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1,4-Bis(5,5-dimethyl-2-oxo-4-phenyl-1,3,2-dioxaphosphorinan-2-yl)piperazine

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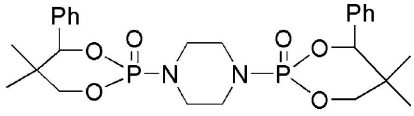
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.005 Å; R factor = 0.062; wR factor = 0.151; data-to-parameter ratio = 15.9.

The title compound, $C_{26}H_{36}N_2O_6P_2$, possesses a crystallographically imposed inversion centre. The molecules are linked into a one-dimensional structure along the c axis by a centrosymmetric system of weak C—H···O hydrogen bonds.

Related literature

For ring puckering analysis, see: Cremer & Pople (1975).



Experimental

Crystal data

$C_{26}H_{36}N_2O_6P_2$
M_r = 534.51
Triclinic, $P\overline{1}$
a = 6.4436 (7) Å
b = 10.4563 (11) Å
c = 11.5762 (13) Å
α = 65.680 (2)°
β = 86.755 (2)°
γ = 72.641 (2)°
V = 676.40 (18) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.20 mm^{-1}
T = 295 (2) K
0.20 × 0.20 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
T_{min} = 0.951, T_{max} = 0.980
7086 measured reflections
2616 independent reflections
1754 reflections with $I > 2\sigma(I)$
R_{int} = 0.047

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.151
S = 1.06
2616 reflections
165 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C9—H9B···O8^{ii}	0.96	2.56	3.459 (4)	157

Symmetry code: (ii) $-x+2, -y+1, -z+1$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BG2110).

References

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Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
Sheldrick, G. M. (1997). *SADABS*, *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

supplementary materials

Acta Cryst. (2007). E**63**, o4271 [**doi:10.1107/S1600536807048349**]

1,4-Bis(5,5-dimethyl-2-oxo-4-phenyl-1,3,2-dioxaphosphorinan-2-yl)piperazine

X. Chen, W. Xiao and P. Jiao

Comment

Organic phosphorus compounds are widely used in pesticide science because of their significant biological properties. The title compound has been used as a precursor of organic phosphorus pesticides. The title dimeric molecule possesses a crystallographically imposed centre of symmetry, as shown in Fig.1. The central piperazine ring (N1, C12, C13, $N1^{i}$,$C12^{i}$,$C13^{i}$; (*i*): −*x*, 1 − *y*, 2 − *z*) adopts a chair conformation,with puckering parameters (Cremer & Pople,1975) θ = 180°, φ = 173.6° and Q = 0.5916 (1) Å. The oxaphosphorinane ring (P1, O6, O7, C7, C8, C11) also displays a chair conformation, with puckering parameters θ = 14.20 (13)°, φ = 172.2° and Q = 0.5363 (16) Å. Analysis of P—O bond distances (Table 1) indicate a clear P1═O8 double-bond character. The structure is stabilized by a centrosymmetric array of intermolecular hydrogen bonds. (Table 2).

Experimental

A mixture of 2-chloro-5,5-dimethyl-4-phenyl-1,3,2-dioxaphosphinane 2-oxide(10 mmol) and piperazine (5 mmol) in THF (30 ml) was stirred for 4 h and the solvent was removed under reduced pressure. After filtration, crystals suitable for X-ray investigation were obtained by recrystallization from methanol and chloroform (1:1 *v*/*v*).

Refinement

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.96 Å (methyl) or 0.97 Å (methylene); U_{iso}(H) = xU_{eq}(carrier atom), where *x* = 1.5 for methyl and 1.2 for methylene.

Figures

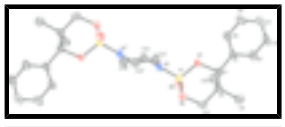


Fig. 1. The structure of (I), with displacement ellipsoids drawn at the 30% probability level. H atoms have been omitted. Symmetry codes: (i) −*x* + 2, −*y* + 1, −*z*.

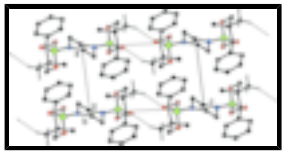


Fig. 2. Packing diagram of the title molecule viewed down the *b* axis.

