Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{int} = 0.008$
θ–2θ scans [width (0.60 + 0.35tanθ)°]	$\theta_{max} = 24.9°$
Absorption correction: ψ scans	$h = 0 \rightarrow 22$
$T_{min} = 0.908$, $T_{max} = 0.998$	$k = 0 \rightarrow 12$
1587 measured reflections	$l = 0 \rightarrow 10$
1474 independent reflections	3 standard reflections
875 observed reflections [$I > 2.0\sigma(I)$]	frequency: 120 min
	intensity variation: < 2%

Refinement

Refinement on F	$(\Delta/\sigma)_{max} = 0.135$
$R = 0.037$	$\Delta\rho_{max} = 0.350$ e Å^{-3}
$wR = 0.028$	$\Delta\rho_{min} = -0.520$ e Å^{-3}
$S = 2.28$	Extinction correction: none
875 reflections	Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)
137 parameters	
Weighting scheme from counting statistics	

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* (2)

$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
Br	0.43730 (4)	0.20775 (8)	0.07844 (8)	3.43 (4)
N(1)	0.4331 (3)	0.1087 (5)	−0.2289 (6)	2.6 (3)
N(2)	0.5418 (3)	0.1553 (6)	−0.1225 (6)	2.8 (3)
C(1)	0.4763 (4)	0.1506 (7)	−0.1111 (7)	2.5 (4)
C(2)	0.3576 (4)	0.1137 (7)	−0.2185 (8)	2.2 (3)
C(3)	0.3184 (4)	0.2056 (8)	−0.2935 (8)	2.7 (4)
C(4)	0.2466 (4)	0.2061 (9)	−0.2715 (9)	3.2 (4)
C(5)	0.2125 (4)	0.1172 (8)	−0.1817 (8)	2.4 (4)
C(6)	0.2522 (5)	0.0235 (9)	−0.1085 (9)	3.1 (4)
C(7)	0.3250 (4)	0.0222 (9)	−0.1280 (9)	3.2 (4)
C(8)	0.1031 (6)	0.031 (1)	−0.092 (2)	4.7 (6)
O	0.1409 (3)	0.1279 (5)	−0.1723 (6)	3.8 (3)

Table 4. *Selected geometric parameters* (Å, °) *for* (2)

N(1a) at (0.5669, 0.1087, −0.2711) and N(2a) at (0.4582, 0.1553, −0.3775).

Br—C(1)	1.880 (7)	N(2)—C(1)	1.247 (9)
N(1)—N(2a)	1.443 (8)	C(5)—O	1.366 (9)
N(1)—C(1)	1.370 (9)	C(8)—O	1.41 (1)
N(1)—C(2)	1.436 (9)		
N(2a)—N(1)—C(1)	110.3 (5)	N(1)—C(1)—N(2)	123.4 (6)
N(2a)—N(1)—C(2)	111.9 (5)	N(1)—C(2)—C(3)	122.6 (6)
C(1)—N(1)—C(2)	122.7 (5)	N(1)—C(2)—C(7)	117.4 (7)
N(1a)—N(2)—C(1)	112.7 (5)	C(4)—C(5)—O	116.9 (7)
Br—C(1)—N(1)	120.0 (5)	C(6)—C(5)—O	125.0 (7)
Br—C(1)—N(2)	116.6 (5)	C(5)—O—C(8)	118.4 (7)

The structure was solved by the heavy-atom method. The H atoms of the phenyl groups were calculated after isotropic refinement; the others were found in difference Fourier maps and refined. The molecules of compound (2) possess a crystallographic twofold axis. *NRCC SDP VAX* (Gabe & Lee, 1981), *ORTEP* (Johnson, 1965) and the Enraf–Nonius (1979) *Structure Determination Package* were used in this work.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AL1011). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Enraf–Nonius (1979). *Structure Determination Package*. Enraf–Nonius, Delft, The Netherlands.
Gabe, E. J. & Lee, F. L. (1981). *Acta Cryst.* A**37**, S-339.
Johnson, C. K. (1965). *ORTEP* Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
Ohta, M. & Kato, H. (1969). *Non-Benzenoid Aromatics*, Vol. 1. ch. 4. New York: Academic Press.
Stewart, F. H. C. (1964). *Chem. Rev.* **64**, 129–147.
Ueng, C.-H., Lee, P. L., Wang, Y. & Yeh, M.-Y. (1985). *Acta Cryst.* C**41**, 1776–1779.
Ueng, C.-H., Wang, Y. & Yeh, M.-Y. (1987). *J. Chin. Chem. Soc. Taipei*, **34**, 105–110.

