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Dichlorido(dicyclohexylamino)-phosphine

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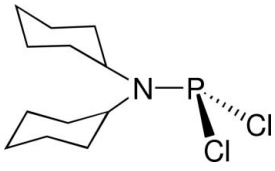
Key indicators: single-crystal X-ray study; T = 218 K; mean σ(C–C) = 0.002 Å; R factor = 0.041; wR factor = 0.097; data-to-parameter ratio = 23.4.

The title molecule, $PCl_2N(C_6H_{11})_2$ or $C_{12}H_{22}Cl_2NP$, adopts an approximate C_s symmetry with a plane passing through the P and N atoms and bisecting each cyclohexyl group. The Cl atoms lie on either side of this plane. The sum of angles around N is 359.98°. There are no intermolecular interactions.

Related literature

The molecular geometry and dimensions are comparable to those of PCl_2NMe_2 except that steric crowding of the cyclohexyl groups increases the C–N–C angle by approximately 3° (Mitzel, 1998). For related literature, see: Märkl & Alig (1984).



Experimental

Crystal data

$C_{12}H_{22}Cl_2NP$
M_r = 282.18
Monoclinic, $P2_1/n$
a = 6.4694 (8) Å
b = 17.8264 (18) Å
c = 12.939 (2) Å
β = 96.872 (10)°
V = 1481.5 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.52 mm^{-1}
T = 218 (2) K
0.60 × 0.60 × 0.40 mm

Data collection

Siemens P4 diffractometer
Absorption correction: ψ scan (*XPREP*; Siemens, 1996)
T_{min} = 0.825, T_{max} = 0.940
(expected range = 0.712–0.811)
4540 measured reflections
3387 independent reflections
3150 reflections with $I > 2\sigma(I)$
R_{int} = 0.087
3 standard reflections
every 97 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.097
S = 1.05
3387 reflections
145 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.44 e Å^{-3}

Data collection: *XSCANS* (Siemens, 1996); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Siemens, 1996); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2580).

References

Märkl, G. & Alig, B. (1984). *J. Organomet. Chem.* **273**, 1–29.
Mitzel, N. W. (1998). *J. Chem. Soc. Dalton Trans.* pp. 3239–3242.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany,.
Siemens (1996). *XSCANS* (Version 2.20) and *SHELXTL-Plus* (Version 5.03). Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

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supplementary materials

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Dichlorido(dicyclohexylamino)phosphine

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Comment

The title molecule, (I) (Fig. 1), adopts an approximate C_s symmetry with a plane bisecting the phosphorus and nitrogen atoms and each of the two cyclohexyl groups which are in chair conformations. The chlorides sit either side of this plane. The nitrogen is essentially planar (sum of angles = 359.98°). There are no intermolecular interactions.

Experimental

The title compound was prepared by literature methods (Märkl & Alig, 1984) and a suitable crystal was grown by crystallization from hexane.

Refinement

All H atoms were placed in calculated positions and refined using a riding model with C—H = 0.98Å for methylene groups and C—H = 0.99Å for tertiary H atoms and U(H)=1.2U_{eq}(C).

Figures

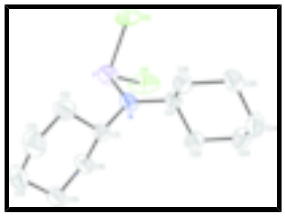

Fig. 1. The molecular structure of (I), with atom labels and 50% probability displacement ellipsoids. Hydrogen atoms have been excluded for clarity.

