

Tableau 4. *Les liaisons hydrogène*

Octaèdre central	Liaison hydrogène	Distance	Octaèdre périphérique (coordonnées de l'atome de Fe)
H(1.1)	O(1)-H(1.1)···Cl	3,219 Å	$0, \frac{1}{2}, \frac{1}{2}$
H(1.2)	O(1)-H(1.2)···Cl	3,230	$0, -\frac{1}{2}, \frac{1}{2}$
H(2.1)	O(2)-H(2.1)···Cl	3,149	$1, 0, 0$
H(2.2)	O(2)-H(2.2)···Cl	3,152	$1, \frac{1}{2}, \frac{1}{2}$
Cl	Cl···(H1.1)-O(1)	3,219	$0, -\frac{1}{2}, \frac{1}{2}$
Cl	Cl···(H1.2)-O(1)	3,230	$0, \frac{1}{2}, \frac{1}{2}$
Cl	Cl···(H2.1)-O(2)	3,149	$1, 0, 0$
Cl	Cl···(H2.2)-O(2)	3,152	$1, \frac{1}{2}, \frac{1}{2}$

distances Cl-O et O-O entre atomes d'un même octaèdre $\text{Fe}(\text{H}_2\text{O})_4\text{Cl}_2$ sont supérieures aux contacts de van der Waals: $1,80 + 1,40 = 3,20$ Å pour Cl-O et 2,80 Å pour O-O.

Environnement des molécules d'eau et des atomes de chlore

La Fig. 1 montre que, entre un édifice octaédrique central et ses 10 plus proches voisins, s'établissent 16 ponts hydrogène O-H···Cl. Il y a, d'une part, des interactions entre les 8 protons centraux et 8 atomes de chlore appartenant à des octaèdres périphériques, d'autre part, chaque atome de chlore central établit 4 ponts et avec des molécules d'eau extérieures. Le Tableau 4 indique au moyen des coordonnées de

l'atome métallique quel octaèdre périphérique est concerné dans chacune de ces liaisons.

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Crystal and Molecular Structure of Ethylene-1,1-bis(triphenylphosphonium)-2,2-bis(phenylamide): $[(\text{C}_6\text{H}_5)_3\text{P}]_2\text{C} \cdot \text{C}(\text{N} \cdot \text{C}_6\text{H}_5)_2^*$

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The structure has been determined by single-crystal X-ray diffraction. At 23°C, $a = 11.863$ (10), $b = 17.670$ (7), $c = 19.330$ (17) Å, $\beta = 95.9$ (2)°, $V = 4030.5$ Å³, $M.W.(\text{calc}) = 730.84$, $Z = 4$, $D_m = 1.22$ (2), $D_x = 1.204$ g.cm⁻³, space group $C2/c$. Full-matrix least-squares refinement of 1335 observed three-dimensional diffractometer data ($\text{Mo } K\alpha$) led to a final weighted residual of 0.045 on F . The molecule has a twofold rotation axis coincident with the central C-C bond of length 1.477 (6) Å, about which there is a twist of 9.7 (2)°, so that the six central atoms are not quite coplanar. Bonding about the central carbon atoms is trigonal planar with P-C-P, N-C-N, C-C-P and C-C-N angles of 136.2 (3), 137.0 (5), 111.9 (2) and 111.5 (2)°.

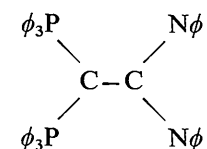
The preparation of hexaphenylcarbodiphosphorane

$\phi_3\text{P}=\text{C}=\text{P}\phi_3$ (ϕ = phenyl) has been described by Ramirez, Pilot, Smith, Hansen & McKelvie (1967), who characterized the products of numerous reactions involving this compound. One such product was the 1:1 adduct formed with diphenylcarbodiimide $\phi\text{N}=\text{C}=\text{N}\phi$; we report here the structure of this adduct, ethylene-

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1,1-bis(triphenylphosphonium)-2,2-bis(phenyl-amide):



The chemistry of these compounds and the structure of a related compound will be reported elsewhere (Ross, Manojlovic-Muir, Hamilton, Ramirez & Pilot, 1971).

Data collection, structure determination and refinement procedures are detailed below and the most significant structural results are presented in the tables and the abstract.

