

## X-Ray Studies of Aminophosphine Complexes of Molybdenum and Palladium, and of an Aminophosphonium Iodide

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THE following derivatives of bis(diphenylphosphino)ethylamine,  $[(C_6H_5)_2P \cdot N(C_2H_5) \cdot P(C_6H_5)_2] = L$ , have been studied by X-ray methods using Cu- $K_\alpha$  radiation and visual estimation of intensities. Refinement was by three-dimensional least-squares analysis.

*A:* Bis(diphenylphosphino)ethylamine-molybdenum tetracarbonyl,  $Mo(CO)_4L$ :  $C_{30}H_{25}NO_4P_2Mo$ ,  $M = 621.4$ .

Orthorhombic,  $a = 16.69$ ,  $b = 17.21$ ,  $c = 20.16$  Å,  $Z = 8$ ; space group,  $Pbcn$  (No. 60).

The final  $R$ -value is 9.46% for 1700 non-zero observed reflections. The molybdenum atom is octahedrally co-ordinated, with  $L$  occupying two neighbouring sites.  $Mo-C = 1.990 \pm 0.011$ ,  $C-O = 1.15 \pm 0.015$ ,  $Mo-P = 2.505 \pm 0.005$  Å;  $P-N-P = 103.8^\circ \pm 1.0$ ,  $P-Mo-P = 64.8^\circ \pm 0.2$ .

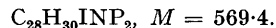
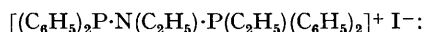
(Here—and below—where there are several chemically equivalent dimensions, a mean value is given.)

*B:* Dichloro-bis(diphenylphosphino)ethylamine-palladium(II),  $PdLCl_2$ :  $C_{28}H_{25}Cl_2NP_2Pd$ ,  $M = 590.8$ .

Orthorhombic,  $a = 20.90$ ,  $b = 17.60$ ,  $c = 13.87$  Å,  $Z = 8$ ; space group,  $Pbca$  (No. 61).

The final  $R$ -value is 10.9% for 2735 terms. The palladium atom is approximately square co-ordinated, with chlorine (or phosphorus) atoms in a *cis*-relationship.  $Pd-Cl = 2.367 \pm 0.003$ ,  $Pd-P = 2.224 \pm 0.003$ ,  $P-N = 1.72 \pm 0.01$  Å;  $P-N-P = 97.7^\circ \pm 0.7$ ,  $P-Pd-P = 71.43^\circ \pm 0.15$ ,  $Cl-Pd-Cl = 94.77^\circ \pm 0.17$ ,  $P-Pd-Cl = 96.2^\circ \pm 0.5$ .

*C:* Ethyl iodide adduct of  $L$  which proves to have the structure



Orthorhombic,  $a = 13.90$ ,  $b = 21.22$ ,  $c = 10.04$  Å,  $Z = 4$ ; space group,  $P2_12_12_1$  (No. 19).

The final  $R$ -value is 13.0% for 1221 terms.  $P-N$  (4-co-ordinated P) =  $1.75 \pm 0.03$ ,  $P-N$  (3-co-ordinated P) =  $1.88 \pm 0.03$ ,  $P-C$  (alkyl) =  $1.81 \pm 0.05$ ,  $P-C$  (aryl) =  $1.81 \pm 0.02$  Å;  $P-N-P = 111.1^\circ \pm 1.6$ , mean angle at 4-co-ordinated P =  $109.5^\circ \pm 0.7$ , at 3-co-ordinated P =  $102.0^\circ \pm 0.7$ .

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