Dichloro(dicyclohexylamino)phosphine

Crystal data

$C_{12}H_{22}Cl_2NP$	F_{000} = 600
M_r = 282.18	D_x = 1.265 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 6.4694 (8) Å	Cell parameters from 30 reflections
b = 17.8264 (18) Å	θ = 5.5–12.5º
c = 12.939 (2) Å	μ = 0.52 mm^{-1}
β = 96.872 (10)º	T = 218 (2) K
V = 1481.5 (3) Å^3	Block, colourless
Z = 4	0.60 × 0.60 × 0.40 mm

Data collection

Siemens P4 diffractometer	R_{int} = 0.087
Radiation source: fine-focus sealed tube	θ_{max} = 27.5º
Monochromator: graphite	θ_{min} = 2.0º
T = 218(2) K	h = −1→8
profile fitting of θ/2θ scans	k = −1→23
Absorption correction: ψ scan (PROGRAM?; REFERENCE?)	l = −16→16
T_{min} = 0.825, T_{max} = 0.940	3 standard reflections
4540 measured reflections	every 97 reflections
3387 independent reflections	intensity decay: none
3150 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.041	H-atom parameters constrained
$wR(F^2)$ = 0.097	$w = 1/[\sigma^2(F_o^2) + (0.0422P)^2 + 0.5948P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.05	$(\Delta/\sigma)_{max} < 0.001$
3387 reflections	$\Delta\rho_{max}$ = 0.42 e Å^{-3}
145 parameters	$\Delta\rho_{min}$ = −0.43 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
P	0.39761 (7)	0.13232 (2)	0.62770 (3)	0.04246 (13)
Cl1	0.25915 (8)	0.17300 (4)	0.75460 (3)	0.06987 (19)
Cl2	0.69212 (6)	0.17705 (3)	0.67608 (3)	0.04867 (13)

N	0.3064 (2)	0.18837 (7)	0.53336 (9)	0.0338 (3)
C11	0.2174 (2)	0.15378 (8)	0.43283 (10)	0.0304 (3)
H11	0.1720	0.1955	0.3849	0.036*
C12	0.3785 (3)	0.10924 (11)	0.38213 (13)	0.0460 (4)
H12A	0.4303	0.0679	0.4281	0.055*
H12B	0.4966	0.1418	0.3722	0.055*
C13	0.2853 (3)	0.07775 (12)	0.27726 (15)	0.0558 (5)
H13A	0.2487	0.1192	0.2288	0.067*
H13B	0.3892	0.0466	0.2484	0.067*
C14	0.0926 (3)	0.03115 (10)	0.28753 (14)	0.0522 (4)
H14A	0.0311	0.0148	0.2183	0.063*
H14B	0.1321	−0.0137	0.3291	0.063*
C15	−0.0672 (3)	0.07542 (10)	0.33883 (14)	0.0456 (4)
H15A	−0.1855	0.0429	0.3484	0.055*
H15B	−0.1189	0.1169	0.2932	0.055*
C16	0.0250 (2)	0.10661 (9)	0.44403 (12)	0.0394 (3)
H16A	−0.0792	0.1375	0.4731	0.047*
H16B	0.0627	0.0651	0.4923	0.047*
C21	0.3110 (2)	0.27110 (7)	0.54083 (10)	0.0285 (3)
H21	0.3746	0.2839	0.6121	0.034*
C22	0.0923 (2)	0.30448 (9)	0.52723 (13)	0.0393 (3)
H22A	0.0125	0.2843	0.5807	0.047*
H22B	0.0208	0.2903	0.4589	0.047*
C23	0.1021 (3)	0.38991 (10)	0.53599 (16)	0.0507 (4)
H23A	−0.0390	0.4105	0.5225	0.061*
H23B	0.1590	0.4039	0.6070	0.061*
C24	0.2366 (3)	0.42339 (9)	0.45938 (15)	0.0508 (4)
H24A	0.1733	0.4131	0.3881	0.061*
H24B	0.2441	0.4779	0.4687	0.061*
C25	0.4545 (3)	0.39077 (9)	0.47516 (13)	0.0424 (3)
H25A	0.5223	0.4048	0.5443	0.051*
H25B	0.5366	0.4117	0.4231	0.051*
C26	0.4490 (2)	0.30532 (8)	0.46541 (11)	0.0333 (3)
H26A	0.3954	0.2913	0.3940	0.040*
H26B	0.5906	0.2854	0.4803	0.040*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P	0.0523 (3)	0.0380 (2)	0.0340 (2)	−0.00576 (17)	−0.00762 (17)	0.01147 (15)
Cl1	0.0472 (2)	0.1311 (5)	0.0323 (2)	−0.0049 (3)	0.00896 (17)	0.0195 (2)
Cl2	0.0336 (2)	0.0675 (3)	0.0429 (2)	0.00425 (17)	−0.00350 (15)	0.00985 (18)
N	0.0439 (7)	0.0295 (6)	0.0260 (5)	−0.0043 (5)	−0.0046 (5)	0.0024 (4)
C11	0.0345 (7)	0.0303 (6)	0.0254 (6)	−0.0021 (5)	0.0000 (5)	0.0000 (5)
C12	0.0372 (8)	0.0569 (10)	0.0446 (9)	0.0003 (7)	0.0073 (6)	−0.0099 (7)
C13	0.0584 (11)	0.0672 (12)	0.0441 (9)	0.0000 (9)	0.0160 (8)	−0.0195 (8)
C14	0.0636 (11)	0.0451 (9)	0.0474 (9)	−0.0027 (8)	0.0035 (8)	−0.0180 (7)
C15	0.0420 (8)	0.0460 (9)	0.0475 (9)	−0.0091 (7)	0.0002 (7)	−0.0118 (7)