Crystal data

Light yellow monoclinic prisms, a = 11.863 (10), b = 17.670 (7), c = 19.330 (17) Å, β = 95.9 (2)°.

T = 23 (2)°C, λ(Mo Kα) = 0.71069 Å. Z = 4, D_x = 1.204, D_{meas} = 1.22 (2) g.cm⁻³ by flotation.

Systematic absences: hkl for h+k ≠ 2n; h0l for l ≠ 2n. Possible space groups Cc and C2/c. Probable space group C2/c because of successful refinement, intensity statistics, and absence of piezoelectric effect. Linear absorption coefficient μ = 1.48 cm⁻¹ (Mo Kα).

* Numbers in parentheses here and elsewhere are estimated standard errors in the last significant figure.

Table 1. Observed and calculated structure amplitudes and estimated standard deviations of the observed values used in the least-squares refinement

Table with multiple columns (h, k, l, F, Fc, etc.) and rows of numerical data representing observed and calculated structure amplitudes. Includes a note: 'All values in (electrons/cell) × 10 (10 · F(000) = 15360)'.

Table 2. Fractional coordinates and thermal motion parameters for non-hydrogen atoms derived from the least-squares refinement

Estimated standard deviations are in parentheses. The Debye-Waller factor is defined as

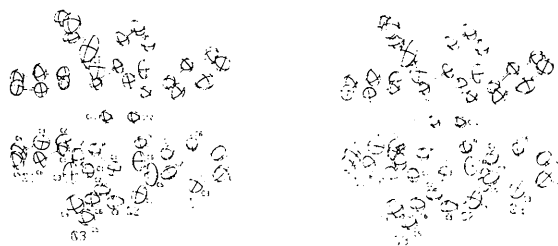
$$T = \exp \left[-2\pi^2 \sum_i \sum_j a_i^* a_j^* h_i h_j U^{ij} \right]$$

The values for U are in \AA^2 and have been multiplied by 10^4 . The fractional coordinates have been multiplied by 10^5 . $\phi C(2)$ denotes carbon atom 2 on phenyl group 1, etc.

