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Structure of 1-Benzoyl-4,4,5,5-tetramethyl-2-phenylimidazoline Hydrate

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Abstract

The imidazoline ring is in an envelope conformation and the two phenyl rings form a dihedral angle of 49.3° with respect to each other. An intermolecular hydrogen bond is formed between the water and the unsubstituted N atom.

Comment

Imines can be oxidized directly to nitrones by means of aqueous potassium permanganate at pH 4.1 (Christensen & Jorgensen, 1989*a*,*b*). We attempted to apply this methodology to the oxidation of *N*,*N'*-dibenzylidine-2,3-dimethyl-2,3-butanediamine and instead of obtaining the bis nitrone, the title compound was produced unexpectedly.

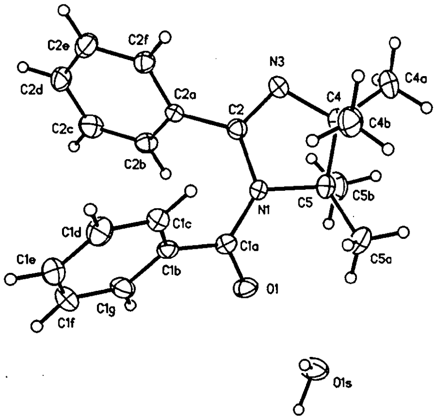

Fig. 1. Structure of the title compound showing 20% probability displacement ellipsoids.

Experimental

Crystal data

$C_{20}H_{22}N_2O.H_2O$
$M_r = 324.4$
Orthorhombic
$P2_12_12_1$
$a = 9.755$ (4) Å
$b = 10.800$ (4) Å
$c = 17.797$ (7) Å
$V = 1875.0$ (12) Å^3
$Z = 4$
$D_x = 1.149$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 11.53$-$17.0°$
$\mu = 0.074$ mm^{-1}
$T = 293$ (2) K
Irregular
$0.52 \times 0.48 \times 0.22$ mm
Colorless, translucent

Data collection

Siemens *R*3*m*/*V* four-circle diffractometer
Profile-fitted $\theta/2\theta$ scans
Absorption correction: none
1492 measured reflections
1438 independent reflections
1220 observed reflections [$I > 2\sigma(I)$]

$R_{\rm int} = 0.0161$
$\theta_{\rm max} = 22.55°$
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 11$
$l = 0 \rightarrow 19$
3 standard reflections monitored every 97 reflections intensity variation: ±2.0%

Refinement

Refinement on F^2
Final $R(F) = 0.0440$
$wR(F^2) = 0.1148$
$S = 1.111$
1436 reflections
217 parameters
H atoms: riding
Calculated weights
$w = 1/[\sigma^2(F_o^2) + (0.0737P)^2 + 0.1920P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = 0.052$
$\Delta\rho_{\rm max} = 0.115$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.211$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Refinement on F^2 for all reflections except for two with very negative F^2 or those flagged by the user for potential systematic errors. Weighted *R* factors $wR(F^2)$ and all values of the goodness-of-fit *S* are based on F^2, conventional *R* factors $R(F)$ are based on *F*, with *F* set to zero for negative F^2. The observed criterion [$F^2 > 2\sigma(F^2)$] is used only for calculating observed *R 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*; *R* factors based on all data will be even larger.

Data collection: *SHELXTL-Plus* (Sheldrick, 1990). Cell refinement: *SHELXTL-Plus*. Data reduction: *SHELXTL-Plus*. Program(s) used to solve structure: *SHELXTL-Plus*. Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus*. Software used to prepare material for publication: *SHELXL93*.

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Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$$U_{\rm eq} = \tfrac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	$U_{\rm eq}$
N1	0.1695 (3)	0.0083 (3)	0.6461 (2)	0.047 (2)
C1*A*	0.1202 (4)	−0.0010 (4)	0.7184 (2)	0.049 (2)
O1	0.1975 (3)	−0.0251 (3)	0.7711 (2)	0.066 (2)
C1*B*	−0.0257 (4)	0.0297 (4)	0.7336 (2)	0.046 (2)
C1*C*	−0.0927 (4)	0.1225 (4)	0.6950 (2)	0.056 (3)
C1*D*	−0.2247 (5)	0.1573 (5)	0.7158 (3)	0.070 (3)
C1*E*	−0.2882 (5)	0.0981 (6)	0.7740 (3)	0.078 (3)
C1*F*	−0.2235 (5)	0.0055 (6)	0.8122 (3)	0.077 (3)
C1*G*	−0.0925 (5)	−0.0274 (5)	0.7927 (2)	0.064 (3)
C2	0.1008 (4)	−0.0165 (4)	0.5786 (2)	0.047 (2)
C2*A*	−0.0250 (4)	−0.0916 (3)	0.5744 (2)	0.041 (2)
C2*B*	−0.0435 (5)	−0.1951 (4)	0.6194 (2)	0.052 (3)
C2*C*	−0.1611 (6)	−0.2655 (4)	0.6127 (2)	0.068 (4)
C2*D*	−0.2600 (5)	−0.2343 (5)	0.5612 (3)	0.068 (3)
C2*E*	−0.2423 (5)	−0.1334 (5)	0.5154 (3)	0.066 (3)
C2*F*	−0.1250 (4)	−0.0625 (4)	0.5220 (2)	0.054 (2)
N3	0.1645 (3)	0.0226 (3)	0.5200 (2)	0.052 (2)
C4	0.2872 (4)	0.0912 (4)	0.5453 (2)	0.060 (2)
C4*A*	0.4062 (5)	0.0685 (5)	0.4908 (3)	0.086 (3)
C4*B*	0.2488 (6)	0.2291 (5)	0.5437 (3)	0.082 (4)
C5	0.3142 (4)	0.0418 (4)	0.6268 (2)	0.057 (2)
C5*A*	0.3729 (5)	0.1389 (6)	0.6802 (3)	0.084 (3)
C5*B*	0.3994 (5)	−0.0771 (5)	0.6281 (3)	0.083 (3)
O1*S*	0.4341 (4)	−0.0760 (4)	0.8687 (2)	0.103 (2)