1,4-Bis(5,5-dimethyl-2-oxo-4-phenyl-1,3,2-dioxaphosphorinan-2-yl)piperazine

Crystal data

$C_{26}H_{36}N_2O_6P_2$	$Z = 1$
$M_r = 534.51$	$F_{000} = 284$
Triclinic, $P\overline{1}$	$D_x = 1.312$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 6.4436$ (7) Å	Cell parameters from 1049 reflections
$b = 10.4563$ (11) Å	$\theta = 2.3$–20.0º
$c = 11.5762$ (13) Å	$\mu = 0.20$ mm^{-1}
$\alpha = 65.680$ (2)º	$T = 295$ (2) K
$\beta = 86.755$ (2)º	Plate, colorless
$\gamma = 72.641$ (2)º	0.20 × 0.20 × 0.10 mm
$V = 676.40$ (18) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	2616 independent reflections
Radiation source: fine-focus sealed tube	1754 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.047$
$T = 295$(2) K	$\theta_{max} = 26.0$º
φ and ω scans	$\theta_{min} = 1.9$º
Absorption correction: multi-scan (SADABS; Sheldrick, 1997)	$h = -7 \rightarrow 7$
$T_{min} = 0.951$, $T_{max} = 0.980$	$k = -12 \rightarrow 12$
7086 measured reflections	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.062$	H-atom parameters constrained
$wR(F^2) = 0.151$	$w = 1/[\sigma^2(F_o^2) + (0.0717P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} < 0.001$
2616 reflections	$\Delta\rho_{max} = 0.30$ e Å^{-3}
165 parameters	$\Delta\rho_{min} = -0.23$ 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}
C1	0.8132 (4)	0.8979 (3)	0.2863 (3)	0.0363 (7)
C2	0.7646 (5)	0.9464 (4)	0.3830 (3)	0.0539 (9)
H2	0.8259	0.8852	0.4654	0.065*
C3	0.6243 (6)	1.0863 (4)	0.3556 (4)	0.0690 (11)
H3	0.5952	1.1192	0.4200	0.083*
C4	0.5276 (6)	1.1771 (4)	0.2359 (4)	0.0685 (11)
H4	0.4326	1.2703	0.2192	0.082*
C5	0.5726 (5)	1.1291 (3)	0.1405 (3)	0.0578 (9)
H5	0.5079	1.1899	0.0588	0.069*
C6	0.7137 (5)	0.9904 (3)	0.1661 (3)	0.0441 (8)
H6	0.7423	0.9586	0.1011	0.053*
C7	0.9697 (4)	0.7486 (3)	0.3152 (2)	0.0353 (7)
H7	0.9530	0.6826	0.4022	0.042*
C8	1.2127 (5)	0.7404 (3)	0.3031 (3)	0.0403 (7)
C9	1.2908 (5)	0.7813 (4)	0.4025 (3)	0.0565 (9)
H9A	1.4460	0.7625	0.4026	0.085*
H9B	1.2541	0.7231	0.4851	0.085*
H9C	1.2211	0.8838	0.3819	0.085*
C10	1.2518 (5)	0.8427 (4)	0.1706 (3)	0.0517 (9)
H10A	1.1765	0.9430	0.1558	0.078*
H10B	1.1983	0.8196	0.1082	0.078*
H10C	1.4052	0.8301	0.1644	0.078*
C11	1.3430 (5)	0.5815 (4)	0.3337 (3)	0.0519 (9)
H11A	1.4967	0.5736	0.3281	0.062*
H11B	1.3227	0.5200	0.4203	0.062*
C12	0.8516 (5)	0.4487 (3)	0.0935 (3)	0.0468 (8)
H12A	0.7105	0.5178	0.0542	0.056*
H12B	0.8300	0.3779	0.1758	0.056*
C13	1.0454 (5)	0.6299 (3)	−0.0110 (3)	0.0474 (8)
H13A	1.1448	0.6754	0.0054	0.057*
H13B	0.9129	0.7072	−0.0553	0.057*
N1	0.9939 (4)	0.5274 (3)	0.1097 (2)	0.0446 (7)
O6	1.2784 (3)	0.5277 (2)	0.2478 (2)	0.0523 (6)