	x	y	z	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P	-302 (8)	9523 (5)	16693 (5)	180 (3)	159 (2)	170 (3)	11 (2)	35 (2)	0 (2)
N	1538 (23)	24304 (16)	18789 (15)	246 (10)	124 (7)	182 (9)	9 (6)	44 (7)	20 (7)
C(1)	0	13171 (26)	25000	220 (16)	110 (12)	166 (15)	0	14 (12)	0
C(2)	0	21548 (27)	25000	147 (15)	139 (13)	187 (18)	0	-2 (12)	0
$\phi 1C(1)$	-392 (36)	-705 (20)	16477 (19)	190 (12)	176 (9)	151 (11)	-22 (10)	27 (9)	-36 (8)
$\phi 1C(2)$	-9936 (40)	-4754 (29)	17989 (20)	196 (16)	237 (14)	213 (13)	2 (12)	11 (10)	-26 (9)
$\phi 1C(3)$	-10432 (49)	-12528 (31)	17477 (23)	304 (20)	215 (14)	290 (15)	-90 (14)	-2 (13)	-14 (10)
$\phi 1C(4)$	-1357 (55)	-16397 (29)	15368 (26)	434 (22)	158 (12)	403 (19)	-19 (15)	71 (15)	-53 (11)
$\phi 1C(5)$	8126 (50)	-2641 (27)	13766 (24)	380 (20)	155 (13)	423 (18)	-1 (12)	152 (14)	-52 (10)
$\phi 1C(6)$	8594 (38)	-4845 (25)	14329 (21)	232 (15)	168 (12)	298 (14)	-13 (11)	84 (11)	-35 (9)
$\phi 2C(1)$	13040 (30)	11575 (21)	39115 (20)	198 (12)	169 (10)	219 (13)	-4 (8)	50 (9)	-9 (9)
$\phi 2C(2)$	15611 (38)	7123 (27)	44963 (22)	235 (15)	273 (13)	187 (14)	-38 (10)	-6 (11)	29 (10)
$\phi 2C(3)$	25390 (43)	8145 (33)	49328 (26)	273 (16)	324 (15)	245 (15)	9 (12)	-4 (13)	22 (12)
$\phi 2C(4)$	32775 (48)	13684 (32)	47814 (31)	278 (19)	297 (15)	435 (22)	-12 (13)	-128 (16)	-35 (14)
$\phi 2C(5)$	30462 (44)	18083 (30)	41958 (33)	257 (18)	266 (14)	634 (27)	-100 (12)	-121 (17)	113 (15)
$\phi 2C(6)$	20655 (39)	17039 (26)	37588 (29)	224 (16)	220 (12)	420 (19)	-37 (10)	-32 (14)	81 (12)
$\phi 3C(1)$	-12054 (34)	12121 (19)	37580 (21)	229 (14)	132 (9)	173 (13)	-24 (7)	54 (10)	-25 (8)
$\phi 3C(2)$	-22323 (41)	12595 (22)	33534 (27)	256 (17)	184 (10)	233 (16)	-8 (8)	67 (14)	-15 (9)
$\phi 3C(3)$	-32261 (43)	14324 (22)	36495 (30)	217 (17)	186 (10)	414 (21)	15 (9)	90 (17)	0 (11)
$\phi 3C(4)$	-31646 (53)	15630 (25)	43529 (29)	297 (20)	238 (12)	368 (22)	53 (11)	182 (19)	-25 (11)
$\phi 3C(5)$	-21422 (52)	15194 (26)	47569 (28)	335 (20)	292 (13)	265 (18)	33 (11)	109 (17)	-14 (11)
$\phi 3C(6)$	-11723 (46)	13403 (23)	44592 (24)	229 (18)	225 (11)	272 (17)	20 (9)	116 (14)	-15 (9)
$\phi 4C(1)$	262 (32)	31771 (20)	16734 (19)	184 (12)	150 (9)	175 (11)	9 (9)	1 (9)	5 (8)
$\phi 4C(2)$	8099 (36)	34721 (25)	12486 (21)	235 (14)	181 (12)	214 (14)	8 (10)	36 (11)	25 (9)
$\phi 4C(3)$	7763 (47)	42129 (28)	10393 (26)	316 (17)	211 (14)	303 (15)	-33 (12)	33 (12)	50 (11)
$\phi 4C(4)$	-529 (49)	46911 (31)	12290 (28)	370 (19)	175 (12)	364 (19)	-10 (14)	-40 (14)	51 (13)
$\phi 4C(5)$	-8574 (45)	44089 (29)	16237 (26)	250 (17)	235 (14)	297 (16)	81 (12)	-11 (13)	-4 (11)
$\phi 4C(6)$	-8331 (36)	36600 (25)	18461 (21)	201 (14)	165 (11)	233 (13)	15 (9)	7 (11)	23 (9)

Intensity measurement

Spherical crystal, $r = 0.025$ (1) cm; mosaic spread, 0.25° . Four-circle computer-controlled diffractometer. Mo $K\alpha$ radiation with pyrolytic graphite monochromator (002). Take-off angle of 1.6° . Scintillation counter with 4 mm \times 5 mm aperture 26 cm from crystal. Peak shape discrimination used in counting circuitry. Coupled θ - 2θ step scan: step size $\delta 2\theta = 0.05^\circ$; scan range $\Delta 2\theta = 2(1 + 1.5 \tan \theta)$. Background taken as average of ten points at each end of scan. Four octants ($\pm h, \pm k, l$) of reciprocal space were examined. The total number of observations obtained was 4776. Of these, 57 were rejected for known malfunctions in equipment. Of the 2052 independent

Fig. 1. $C_{50}H_{40}N_2P_2$ (stereo pair). C(1) and C(2) define a twofold axis.

reflections, 1819 had been observed more than once; the average discrepancy between the intensities of these reflections was about 6 per cent. Only the 1335

Table 3. Fractional coordinates ($\times 10^4$) and isotropic U values (in \AA^2) $\times 10^3$ for the hydrogen atomsThe Debye-Waller factor is $\exp[-8\pi^2 U \sin^2 \theta / \lambda^2]$. The atom denoted $\phi 3H(5)$ is the hydrogen atom attached to C(5) of phenyl ring 3.