C16	0.0392 (7)	0.0416 (8)	0.0380 (8)	−0.0079 (6)	0.0074 (6)	−0.0064 (6)
C21	0.0298 (6)	0.0292 (6)	0.0258 (6)	−0.0010 (5)	0.0004 (5)	−0.0003 (5)
C22	0.0307 (7)	0.0437 (8)	0.0440 (8)	0.0014 (6)	0.0059 (6)	−0.0004 (6)
C23	0.0454 (9)	0.0454 (9)	0.0608 (11)	0.0131 (7)	0.0039 (8)	−0.0111 (8)
C24	0.0644 (11)	0.0272 (7)	0.0583 (11)	0.0028 (7)	−0.0031 (8)	0.0005 (7)
C25	0.0487 (9)	0.0338 (7)	0.0443 (8)	−0.0109 (6)	0.0034 (7)	0.0015 (6)
C26	0.0332 (7)	0.0332 (7)	0.0340 (7)	−0.0017 (5)	0.0058 (5)	0.0016 (5)

Geometric parameters (Å, °)

P—N	1.6322 (12)	C16—H16A	0.9800
P—Cl2	2.0910 (6)	C16—H16B	0.9800
P—Cl1	2.0920 (8)	C21—C22	1.5259 (19)
N—C21	1.4780 (17)	C21—C26	1.5263 (18)
N—C11	1.4915 (17)	C21—H21	0.9900
C11—C12	1.520 (2)	C22—C23	1.528 (2)
C11—C16	1.523 (2)	C22—H22A	0.9800
C11—H11	0.9900	C22—H22B	0.9800
C12—C13	1.525 (2)	C23—C24	1.518 (3)
C12—H12A	0.9800	C23—H23A	0.9800
C12—H12B	0.9800	C23—H23B	0.9800
C13—C14	1.517 (3)	C24—C25	1.516 (3)
C13—H13A	0.9800	C24—H24A	0.9800
C13—H13B	0.9800	C24—H24B	0.9800
C14—C15	1.516 (2)	C25—C26	1.529 (2)
C14—H14A	0.9800	C25—H25A	0.9800
C14—H14B	0.9800	C25—H25B	0.9800
C15—C16	1.524 (2)	C26—H26A	0.9800
C15—H15A	0.9800	C26—H26B	0.9800
C15—H15B	0.9800		
N—P—Cl2	103.02 (5)	C11—C16—H16B	109.5
N—P—Cl1	102.97 (5)	C15—C16—H16B	109.5
Cl2—P—Cl1	95.35 (3)	H16A—C16—H16B	108.1
C21—N—C11	118.18 (10)	N—C21—C22	111.74 (12)
C21—N—P	124.00 (9)	N—C21—C26	111.44 (11)
C11—N—P	117.80 (9)	C22—C21—C26	112.07 (11)
N—C11—C12	112.28 (12)	N—C21—H21	107.1
N—C11—C16	112.33 (12)	C22—C21—H21	107.1
C12—C11—C16	111.28 (13)	C26—C21—H21	107.1
N—C11—H11	106.8	C21—C22—C23	110.51 (13)
C12—C11—H11	106.8	C21—C22—H22A	109.5
C16—C11—H11	106.8	C23—C22—H22A	109.5
C11—C12—C13	110.96 (14)	C21—C22—H22B	109.5
C11—C12—H12A	109.4	C23—C22—H22B	109.5
C13—C12—H12A	109.4	H22A—C22—H22B	108.1
C11—C12—H12B	109.4	C24—C23—C22	111.44 (14)
C13—C12—H12B	109.4	C24—C23—H23A	109.3
H12A—C12—H12B	108.0	C22—C23—H23A	109.3
C14—C13—C12	111.44 (15)	C24—C23—H23B	109.3