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Diethyl 2-(2,3-Diphenylquinoxalin-6-ylaminomethylene)malonate

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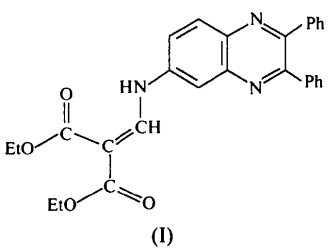
Abstract

The substituted aminoethylenic group in the title compound, $C_{28}H_{25}N_3O_4$, is approximately coplanar with the heterocyclic plane [dihedral angle 16 (2)°] and the α-C atom C(23), is disordered between two well defined sites. This disorder originates from a flipping of the aminoethylenic moiety around the

C(6)—N(5) bond. The phenyl rings at C(2) and C(3) are twisted out of the plane of the quinoxaline ring system by 46.7 (8) and 38.0 (9)°, respectively.

Comment

The overall conformation of the title molecule (I) and the numbering scheme are shown in Fig. 1. The dihedral angles that the phenyl groups make with the planar quinoxaline moiety in the title compound [46.7 (8) and 38.0 (9)°] agree with those observed previously for 2,3-diphenylquinoxaline in a benzene solution (*ca* 39°; Hurley & Le Fevre, 1967). A greater difference in these angles [22.1 (1) and 48.1 (1)°] has been reported for the crystal structure of 6,7-dimethyl-2,3-diphenylquinoxaline (Woźniak, Krygowski & Filipek, 1991).



(I)

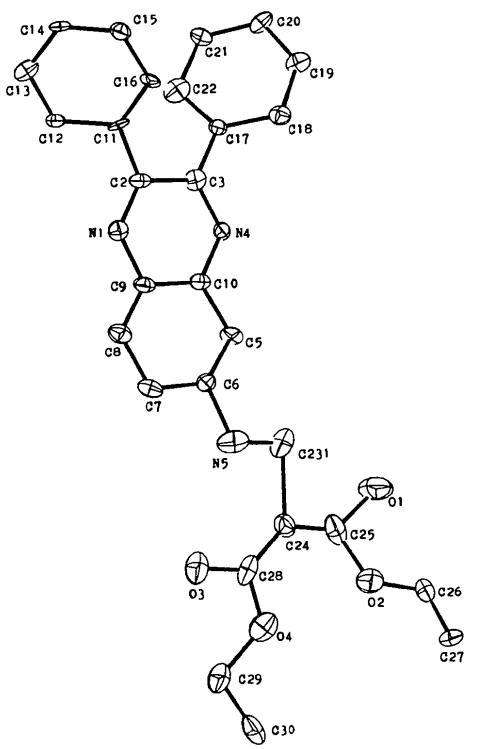


Fig. 1. Perspective drawing of the title compound and atom-numbering scheme. Displacement ellipsoids are drawn at the 40% probability level. Only one of the two disordered sites for C(23) is shown.

As a consequence of the disorder of the methylenic atom C(23), the N(5)—C(231) and N(5)—C(232) bond lengths are very short, while the C(231)—C(24) and C(232)—C(24) distances are unusually long (Table 2). Other bond distances and angles in those parts of the molecule not affected by the disorder are quite normal.

The substituted aminoethylenic moiety is approximately planar with the relevant torsion angles around the N(5)—C(23) bond being C(6)—N(5)—C(231)—C(24) = −179.2 (6) and C(6)—N(5)—C(232)—C(24) = −179.3 (9)°. Although a fast rotation around the C(6)—NH bond in several related compounds has been reported to occur in solution (Goljer, Milata & Ilavský, 1989), the occurence of the two conformations of the aminoethylenic group has not yet been observed in the solid state.