O7	0.9065 (3)	0.69522 (19)	0.22708 (16)	0.0366 (5)
O8	0.9706 (4)	0.4198 (2)	0.35255 (19)	0.0615 (7)
P1	1.03157 (13)	0.53246 (8)	0.24428 (7)	0.0406 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0407 (17)	0.0387 (18)	0.0388 (17)	−0.0189 (14)	0.0090 (13)	−0.0210 (14)
C2	0.064 (2)	0.069 (2)	0.0484 (19)	−0.0254 (19)	0.0118 (16)	−0.0397 (19)
C3	0.081 (3)	0.073 (3)	0.083 (3)	−0.026 (2)	0.026 (2)	−0.062 (2)
C4	0.068 (3)	0.047 (2)	0.094 (3)	−0.0086 (19)	0.016 (2)	−0.040 (2)
C5	0.061 (2)	0.042 (2)	0.060 (2)	−0.0040 (17)	0.0071 (17)	−0.0196 (18)
C6	0.0518 (19)	0.0436 (19)	0.0415 (18)	−0.0151 (16)	0.0099 (14)	−0.0225 (16)
C7	0.0467 (17)	0.0381 (17)	0.0278 (15)	−0.0185 (14)	0.0027 (12)	−0.0162 (13)
C8	0.0425 (17)	0.048 (2)	0.0389 (17)	−0.0194 (15)	0.0017 (13)	−0.0224 (15)
C9	0.061 (2)	0.075 (2)	0.0468 (19)	−0.0311 (19)	−0.0022 (16)	−0.0301 (19)
C10	0.054 (2)	0.068 (2)	0.0471 (19)	−0.0331 (18)	0.0125 (15)	−0.0274 (18)
C11	0.0426 (19)	0.061 (2)	0.052 (2)	−0.0089 (17)	−0.0087 (15)	−0.0269 (18)
C12	0.057 (2)	0.049 (2)	0.0458 (18)	−0.0274 (17)	0.0095 (15)	−0.0241 (16)
C13	0.067 (2)	0.0448 (19)	0.0462 (18)	−0.0309 (17)	0.0106 (16)	−0.0255 (16)
N1	0.0675 (18)	0.0442 (16)	0.0407 (14)	−0.0323 (14)	0.0144 (12)	−0.0259 (13)
O6	0.0456 (13)	0.0540 (14)	0.0625 (14)	−0.0031 (11)	−0.0054 (10)	−0.0362 (12)
O7	0.0406 (11)	0.0384 (12)	0.0393 (11)	−0.0137 (9)	0.0003 (9)	−0.0226 (10)
O8	0.105 (2)	0.0458 (14)	0.0401 (13)	−0.0361 (13)	0.0091 (12)	−0.0151 (11)
P1	0.0545 (5)	0.0346 (5)	0.0364 (5)	−0.0135 (4)	0.0018 (4)	−0.0181 (4)

Geometric parameters (Å, °)

P1—N1	1.615 (2)	C8—C10	1.525 (4)
P1—O6	1.578 (2)	C8—C9	1.536 (4)
P1—O7	1.583 (2)	C9—H9A	0.9600
P1—O8	1.455 (2)	C9—H9B	0.9600
C1—C6	1.380 (4)	C9—H9C	0.9600
C1—C2	1.392 (3)	C10—H10A	0.9600
C1—C7	1.495 (4)	C10—H10B	0.9600
C2—C3	1.386 (4)	C10—H10C	0.9600
C2—H2	0.9300	C11—O6	1.458 (3)
C3—C4	1.368 (5)	C11—H11A	0.9700
C3—H3	0.9300	C11—H11B	0.9700
C4—C5	1.374 (4)	C12—N1	1.466 (4)
C4—H4	0.9300	C12—C13^{i}	1.509 (4)
C5—C6	1.381 (4)	C12—H12A	0.9700
C5—H5	0.9300	C12—H12B	0.9700
C6—H6	0.9300	C13—N1	1.462 (4)
C7—O7	1.473 (3)	C13—C12^{i}	1.509 (4)
C7—C8	1.544 (4)	C13—H13A	0.9700
C7—H7	0.9800	C13—H13B	0.9700
C8—C11	1.519 (4)		

