

## 09. STRUCTURES OF ORGANIC AND COORDINATION COMPOUNDS C-177

### 09.3-7 STRUCTURES OF THREE POLYMORPHS OF trans-[Pt(C<sub>6</sub>H<sub>9</sub>)H{P(C<sub>6</sub>H<sub>5</sub>)<sub>3</sub>}<sub>2</sub>]

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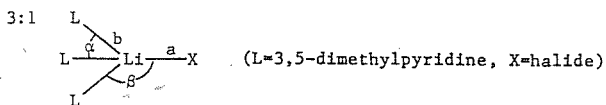
Bulk samples of trans-[Pt(C<sub>6</sub>H<sub>9</sub>)H{P(C<sub>6</sub>H<sub>5</sub>)<sub>3</sub>}<sub>2</sub>], recrystallised from methanol/benzene solution, exhibit two Pt-H stretching frequencies [1855 and 1940 cm<sup>-1</sup>] in their solid state infra red spectra and contain two morphologically distinct crystalline species. Recrystallisation from each of the solvent systems methanol, acetone and acetone/dichloromethane (trace) yields separate polymorphs. Crystals from methanol, 1, are triclinic, P $\bar{1}$ , with Z=2. Crystals from acetone, 2, and acetone/dichloromethane, 3, are each monoclinic, P2<sub>1</sub>/c, with Z=4. 1 and 3 exhibit similarly disordered cyclohexenyl ligands but eclipsed and staggered conformations, respectively, of the phosphine ligands about the P-P vectors. The cyclohexenyl disorder has been modelled successfully with RAELS [A.D. Rae. RAELS. A comprehensive constrained least squares refinement program. 1976. University of New South Wales, Kensington, New South Wales, 2033, Australia] as the sum of two 180° rotated orientations (about the Pt-C bond) of each of the two ring enantiomers. 2 has a similar phosphine ligand conformation to 1 (eclipsed) but, in contrast to 1, contains ordered cyclohexenyl ligands.

### 09.4-2 CRYSTALLOGRAPHIC STUDIES OF LITHIUM HALIDE COMPLEXES WITH AROMATIC AMINES.

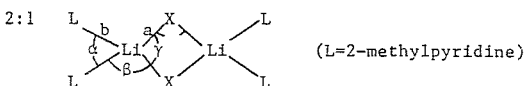
C.L. Raston, C.R. Whitaker, A.H. White

A variety of structural types has been found in lithium halide complexes with substituted pyridines, closely related to the analogous copper series<sup>1-4</sup>, with amine-to-lithium halide ratios of 3:1 (monomers), 2:1 (dimers), and 3:2 (a tetramer).

Examples of these types (bond lengths, angles degrees):



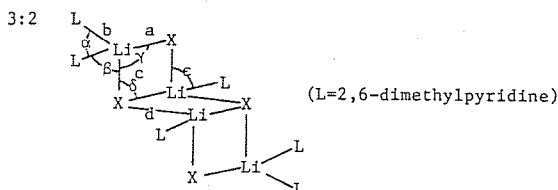
	Cl	Br	I
a	2.320(9)	2.51(2)	2.80(1)
b	2.05(1)	2.07(2)	2.02(2)-2.07(2)
α	106.4(5)	108.8(9)	107.4(8)-113.1(9)
β	112.4(4)	110.1(8)	107.6(7)-110.2(7)



	Cl	Br	I
average a	2.381(9)	2.564(7)	2.794(10)
average b	2.09(1)	2.09(1)	2.08(1)
α	112.2(3)	115.0(3)	109.2(4)
average β	111.1(5)	110.1(4)	112.7(5)
γ	101.7(2)	101.5(2)	100.1(3)
δ	78.2(3)	78.4(2)	79.9(3)

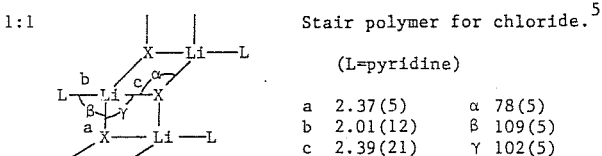
### 09.4-1 THE CRYSTAL AND MOLECULAR STRUCTURE OF CALCIUM BIPYRIDYL PICRATE. By V.S.Yadava and V.M. Padmanabhan, Neutron Physics Division, Bhabha Atomic Research Centre, Trombay, Bombay 400 085, India and N.S.Poonia, Chemistry Department, Devi Ahilya Vishwavidyalaya, Indore, India.

The title compound CaC<sub>22</sub>N<sub>10</sub>O<sub>14</sub>H<sub>20</sub> crystallises in the orthorhombic space group P2<sub>1</sub>2<sub>1</sub>2<sub>1</sub> with a = 24.154, b = 16.128, c = 18.068 Å and Z = 4. The structure is based on 3904 reflections collected with Trombay Computer-controlled four-circle diffractometer. The structure was solved by Patterson and Fourier methods and refined by least squares to an R-index of 0.068. The calcium is eight coordinated to four nitrogens of bipyridyl rings and four oxygens of picrate groups forming the corners of a distorted cube. The ortho nitro groups of picrate are twisted out of the plane of the benzene ring while the para nitro group is nearly planar. The two pyridine rings of bipyridyl are slightly twisted.



Average distances and selected angles for bromide:

a	2.54(2)	α	109.6(7)
b	2.16(1)	β	127.6(7)
c	2.61(1)	γ	97.5(5)
d	2.65(2)	δ	79.4(5)
		ε	99.8(5)



#### References:

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3. P.C. Healy et al, J. Chem. Soc.Dalton Trans(1983),1905
4. J.A. Campbell et al, Aust. J. Chem(1977),30,1937
5. F. Durant et al, Bull. Soc. Chim. Belges(1966),75,806