C14—C13—H13A	109.3	C22—C23—H23B	109.3
C12—C13—H13A	109.3	H23A—C23—H23B	108.0
C14—C13—H13B	109.3	C25—C24—C23	110.97 (14)
C12—C13—H13B	109.3	C25—C24—H24A	109.4
H13A—C13—H13B	108.0	C23—C24—H24A	109.4
C15—C14—C13	111.35 (14)	C25—C24—H24B	109.4
C15—C14—H14A	109.4	C23—C24—H24B	109.4
C13—C14—H14A	109.4	H24A—C24—H24B	108.0
C15—C14—H14B	109.4	C24—C25—C26	111.05 (13)
C13—C14—H14B	109.4	C24—C25—H25A	109.4
H14A—C14—H14B	108.0	C26—C25—H25A	109.4
C14—C15—C16	111.49 (14)	C24—C25—H25B	109.4
C14—C15—H15A	109.3	C26—C25—H25B	109.4
C16—C15—H15A	109.3	H25A—C25—H25B	108.0
C14—C15—H15B	109.3	C21—C26—C25	110.79 (12)
C16—C15—H15B	109.3	C21—C26—H26A	109.5
H15A—C15—H15B	108.0	C25—C26—H26A	109.5
C11—C16—C15	110.70 (13)	C21—C26—H26B	109.5
C11—C16—H16A	109.5	C25—C26—H26B	109.5
C15—C16—H16A	109.5	H26A—C26—H26B	108.1
Cl2—P—N—C21	−48.37 (12)	C12—C11—C16—C15	−55.90 (18)
Cl1—P—N—C21	50.35 (12)	C14—C15—C16—C11	55.61 (19)
Cl2—P—N—C11	130.23 (10)	C11—N—C21—C22	63.11 (16)
Cl1—P—N—C11	−131.06 (10)	P—N—C21—C22	−118.30 (13)
C21—N—C11—C12	116.26 (14)	C11—N—C21—C26	−63.14 (16)
P—N—C11—C12	−62.42 (16)	P—N—C21—C26	115.45 (12)
C21—N—C11—C16	−117.42 (14)	N—C21—C22—C23	179.66 (13)
P—N—C11—C16	63.90 (15)	C26—C21—C22—C23	−54.44 (17)
N—C11—C12—C13	−177.33 (14)	C21—C22—C23—C24	55.33 (19)
C16—C11—C12—C13	55.79 (19)	C22—C23—C24—C25	−57.0 (2)
C11—C12—C13—C14	−55.2 (2)	C23—C24—C25—C26	56.89 (19)
C12—C13—C14—C15	55.1 (2)	N—C21—C26—C25	−179.22 (12)
C13—C14—C15—C16	−55.3 (2)	C22—C21—C26—C25	54.72 (16)
N—C11—C16—C15	177.25 (12)	C24—C25—C26—C21	−55.55 (17)

Fig. 1