Experimental

Crystal data

$C_{28}H_{25}N_3O_4$
$M_r = 467.5$
Monoclinic
$P2_1/c$
$a = 17.714$ (9) Å
$b = 7.571$ (5) Å
$c = 17.974$ (10) Å
$\beta = 103.85$ (6)°
$V = 2340$ (2) Å^3
$Z = 4$
$D_x = 1.327$ Mg m^{-3}
$D_m = 1.33$ (1) Mg m^{-3}
D_m measured by flotation in bromoform/cyclohexane

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 15 reflections
$\theta = 6$–18°
$\mu = 0.084$ mm^{-1}
$T = 293$ K
Prism
0.35 × 0.20 × 0.15 mm
Brownish yellow
Crystal source: crystallization from ethanol

Data collection

$P2_1$ diffractometer
$\theta/2\theta$ scans
Absorption correction: none
2946 measured reflections
2702 independent reflections
1311 observed reflections [$I \geq 2\sigma(I)$]
$R_{int} = 0.035$

$\theta_{max} = 25°$
$h = 0 \rightarrow 18$
$k = 0 \rightarrow 7$
$l = -18 \rightarrow 17$
2 standard reflections monitored every 100 reflections
intensity variation: ±4%

Refinement

Refinement on F
$R = 0.072$
$wR = 0.068$
$S = 1.23$
1311 reflections
325 parameters
H-atom parameters not refined

$w = 1/[\sigma^2(F_o) + (0.04F_o)^2]$
$(\Delta/\sigma)_{max} = 0.15$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

$B_{eq} = (1/3)\sum_i\sum_j B_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
N(1)	0.5465 (2)	0.1990 (6)	0.4560 (3)	4.70 (17)
C(2)	0.5855 (3)	0.2136 (8)	0.4033 (3)	3.95 (22)
C(3)	0.5574 (3)	0.3366 (8)	0.3384 (3)	4.90 (23)
N(4)	0.4891 (2)	0.4177 (7)	0.3340 (2)	3.94 (16)
C(5)	0.3764 (3)	0.4744 (9)	0.3788 (3)	4.56 (23)
C(6)	0.3412 (3)	0.4479 (8)	0.4340 (3)	3.83 (20)
C(7)	0.3724 (3)	0.3527 (9)	0.5025 (4)	5.93 (26)
C(8)	0.4440 (3)	0.2750 (9)	0.5086 (3)	5.40 (25)
C(9)	0.4791 (3)	0.2945 (9)	0.4543 (3)	4.64 (25)
C(10)	0.4482 (3)	0.3894 (9)	0.3873 (3)	4.31 (24)
C(11)	0.6481 (3)	0.0964 (8)	0.3995 (3)	3.89 (21)
C(12)	0.7036 (3)	0.0755 (9)	0.4720 (3)	4.00 (22)
C(13)	0.7651 (3)	−0.0379 (9)	0.4779 (4)	6.18 (26)
C(14)	0.7705 (3)	−0.1308 (10)	0.4149 (3)	5.99 (26)
C(15)	0.7157 (3)	−0.1190 (8)	0.3470 (3)	4.78 (24)
C(16)	0.6553 (3)	−0.0004 (9)	0.3405 (3)	4.49 (23)
C(17)	0.6007 (3)	0.3795 (8)	0.2849 (3)	3.67 (21)
C(18)	0.5662 (3)	0.4077 (9)	0.2045 (3)	4.76 (23)
C(19)	0.6116 (3)	0.4526 (9)	0.1565 (3)	5.51 (24)
C(20)	0.6909 (3)	0.4680 (9)	0.1786 (4)	5.99 (25)
C(21)	0.7247 (3)	0.4485 (9)	0.2575 (3)	5.38 (22)
C(22)	0.6787 (3)	0.4041 (9)	0.3127 (4)	5.79 (28)
C(231)	0.2255 (7)	0.5849 (14)	0.3747 (9)	7.53 (57)
C(232)	0.2207 (6)	0.5297 (19)	0.4633 (8)	7.39 (60)
N(5)	0.2643 (3)	0.5178 (8)	0.4330 (4)	8.39 (26)
C(24)	0.1457 (4)	0.6389 (11)	0.4134 (4)	7.66 (33)
C(25)	0.1073 (4)	0.7137 (11)	0.3543 (4)	8.39 (33)
O(1)	0.1248 (3)	0.7420 (12)	0.2906 (3)	16.79 (37)
O(2)	0.0289 (2)	0.7325 (7)	0.3454 (3)	8.64 (20)
C(26)	−0.0224 (4)	0.7977 (14)	0.2843 (4)	10.07 (37)
C(27)	−0.0975 (4)	0.7691 (14)	0.2762 (5)	11.47 (40)
C(28)	0.1297 (3)	0.6279 (10)	0.4857 (4)	8.42 (31)
O(3)	0.1618 (3)	0.5455 (8)	0.5431 (3)	11.04 (26)
O(4)	0.0668 (2)	0.7201 (7)	0.4961 (3)	9.32 (20)
C(29)	0.0449 (5)	0.7103 (13)	0.5744 (4)	11.30 (39)
C(30)	−0.0272 (5)	0.7981 (12)	0.5605 (4)	9.79 (38)

