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X-ray Structure of *cis*-(bis-(*o*-Amidophenyl)diphenylphosphine) Nickel(II) Acetone Solvate

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In these laboratories we have been investigating some aspects of the synthesis and chelation of bidentate *o*-aminophenyldiphenylphosphines, particularly with regard to factors affecting geometrical isomerism in the square planar complexes of such ligands [1]. In this preliminary paper we report the X-ray structure of the neutral complex *cis*-(bis-(*o*-amidophenyl)diphenylphosphine)nickel(II) acetone solvate. The synthesis and properties of this species are to be described elsewhere [2].

Crystals of the title complex suitable for diffraction studies were grown from acetone. The complex crystallises in monoclinic space group *I2* with a = 14.645(4), b = 9.264(4), c = 12.996(4) Å, β = 110.132(4), V = 1660 Å^3 and Z = 2. The X-ray data were collected as previously described [3], using graphite-monochromated Mo-K$_\alpha$ radiation (λ = 0.71069 Å). The structure was solved by a combination of Patterson and Fourier difference techniques and refined to R = 0.07 (Rw = 0.07) for 1388 reflections, $F > 6\sigma(F)$.

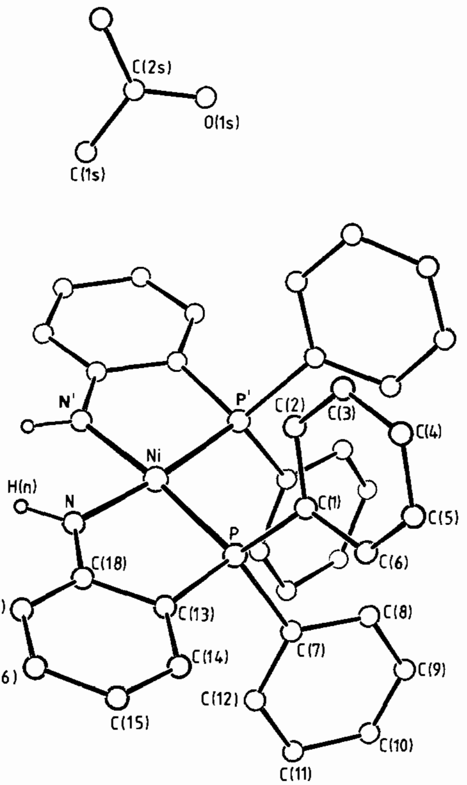

Fig. 1. The molecular structure of the title complex. Principal bond lengths and angle are: Ni–N 1.862(9), Ni–P 2.190(3) Å. N–Ni–P 84.9(3), P′–Ni–P 101.5(2), N′–Ni–N 88.7(6)°.

Figure 1 is an ORTEP drawing of the complex, including selected bond lengths and angles. The *cis*-square planar geometry of the donors around the metal ion is immediately apparent, with the nickel bound to two singly deprotonated primary amine nitrogens and both phosphorus atoms. A crystallographic two-fold axis passes through the nickel atom and bisects the N′–Ni–N and P′–Ni–P angles. The acetone solvate molecule also lies on a two-fold axis, and does not interact with the metal. The bond lengths to the metal ion are quoted in the caption to the Fig. 1 and fall close to the expected [4] ranges for Ni–L bonds of these particular types. The atoms of the donor set are rigorously coplanar with the metal ion (maximum devaition < 0.003 Å) and these five atoms themselves deviate from coplanarity with the phenyl rings to which the donors are attached by less than 0.09 Å. Interaction of the bulky phenyl groups does not seem to occur and intramolecular contact distances all approximate to the sum of the van der Waals radii for the relevant atoms.

^{31}P NMR evidence indicates that the *cis* species remains the preferred form in solution, with the *trans* isomer present in only relatively slight abundance. The possible role of the *trans*-effect in this system, leading to the enhanced stability of the *cis*-isomer over the *trans*-, has yet to be unequivocally established.

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