	x	y	z	U
$\phi 1H(2)$	-1598 (25)	-222 (15)	1923 (15)	-16 (10)
$\phi 1H(3)$	-1701 (34)	-1493 (22)	1861 (20)	35 (15)
$\phi 1H(4)$	-146 (34)	-2181 (26)	1495 (21)	57 (15)
$\phi 1H(5)$	1523 (39)	-1538 (26)	1218 (23)	70 (16)
$\phi 1H(6)$	1522 (28)	-200 (19)	1325 (17)	15 (11)
$\phi 2H(2)$	1065 (32)	309 (21)	4596 (20)	30 (13)
$\phi 2H(3)$	2774 (30)	452 (22)	5303 (21)	40 (13)
$\phi 2H(4)$	3925 (34)	1468 (20)	5095 (20)	33 (13)
$\phi 2H(5)$	3597 (37)	2185 (26)	4091 (22)	61 (16)
$\phi 2H(6)$	1866 (33)	1978 (22)	3352 (21)	39 (13)
$\phi 3H(2)$	-2238 (25)	1180 (17)	2898 (18)	1 (10)
$\phi 3H(3)$	-3953 (40)	1467 (24)	3326 (23)	58 (18)
$\phi 3H(4)$	-3770 (36)	1718 (21)	4549 (20)	32 (13)
$\phi 3H(5)$	-2086 (39)	1615 (26)	5296 (26)	81 (18)
$\phi 3H(6)$	-436 (35)	1306 (20)	4706 (21)	34 (15)
$\phi 4H(2)$	1367 (29)	3112 (20)	1094 (17)	20 (11)
$\phi 4H(3)$	1322 (34)	4384 (23)	751 (22)	44 (15)
$\phi 4H(4)$	-9 (38)	5203 (30)	1137 (27)	75 (19)
$\phi 4H(5)$	-1415 (36)	4669 (24)	1798 (23)	51 (18)
$\phi 4H(6)$	-1394 (27)	3476 (17)	2123 (16)	1 (10)

independent reflections with a measured intensity greater than $3\sigma_{\text{count}}$ (the Poisson counting statistical error) were used in the subsequent analysis. Structure amplitudes were derived by application of Lorentz and polarization corrections. No correction was necessary for absorption ($\mu r = 0.037$).

Table 4. Interatomic distances (Å) and angles (°)

Estimated standard deviations are in parentheses.

P-C(1)	1.726 (2)	N-C(2)	1.325 (4)
P- ϕ 1C(1)	1.804 (4)	N- ϕ 4C(1)	1.379 (4)
P- ϕ 2C(1)	1.823 (8)	C(1)-C(2)	1.477 (6)
P- ϕ 3C(1)	1.813 (6)		
P-C(1)-P	136.2 (3)	N-C(2)-C(1)	111.5 (2)
N-C(2)-N	137.0 (5)	P- ϕ 1C(1)- ϕ 1C(2)	120.7 (3)
P-C(1)-C(2)	111.9 (2)	P- ϕ 1C(1)- ϕ 1C(6)	121.9 (3)
C(1)-P- ϕ 1C(1)	113.2 (2)	P- ϕ 2C(1)- ϕ 2C(2)	119.3 (3)
C(1)-P- ϕ 2C(1)	115.8 (2)	P- ϕ 2C(1)- ϕ 2C(6)	121.9 (3)
C(1)-P- ϕ 3C(1)	112.9 (2)	P- ϕ 3C(1)- ϕ 3C(2)	117.7 (3)
ϕ 1C(1)-P- ϕ 2C(1)	100.5 (2)	P- ϕ 3C(1)- ϕ 3C(6)	123.3 (3)
ϕ 1C(1)-P- ϕ 3C(1)	104.2 (2)	N- ϕ 4C(1)- ϕ 4C(2)	117.6 (4)
ϕ 2C(1)-P- ϕ 3C(1)	109.1 (2)	N- ϕ 4C(1)- ϕ 4C(6)	125.1 (4)
C(2)-N- ϕ 4C(1)	126.1 (3)	P-C(1)-C(2)-N	9.7 (2)

Intra-ring distances and angles

	ϕ 1	ϕ 2	ϕ 3	ϕ 4
C(1)-C(2)	1.395 (5)	1.384 (6)	1.380 (7)	1.402 (6)
C(2)-C(3)	1.375 (6)	1.374 (8)	1.396 (7)	1.366 (6)
C(3)-C(4)	1.371 (7)	1.363 (7)	1.373 (6)	1.373 (6)
C(4)-C(5)	1.368 (7)	1.376 (7)	1.375 (8)	1.374 (7)
C(5)-C(6)	1.379 (6)	1.378 (8)	1.374 (7)	1.388 (6)
C(6)-C(1)	1.390 (5)	1.373 (5)	1.370 (5)	1.394 (5)
Mean C-C	1.379 (2)			

