R value of 0.092.



for computing facilities.

The title compound adopts a dimeric Cl-bridged structure in which Mn is penta-coordinated. This work was supported by the Conselho Nacional de Desenvolvimento Cientifico e Tecnológico. We thank the Instituto de Fisica e Quimica de Sao Carlos, Brasil