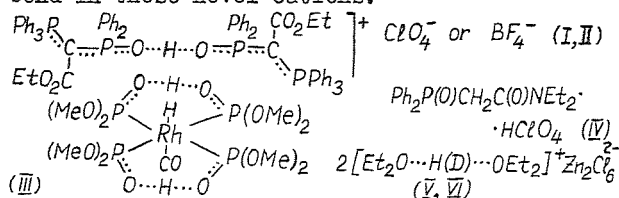


04.6-4 X-RAY INVESTIGATION OF CRYSTALS WITH STRONG HYDROGEN BONDS. By M.Yu. Antipin and Yu.T. Struchkov, Nesmeyanov Institute of Organoelement Compounds of the USSR Academy of Sciences Moscow USSR

Data of low temperature (-120°C) X-ray studies have been used to analyze the specific features of geometry and thermal vibrations in a large series of crystal structures with very strong and probably symmetrical H-bonds of the O...H...O type (O...O 2.4-2.5 Å). This series includes complexes of phosphoryl compounds with acids, phosphite complexes, tautomeric organophosphorus compounds, etherates etc., whose typical examples are represented by compounds I-VI. Thermal ellipsoids of the central H-atom in the hydrogen bonds of these structures correspond to intense vibrations of this atom preferably along the O...O direction. Precise X-ray data can give a qualitative description of such movement. On the contrary, in the case of weak or medium strength H-bonds thermal vibration of H-atom occur preferably in the direction normal to the O...O line. Isotopic substitution in V and VI (exchange of H for D) does not cause an increase of the O...O distance which is in both cases equal to 2.396 Å and this result is a good evidence for a single-minimum potential of the H-bond in these novel cations.



The molecule possesses a symmetry center. The palladium atom, occupying the center of symmetry, is coordinated in a square-planar fashion to the Cl atoms and the N atom of pyridine ring.

There is an intramolecular hydrogen bond $\text{N}_2\text{-H}_{21}\dots\text{Cl}_1 = 3.448(6)\text{Å}$ and on the other hand the molecules are packed in the crystal in a network of hydrogen bonds, there being between the latter bifurcated ones as indicated in the following table:

DONOR-H		DONOR...ACCEPTOR	
N2	-H21	N2Cl1 (0)
0.757(.085)		3.448(.006)	
N2	-H21	N2Cl1 (1)
0.757(.085)		3.378(.006)	
N2	-H22	N2Cl1 (2)
0.910(.098)		3.429(.006)	
H...ACCEPTOR		DONOR-H.....ACCEPTOR	
H21	...Cl1 (0)	N2	-H21 ...Cl1 (0)
2.998(.094)		120.82(7.70)	
H21	...Cl1 (1)	N2	-H21 ...Cl1 (1)
2.776(.088)		138.20(8.43)	
H22	...Cl1 (2)	N2	-H22 ...Cl1 (2)
2.550(.094)		162.44(7.58)	

EQUIVALENT POSITIONS:

- (0) X,Y,Z,
 (1) -X,-1, -Y,-Z
 (2) +X,+Y,+Z+1

04.6-5 X-RAY CRYSTAL AND MOLECULAR STRUCTURE OF

TRANS-BIS(2-AMINOPYRIDINE) DICHLOROPALLADIUM(II)

M.C. Navarro-Ranninger*, S. Martínez-Carrera and S. García-Blanco.

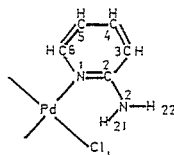
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The title compound have been prepared according to (1) Suitable crystals were obtained by recrystallization from dimethylformamide.

Crystal data: $\text{Pd}(\text{C}_5\text{N}_2\text{H}_6)_2\text{Cl}_2$, $M = 235.97$, $a = 5.922(2)$, $b = 16.589(12)$, $c = 6.722(3)\text{Å}$. $\beta = 104.52(3)^\circ$. $V = 639.3(6)\text{Å}^3$, $D_x = 1.226\text{ Mg m}^{-3}$, F_2/n , $Z = 2$.

Intensities were measured on a CAD-4 diffractometer with $\text{MoK}\alpha$ radiation. The structure was solved by Patterson and Fourier methods and refined anisotropically by full matrix least-squares technique to $R = 0.050$, $R_w = 0.060$ for 1737 reflections ($I \geq 2\sigma(I_0)$). The hydrogen atoms were found from the difference Fourier syntheses. The final $R = 0.045$, $R_w = 0.054$, ($w = 1.0$).



(1) C. Gómez, A. Alvarez, C. Navarro-Ranninger, J.R. Masaguer, Trans. Met. Chem. (in press).

04.6-6 STRUCTURE AND HYDROGEN ORDERING IN ICES VI,

VII, AND VIII BY NEUTRON POWDER DIFFRACTION. By W.F. Kuhs*, J.L. Finney† C. Vettier* and D.V. Bliss* Institut Laue-Langevin, 156X, 38042 Grenoble, France †Department of Crystallography, Birbeck College, University of London, Malet Street, London WC1E7HX, England.

The ice phase diagram is extremely rich, due partly to the structural versatility inherent in the tetrahedral hydrogen-bonding possibilities of the water molecule and partly to the possibilities of proton ordering. Especially the high pressure phases have been used to explore details of the water-water interaction. Recently, the cell volumes, c/a ratios, and proton orderings have been used to test model water-water potential functions that are extensively used in computer simulations of the pure liquid and more complex aqueous solutions. Many uncertainties remain, however, concerning the details of the structures of several of these phases. This is especially so for the highest pressure phases, where implications concerning proton ordering from dielectric and thermodynamic measurements are sometimes in conflict with the limited crystallographic data available. Moreover, much of this crystallographic data was collected on samples quenched under pressure to liquid nitrogen temperatures, and then recovered to ambient pressure, rather than on samples within the stability ranges of the respective phases. Although it appears to be generally assumed that such recovered samples are structurally similar to the original stable phases, the possibility remains that structural changes (especially of the hydrogen ordering) may occur on recovery, and these might be responsible for some of the apparent discrepancies between crystallographic and other results. These uncertainties can in principle be resolved by neutron crystallography. The recent development of high pressure cells for neutron work allows powder measurements to be taken throughout the whole of the ice phase diagram up