Crystal and Molecular Structure of Iodo[1,1,1-tris(diphenylphosphinomethyl)-ethane]nickel(I)

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Summary The co-ordination polyhedron in the Ni[MeC-$(CH_2PPh_2)_3$]I complex is a distorted tetrahedron with a P_3I donor set; this is the first recorded *X*-ray structure of a complex of nickel in an unambiguously monovalent state.

THE tripod ligand, 1,1,1-tris(diphenylphosphinomethyl)-ethane, tpe, reduces Ni^{II} and Co^{II} iodides spontaneously to give complexes of Ni^{I} and Co^{I} with the formula M (tpe)I.[1]

An *X*-ray analysis of the compound Ni(tpe)I was undertaken in order to determine the geometry of the complexes.

Crystal data: space group $Pn2_1a$, $a = 20{\cdot}439(6)$, $b = 17{\cdot}143(7)$, $c = 10{\cdot}359(3)$ Å, $Z = 4$, $D_c = 1{\cdot}491$, $D_m = 1{\cdot}49$.

The intensity data were collected on a four-cycles Hilger automatic diffractometer, with Mo-K_α radiation, using the θ-ω scan technique. These data (2357 observed reflections) were corrected for Lorentz-polarization and for absorption effects. The structure was solved by the heavy-atom technique and subsequent Fourier syntheses which showed all non-hydrogen atoms. The structure was then refined by full-matrix least-squares to an *R* factor of 0·054. The refinement is still in progress.

The structure consists of discrete Ni(tpe)I molecules. The nickel atoms are four-co-ordinate, linked to the three phosphorus atoms of the tpe ligand and to the iodine atom. The co-ordination polyhedron can be described as a distorted tetrahedron. Bond lengths and angles about the nickel atom are: Ni–I 2·546(2), Ni–P(1) 2·220(4), Ni–P(2) 2·211(4), Ni–P(3) 2·210(4) Å, I–Ni–P(1) 124·5(1), I–Ni–P(2) 125·0(1), I–Ni–P(3) 115·9(1), P(1)–Ni–P(2) 92·2(1), P(1)–Ni–P(3) 94·4(1), P(2)–Ni–P(3) 97·7(1)°.

The three P–Ni–P angles have values much lower than the regular tetrahedral angle (109°28′); this distortion seems to be imposed by the steric requirements of the tpe tripod ligand which prevent the three phosphorus atoms moving apart.

(*Received, 24th July* 1972; *Com.* 1278.)

[1] L. Sacconi and S. Midollini, *J.C.S. Dalton*, 1972, 1213.