C6—C1—C2	118.4 (3)	C8—C10—H10A	109.5
C6—C1—C7	122.1 (2)	C8—C10—H10B	109.5
C2—C1—C7	119.5 (3)	H10A—C10—H10B	109.5
C3—C2—C1	119.6 (3)	C8—C10—H10C	109.5
C3—C2—H2	120.2	H10A—C10—H10C	109.5
C1—C2—H2	120.2	H10B—C10—H10C	109.5
C4—C3—C2	121.3 (3)	O6—C11—C8	112.3 (2)
C4—C3—H3	119.3	O6—C11—H11A	109.1
C2—C3—H3	119.3	C8—C11—H11A	109.1
C3—C4—C5	119.3 (3)	O6—C11—H11B	109.1
C3—C4—H4	120.3	C8—C11—H11B	109.1
C5—C4—H4	120.3	H11A—C11—H11B	107.9
C4—C5—C6	120.0 (3)	N1—C12—C13^{i}	109.5 (2)
C4—C5—H5	120.0	N1—C12—H12A	109.8
C6—C5—H5	120.0	C13^{i}—C12—H12A	109.8
C1—C6—C5	121.3 (3)	N1—C12—H12B	109.8
C1—C6—H6	119.3	C13^{i}—C12—H12B	109.8
C5—C6—H6	119.3	H12A—C12—H12B	108.2
O7—C7—C1	107.3 (2)	N1—C13—C12^{i}	110.8 (2)
O7—C7—C8	108.33 (19)	N1—C13—H13A	109.5
C1—C7—C8	116.0 (2)	C12^{i}—C13—H13A	109.5
O7—C7—H7	108.3	N1—C13—H13B	109.5
C1—C7—H7	108.3	C12^{i}—C13—H13B	109.5
C8—C7—H7	108.3	H13A—C13—H13B	108.1
C11—C8—C10	111.0 (2)	C13—N1—C12	113.1 (2)
C11—C8—C9	107.0 (2)	C13—N1—P1	123.04 (18)
C10—C8—C9	109.4 (2)	C12—N1—P1	121.8 (2)
C11—C8—C7	107.8 (2)	C11—O6—P1	116.17 (17)
C10—C8—C7	111.6 (2)	C7—O7—P1	118.41 (16)
C9—C8—C7	109.9 (2)	O8—P1—O6	115.83 (13)
C8—C9—H9A	109.5	O8—P1—O7	113.86 (12)
C8—C9—H9B	109.5	O6—P1—O7	102.54 (10)
H9A—C9—H9B	109.5	O8—P1—N1	114.05 (12)
C8—C9—H9C	109.5	O6—P1—N1	103.11 (12)
H9A—C9—H9C	109.5	O7—P1—N1	106.11 (11)
H9B—C9—H9C	109.5		
C6—C1—C2—C3	−1.9 (5)	C7—C8—C11—O6	59.9 (3)
C7—C1—C2—C3	178.2 (3)	C12^{i}—C13—N1—C12	−56.6 (4)
C1—C2—C3—C4	1.7 (5)	C12^{i}—C13—N1—P1	139.4 (2)
C2—C3—C4—C5	−0.7 (6)	C13^{i}—C12—N1—C13	55.9 (4)
C3—C4—C5—C6	0.1 (5)	C13^{i}—C12—N1—P1	−139.9 (2)
C2—C1—C6—C5	1.3 (5)	C8—C11—O6—P1	−58.8 (3)
C7—C1—C6—C5	−178.8 (3)	C1—C7—O7—P1	−174.51 (16)
C4—C5—C6—C1	−0.4 (5)	C8—C7—O7—P1	59.6 (3)
C6—C1—C7—O7	−29.3 (3)	C11—O6—P1—O8	−75.5 (2)
C2—C1—C7—O7	150.5 (2)	C11—O6—P1—O7	49.2 (2)
C6—C1—C7—C8	91.9 (3)	C11—O6—P1—N1	159.3 (2)

C2—C1—C7—C8	−88.3 (3)	C7—O7—P1—O8	74.4 (2)
O7—C7—C8—C11	−58.3 (3)	C7—O7—P1—O6	−51.54 (19)
C1—C7—C8—C11	−179.0 (2)	C7—O7—P1—N1	−159.37 (17)
O7—C7—C8—C10	63.8 (3)	C13—N1—P1—O8	−179.7 (2)
C1—C7—C8—C10	−56.9 (3)	C12—N1—P1—O8	17.8 (3)
O7—C7—C8—C9	−174.7 (2)	C13—N1—P1—O6	−53.2 (3)
C1—C7—C8—C9	64.7 (3)	C12—N1—P1—O6	144.2 (2)
C10—C8—C11—O6	−62.6 (3)	C13—N1—P1—O7	54.2 (3)
C9—C8—C11—O6	178.1 (2)	C12—N1—P1—O7	−108.4 (2)

