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_α-radiation and visual estimation of intensities. Refinement was by three-dimensional least-squares analysis.

A: Bis(diphenylphosphino)ethylamine-molybdenum tetracarbonyl, $Mo(CO)_4L$: $C_{30}H_{25}NO_4P_2Mo$, $M = 621{\cdot}4$.

Orthorhombic, $a = 16{\cdot}69$, $b = 17{\cdot}21$, $c = 20{\cdot}16$ 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 ± 0·011, C–O = 1·15 ± 0·015, Mo–P = 2·505 ± 0·005 Å; P–N–P = 103·8° ± 1·0, P–Mo–P = 64·8° ± 0·2.

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

B: Dichloro-bis(diphenylphosphino)ethylamine-palladium(II), $PdLCl_2$: $C_{26}H_{25}Cl_2NP_2Pd$, $M = 590{\cdot}8$.

Orthorhombic, $a = 20{\cdot}90$, $b = 17{\cdot}60$, $c = 13{\cdot}87$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 ± 0·003, Pd–P = 2·224 ± 0·003, P–N = 1·72 ± 0·01Å; P–N–P = 97·7° ± 0·7, P–Pd–P = 71·43° ± 0·15, Cl–Pd–Cl = 94·77° ± 0·17, P–Pd–Cl = 96·2° ± 0·5.

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

$$[(C_6H_5)_2P{\cdot}N(C_2H_5){\cdot}P(C_2H_5)(C_6H_5)_2]^+ I^-:$$

$$C_{28}H_{30}INP_2,\ M = 569{\cdot}4.$$

Orthorhombic, $a = 13{\cdot}90$, $b = 21{\cdot}22$, $c = 10{\cdot}04$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 ± 0·03, P–N (3-co-ordinated P) = 1·88 ± 0·03, P–C (alkyl) = 1·81 ± 0·05, P–C (aryl) = 1·81 ± 0·02Å; P–N–P = 111·1° ± 1·6, mean angle at 4-co-ordinated P = 109·5° ± 0·7, at 3-co-ordinated P = 102·0° ± 0·7.

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