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Structure of Diethyl 4-Ethoxycarbonyl-3,4-dihydrobenzo[*f*]quinoline-3-phosphonate

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Abstract. $C_{20}H_{24}NO_5P$, $M_r = 389{\cdot}40$, monoclinic, $P2_1/a$, $a = 15{\cdot}332\ (3)$, $b = 10{\cdot}603\ (6)$, $c = 12{\cdot}442\ (6)$ Å, $\beta = 100{\cdot}89\ (3)°$, $V = 1986\ (1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}30$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}7107$ Å, $\mu = 1{\cdot}84$ cm^{-1}, $F(000) = 824$, room temperature, $R = 0{\cdot}059$ for 2108 observed reflections [$F_o > 3\sigma(F_o)$]. Out of 11 C—C bonds of the biphenyl ring, four bonds (1·35–1·38 Å) are significantly shorter than the others (1·40–1·43 Å). Two P—O single bonds are 1·564 (4) and 1·560 (4) Å in length, and the P—O double-bond length is 1·454 (4) Å.

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Experimental. Title compound prepared according to the literature (Takeuchi, Shibata & Hamada, 1984). Colorless crystals obtained from ethanol solution. Crystal of dimensions 0·3 × 0·2 × 0·2 mm, Rigaku AFC-1 rotating-anode four-circle diffractometer, graphite-monochromatized Mo $K\alpha$ radiation. Cell dimensions determined from 16 2θ angles in the range $17 < 2\theta < 23°$. Intensities collected to $\sin\theta/\lambda =$

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{4}{3}[B_{11}a^2 + B_{22}b^2 + B_{33}c^2 + abB_{12}(\cos\gamma) + acB_{13}(\cos\beta) + bcB_{23}(\cos\alpha)].$$

	x	y	z	B_{eq}
P	0·5962 (1)	0·8906 (1)	0·1953 (1)	4·6 (0)
N	0·7481 (3)	0·7466 (3)	0·2224 (3)	4·3 (1)
O1	0·6079 (2)	0·8781 (3)	0·3226 (2)	5·2 (1)
O2	0·5597 (3)	1·0084 (3)	0·1463 (3)	6·8 (1)
O3	0·5422 (2)	0·7693 (3)	0·1552 (2)	5·8 (1)
O4	0·7216 (3)	0·6507 (4)	0·0566 (2)	6·8 (1)
O5	0·8056 (2)	0·5560 (3)	0·2010 (2)	5·4 (1)
C1	0·7090 (3)	0·8638 (4)	0·1737 (3)	4·7 (1)
C2	0·7684 (3)	0·9730 (5)	0·2099 (4)	5·0 (1)
C3	0·8237 (3)	0·9699 (4)	0·3063 (4)	4·8 (1)
C4	0·8246 (3)	0·8616 (4)	0·3794 (3)	3·8 (1)
C5	0·8607 (3)	0·8687 (4)	0·4942 (3)	3·8 (1)
C6	0·9025 (3)	0·9773 (5)	0·5451 (4)	5·0 (1)
C7	0·9369 (4)	0·9797 (5)	0·6543 (4)	6·1 (2)
C8	0·9317 (4)	0·8741 (6)	0·7198 (4)	6·4 (2)
C9	0·8915 (4)	0·7688 (5)	0·6748 (4)	5·5 (1)
C10	0·8540 (3)	0·7626 (4)	0·5607 (3)	4·1 (1)
C11	0·8130 (3)	0·6513 (4)	0·5125 (3)	4·3 (1)
C12	0·7797 (3)	0·6448 (4)	0·4034 (3)	4·0 (1)
C13	0·7872 (3)	0·7498 (4)	0·3367 (3)	3·5 (1)
C14	0·5358 (4)	0·8644 (7)	0·3794 (5)	7·5 (2)
C15	0·5730 (4)	0·8440 (5)	0·4958 (5)	7·1 (2)
C16	0·4985 (4)	0·7545 (6)	0·0410 (4)	7·4 (2)
C17	0·4262 (5)	0·6682 (11)	0·0347 (6)	14·4 (4)
C18	0·7558 (3)	0·6491 (5)	0·1520 (4)	5·0 (1)
C19	0·8167 (4)	0·4481 (5)	0·1311 (5)	7·0 (2)
C20	0·8487 (5)	0·3426 (6)	0·1990 (5)	8·6 (2)