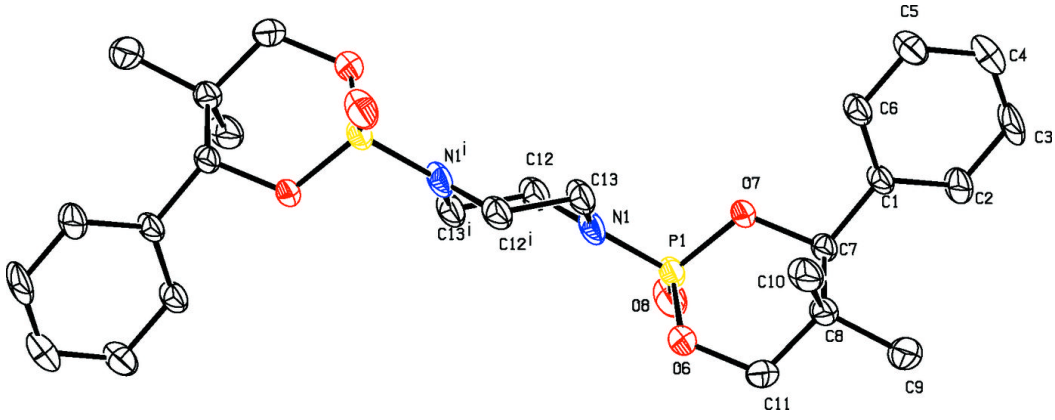
Symmetry codes: (i) −*x*+2, −*y*+1, −*z*.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12B···O8	0.97	2.53	3.008 (4)	111
C9—H9B···O8^{ii}	0.96	2.56	3.459 (4)	157

Symmetry codes: (ii) −*x*+2, −*y*+1, −*z*+1.

Fig. 1


C5
C4
C6
C3
C1
C2
O7
C7
C10
C8
C9
C11
O6
O8
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N1
C13
C12
N1i
C12i
C13i

Fig. 2

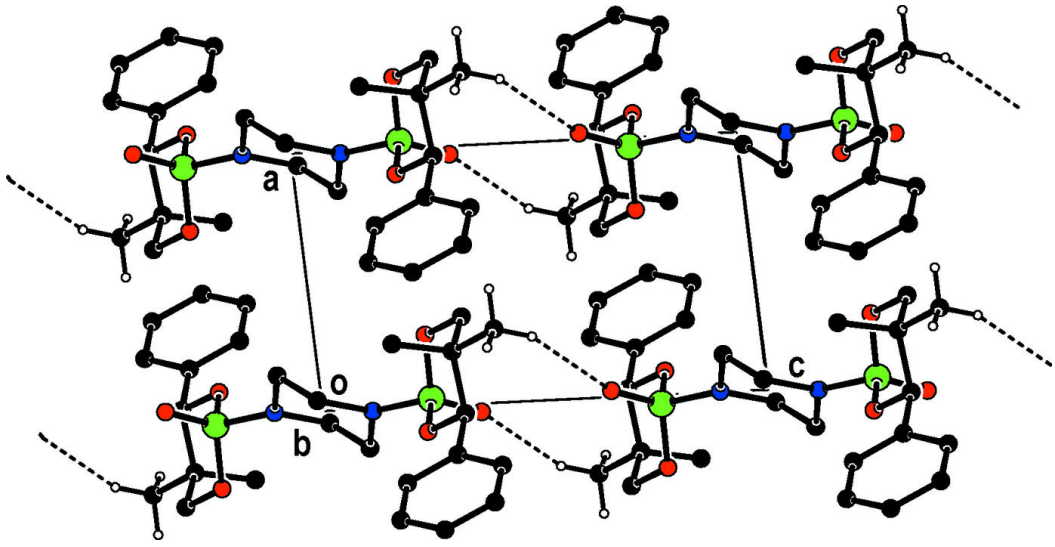

Fig. 3