C(2)-H(2)	0.90 (3)	0.96 (4)	0.89 (3)	0.98 (3)
C(3)-H(3)	0.93 (4)	0.98 (4)	1.02 (4)	0.94 (4)
C(4)-H(4)	0.96 (4)	0.94 (4)	0.88 (4)	0.92 (5)
C(5)-H(5)	1.04 (5)	0.97 (4)	1.06 (5)	0.89 (4)
C(6)-H(6)	0.97 (3)	0.93 (4)	0.95 (4)	0.95 (3)
Mean C-H	0.95 (1)			

C(1)-C(2)-C(3)	121.7 (5)	121.9 (5)	121.0 (5)	121.8 (4)
C(2)-C(3)-C(4)	119.2 (5)	118.9 (5)	118.7 (5)	120.5 (5)
C(3)-C(4)-C(5)	121.0 (5)	120.1 (5)	120.5 (5)	118.5 (5)
C(4)-C(5)-C(6)	119.5 (5)	120.9 (5)	120.1 (5)	121.5 (5)
C(5)-C(6)-C(1)	121.4 (4)	119.6 (5)	120.8 (5)	119.9 (4)
C(6)-C(1)-C(2)	117.2 (4)	118.6 (4)	118.9 (4)	117.3 (4)

Structure solution and refinement

Direct methods, using the program *REL* (Long, 1965) followed by calculation of an *E* map with 276 data resulted in a satisfactory trial structure. Hydrogen atoms were located in a difference electron density synthesis. Final refinement was by full-matrix least-squares with two overlapping blocks of parameters; each block consisted of all parameters for two phenyl rings plus all other non-phenyl-ring parameters. Alternate refinement of these blocks was carried out until the shifts were less than 10 per cent of the corresponding standard deviations for all parameters.

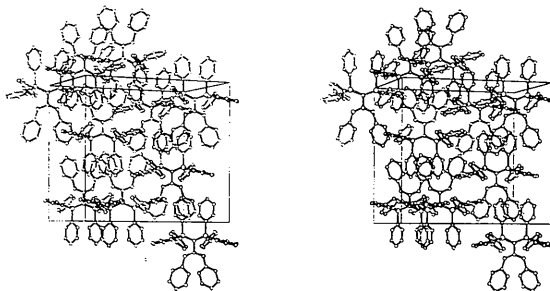


Fig. 2. Crystal structure of $C_{50}H_{40}N_2P_2$. The *b* axis is vertical and the *a* axis approximately perpendicular to the plane of the drawing (stereo pair).

The function minimized was $\sum w(|F_o| - |F_c|)^2$ with $w = 1/\sigma^2(F)$ and

$$\sigma(F) = \frac{1}{2F} [\sigma_{\text{count}}^2 + (0.05 F^2)^2]^{1/2}.$$

Atomic scattering factors for neutral P, N, C, and H (Cromer & Waber, 1972) and anomalous scattering coefficients f' and f'' (Cromer, 1965) for P were used in F_{calc} . An extinction parameter (Zachariasen, 1967) was included in the refinement, and its final value was $g = 2.13 (5) \times 10^{-5}$. The final measures of fit were

$$R = \sum |F_o| - |F_c| / \sum |F_o| = 0.037$$

$$R' = [\sum w |F_o| - |F_c|^2 / \sum w |F_o|^2]^{1/2} = 0.045$$

$$S = [\sum w |F_o| - |F_c|^2 / (N_o - N_v)]^{1/2} = 1.23$$

$$N_o = 1335, \quad N_v = 326.$$

Plots of $|F_o - F_c|/\sigma$ vs. $|F_o|$ and vs. $\sin \theta/\lambda$ indicated that the errors were independent of these parameters. Similarly, individual residuals (R and R') calculated for layers of data of constant h , constant k and constant l were in good agreement. Computer programs used have been described briefly by Schlemper, Hamilton & La Placa (1971).

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