Table 2. *Selected geometric parameters* (Å, °)

N(1)—C(2)	1.304 (7)	C(7)—C(8)	1.380 (9)
C(2)—C(3)	1.482 (8)	C(8)—C(9)	1.285 (8)
C(3)—N(4)	1.342 (7)	C(9)—N(1)	1.390 (7)
N(4)—C(10)	1.348 (7)	C(9)—C(10)	1.397 (8)
C(10)—C(5)	1.400 (8)	C(2)—C(11)	1.434 (7)
C(5)—C(6)	1.308 (8)	C(3)—C(17)	1.404 (8)
C(6)—C(7)	1.419 (8)	C(6)—N(5)	1.457 (8)
N(5)—C(231)	1.219 (16)	C(231)—C(24)	1.767 (15)
N(5)—C(232)	1.051 (14)	C(232)—C(24)	1.638 (15)
C(2)—N(1)—C(9)	122.8 (5)	C(5)—C(10)—C(9)	119.2 (6)
N(1)—C(2)—C(3)	119.2 (5)	C(6)—C(5)—C(10)	115.7 (6)
N(1)—C(2)—C(11)	122.1 (5)	C(5)—C(6)—C(7)	125.4 (6)
C(3)—C(2)—C(11)	118.0 (5)	C(5)—C(6)—N(5)	123.3 (6)
C(2)—C(3)—N(4)	117.5 (5)	C(7)—C(6)—N(5)	111.3 (6)
C(2)—C(3)—C(17)	123.2 (5)	C(6)—C(7)—C(8)	116.4 (6)
N(4)—C(3)—C(17)	119.2 (5)	C(7)—C(8)—C(9)	119.6 (6)
C(3)—N(4)—C(10)	121.0 (5)	C(8)—C(9)—N(1)	119.6 (6)
N(4)—C(10)—C(5)	118.5 (6)	C(8)—C(9)—C(10)	123.6 (6)
N(4)—C(10)—C(9)	121.9 (6)	N(1)—C(9)—C(10)	116.3 (5)
C(6)—N(5)—C(231)	119.7 (8)	N(5)—C(231)—C(24)	95.6 (9)
C(6)—N(5)—C(232)	145.7 (9)	N(5)—C(232)—C(24)	111.1 (10)

There is a twofold disorder at the C(23) atom and occupancy factors were fixed at 0.50 for both sites, C(231) and C(232), based on relative heights in a difference Fourier map. Positions of the H atoms, except those attached to the disordered C atom and its neighbouring N atom, were calculated and included in the F_c calculation with B_{iso} set at 0.5 Å^2 higher than the B_{eq} value of the parent C atom. The structure was solved using *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). All remaining calculations were performed with a local version of the *NRC* program system (Ahmed & Singh, 1973).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and least-squares-planes data have been deposited with the IUCr (Reference: KA1063). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Ahmed, F. R. & Singh, P. (1973). *J. Appl. Cryst.* **6**, 309–346, accession Nos. 133–147.

Goljer, I., Milata, V. & Ilavský, D. (1989). *Magn. Reson. Chem.* **27**, 138–144.

Hurley, J. & Le Fevre, R. J. W. (1967). *J. Chem. Soc. B*, pp. 824–827.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN*80. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Woźniak, K., Krygowski, T. M. & Filipek, S. (1991). *Acta Cryst.* C**47**, 1326–1328.

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4,4′-Methylenediiminobis(benzophenone)

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Abstract

The title compound [*N*,*N*′-bis(benzoylphenyl)methanediamine, $C_{27}H_{22}N_2O_2$] was prepared by condensation of 4-aminobenzophenone (ABP) and formaldehyde at room temperature. In the molecule, two ABP moieties are linked by a methylene group. The bond lengths and angles within the two ABP groups are very similar; however, the dihedral angles between the sets of ring planes are quite different [83.1 (5) and 57.5 (3)°].

Comment

Recently, crystals of 4-aminobenzophenone (ABP) were discovered to be a new and highly effective non-linear optical (NLO) material (Frazier & Cockerham, 1987). In an attempt to improve on this material, we carried out a condensation reaction