Table 2 *Bond lengths* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

P—O1	1·566 (4)	P—O2	1·454 (4)	P—O3	1·559 (4)
P—C1	1·822 (5)	N—C1	1·460 (6)	N—C13	1·436 (6)
O1—C14	1·427 (8)	O3—C16	1·460 (7)	O4—C18	1·203 (6)
O5—C18	1·324 (6)	O5—C19	1·466 (8)	C2—C3	1·332 (7)
C3—C4	1·464 (7)	C4—C5	1·433 (6)	C4—C13	1·379 (6)
C5—C6	1·409 (6)	C5—C10	1·412 (6)	C6—C7	1·362 (7)
C7—C8	1·396 (8)	C8—C9	1·346 (8)	C9—C10	1·429 (7)
C10—C11	1·416 (7)	C11—C12	1·359 (7)	C12—C13	1·406 (6)
C14—C15	1·468 (10)	C16—C17	1·428 (13)	C19—C20	1·431 (10)
O1—P—O2	117·2 (2)	O1—P—O3	101·9 (2)		
O1—P—C1	101·9 (2)	O2—P—O3	115·8 (2)		
O2—P—C1	111·9 (2)	O3—P—C1	106·6 (2)		
C1—N—C13	116·9 (4)	C1—N—C18	117·0 (4)		
C13—N—C18	125·4 (4)	P—O1—C14	124·0 (4)		
P—O3—C16	121·2 (4)	C18—O5—C19	115·5 (4)		
P—C1—N	113·5 (3)	P—C1—C2	112·2 (4)		
N—C1—C2	110·9 (4)	C1—C2—C3	120·1 (5)		
C2—C3—C4	120·6 (5)	C3—C4—C5	122·3 (4)		
C3—C4—C13	118·8 (4)	C5—C4—C13	118·9 (4)		
C4—C5—C6	123·2 (4)	C4—C5—C10	119·0 (4)		
C6—C5—C10	117·7 (4)	C5—C6—C7	121·4 (5)		
C6—C7—C8	120·9 (5)	C7—C8—C9	119·6 (6)		
C8—C9—C10	121·2 (5)	C5—C10—C9	119·1 (4)		
C5—C10—C11	119·4 (4)	C9—C10—C11	121·5 (4)		
C10—C11—C12	121·2 (4)	C11—C12—C13	119·5 (4)		
N—C13—C4	117·8 (4)	N—C13—C12	120·1 (4)		
C4—C13—C12	121·9 (4)	O1—C14—C15	108·1 (5)		
O3—C16—C17	109·3 (7)	N—C18—O4	122·9 (5)		
N—C18—O5	112·3 (4)	O4—C18—O5	124·8 (5)		
O5—C19—C20	109·0 (6)				

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0·59470 Å^{-1} in h 0/18, k 0/12 and l −14/14, θ–2θ scans, θ-scan width (1·40 + 0·35 tanθ)°, three standard reflections monitored every 100 reflections showed no significant variation in intensity. 2572 unique reflections measured, 2108 intensities observed [$F_o > 3\sigma(F_o)$], no absorption correction. Structure solved by *MULTAN* (Germain, Main & Woolfson, 1971). H atoms located on a difference map. Positional parameters of all atoms and anisotropic thermal parameters for P, O, N and C atoms and isotropic thermal parameters for H atoms refined by block-diagonal least squares (Ashida, 1973). $\sum w|\Delta F|^2$ minimized with $w = 1·0$ for $0 < F_o < 56$ and $w = [1·0 + 0·167(F_o - 56)]^{-1}$ for $F_o > 56$. Final $R = 0·059$, $wR = 0·067$ and $S = 1·229$. Maximum positive and maximum negative electron densities in final difference Fourier synthesis are 0·53 and −0·47 e Å^{-3}. Δ/σ in the final cycle = 0·3 (z coordinate of an H atom). Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). All computations performed on a HITAC 280D at the Tottori University Computing Center and on a FACOM M780/30 at the Data Processing Center of Kyoto University. The final atomic coordinates and equivalent isotropic temperature factors are given in Table 1. Bond distances and angles are listed in Table 2.* A stereoview of the molecule with atomic numbering drawn by *DCM*-3 (Takenaka, 1977) is shown in Fig. 1.

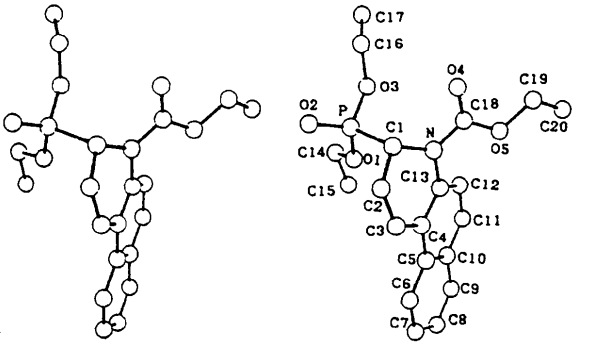

Fig. 1. Stereoview of the title molecule with the atomic numbering system. The absolute configuration of the molecule is arbitrary.

Related literature. The detailed synthetic method and spectral data of the title compound and related compounds are presented by Takeuchi *et al.* (1984).

* Lists of structure factors, anisotropic temperature factors of the non-H atoms, and isotropic temperature factors and positional parameters of H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52206 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of (±)-2-Hydroxymethyl-2,6-dimethylcyclohexan-1-ol

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Abstract. $C_9H_{18}O_2$, $M_r = 158·2$, monoclinic, $C2/c$, $a = 21·592$ (4), $b = 6·096$ (1), $c = 15·166$ (3) Å, $\beta = 115·00$ (1)°, $V = 1809·2$ (6) Å^3, $Z = 8$, $D_m = 1·15$ (1), $D_x = 1·16$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0·074$ mm^{-1}, $F(000) = 704$, $T = 298$ (2) K, $R = 0·044$ for 1274 unique observed reflections. The relative configuration of (±)-2-ethoxycarbonyl-2,6-dimethylcyclohexan-1-one having a 2-methyl substituent chemical shift of δ 1·28 was determined to be (2R*,6R*) by the structure determination of its derivative. The title molecules are linked into infinite chains along **c** by intermolecular hydrogen bonds between the hydroxy groups with O···O distances 2·773 (3) and 2·985 (5) Å.

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