Table 2. *Geometric parameters* (Å, °)

N1—C1*A*	1.378 (5)	C2*A*—C2*B*	1.387 (5)
N1—C2	1.401 (5)	C2*A*—C2*F*	1.385 (5)
N1—C5	1.498 (5)	C2*B*—C2*C*	1.382 (6)
C1*A*—O1	1.230 (5)	C2*C*—C2*D*	1.372 (6)
C1*A*—C1*B*	1.486 (6)	C2*D*—C2*E*	1.373 (6)
C1*B*—C1*C*	1.380 (6)	C2*E*—C2*F*	1.382 (6)
C1*B*—C1*G*	1.383 (6)	N3—C4	1.478 (5)
C1*C*—C1*D*	1.391 (6)	C4—C4*A*	1.531 (6)
C1*D*—C1*E*	1.366 (7)	C4—C4*B*	1.537 (7)
C1*E*—C1*F*	1.364 (8)	C4—C5	1.569 (6)
C1*F*—C1*G*	1.371 (7)	C5—C5*A*	1.527 (7)
C2—N3	1.286 (5)	C5—C5*B*	1.530 (7)
C2—C2*A*	1.472 (5)		
C1*A*—N1—C2	128.3 (3)	C2*F*—C2*A*—C2	119.8 (3)
C1*A*—N1—C5	124.1 (3)	C2*C*—C2*B*—C2*A*	120.1 (4)
C2—N1—C5	107.5 (3)	C2*D*—C2*C*—C2*B*	120.4 (4)
O1—C1*A*—N1	120.9 (4)	C2*C*—C2*D*—C2*E*	120.2 (5)
O1—C1*A*—C1*B*	119.7 (4)	C2*D*—C2*E*—C2*F*	119.5 (4)
N1—C1*A*—C1*B*	119.2 (3)	C2*E*—C2*F*—C2*A*	121.0 (4)
C1*C*—C1*B*—C1*G*	118.6 (4)	C2—N3—C4	108.0 (3)
C1*C*—C1*B*—C1*A*	121.7 (4)	N3—C4—C4*A*	109.9 (4)
C1*G*—C1*B*—C1*A*	119.3 (4)	N3—C4—C4*B*	106.5 (4)
C1*B*—C1*C*—C1*D*	120.2 (4)	C4*A*—C4—C4*B*	109.2 (4)
C1*E*—C1*D*—C1*C*	119.7 (5)	N3—C4—C5	104.3 (3)
C1*F*—C1*E*—C1*D*	120.8 (5)	C4*A*—C4—C5	113.8 (4)
C1*E*—C1*F*—C1*G*	119.7 (5)	C4*B*—C4—C5	112.8 (4)
C1*F*—C1*G*—C1*B*	121.1 (5)	N1—C5—C5*A*	112.1 (4)
N3—C2—N1	113.7 (3)	N1—C5—C5*B*	107.8 (4)
N3—C2—C2*A*	122.8 (3)	C5*A*—C5—C5*B*	111.3 (4)
N1—C2—C2*A*	123.2 (3)	N1—C5—C4	97.8 (3)
C2*B*—C2*A*—C2*F*	118.6 (4)	C5*A*—C5—C4	113.9 (4)
C2*B*—C2*A*—C2	121.5 (4)	C5*B*—C5—C4	113.0 (4)

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71185 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HH1061]

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Structures of the Bis(diarylthiophosphoryl) Disulfides $[Ph_2P(S)]_2S_2$ and $[(PhO)_2P(S)]_2S_2$ and the Question of P—S π Bonding

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Abstract

The structures of bis(diphenylthiophosphoryl) disulfide, $[Ph_2P(S)]_2S_2$, and bis(diphenoxythiophosphoryl) disulfide, $[(PhO)_2P(S)]_2S_2$, are reported. The torsion angles about the disulfide bonds are similar; however, the geometry with respect to the P—S single bonds is different. The S—P—S—S moiety in the phenyl compound tends to *cis* planar, and the phenoxy analog to *trans* planar. The *cisoid* geometry is associated with larger S—P—S valence angles. The drive to planarity is interpreted as a π contribution to the P—S single bond. Two of the aromatic rings in the phenyl compound are offset cofacial with a separation of 3.89 (1) Å.

Comment

It has been reported (Buranda, Gallacher & Pinkerton, 1991; Gallacher & Pinkerton, 1992; Lawton, 1970; Tkachev, Atovmyan & Shchepinov, 1976; Yadav, Bohra, Mehrotra, Rai & Srivastava, 1983) that substitution of the organic moieties in organic disulfides by thiophosphoryl groups gives rise to a wide variety of S—S torsion angles. The values range from 93.6 to 180.0° compared to values close to 90° for the simple organic disulfides. The S—P—S—S moiety may have either a *cisoid* geometry (S—P—S—S torsion angle tending towards 0°) or a *transoid* geometry (S—P—S—S torsion angle tending towards 180°). Indeed, in the cyclohexyl analogs of the present compounds both geometries are observed in the same molecule. Thus, there is a tendency to planarity for the PS_3 unit in both the *cisoid* and *transoid* forms. The