C4'	0.2640 (3)	0.1637 (8)	0.1031 (3)	0.0609 (9)
C13	0.1624 (3)	0.0354 (7)	0.3563 (3)	0.0498 (7)
C14	0.2347 (3)	−0.0878 (8)	0.4376 (3)	0.0627 (9)
C15	0.1617 (3)	0.6619 (7)	0.1388 (3)	0.0550 (8)
C16	0.0844 (4)	0.7310 (9)	0.0334 (4)	0.0874 (15)
C17	0.5048 (3)	0.4028 (8)	0.2816 (3)	0.0574 (8)
C18	0.5996 (3)	0.4797 (10)	0.2590 (4)	0.0775 (13)
C19	0.3693 (3)	0.0051 (9)	0.0249 (3)	0.0655 (10)
C20	0.4488 (5)	−0.1443 (12)	0.0426 (5)	0.105 (2)

Table 2. Selected geometric parameters (Å, °)

O1—N	1.290 (3)	C1—C9	1.462 (4)
O2—C6	1.361 (4)	C1—C1'	1.514 (4)
O2—C11	1.413 (5)	N—C3	1.474 (4)
O3—C7	1.369 (3)	C3—C4	1.508 (5)
O3—C12	1.403 (4)	C4—C10	1.507 (4)
O4—C13	1.353 (4)	C5—C10	1.386 (4)
O4—C1'	1.447 (4)	C5—C6	1.394 (4)
O5—C15	1.348 (5)	C6—C7	1.402 (4)
O5—C2'	1.434 (3)	C7—C8	1.377 (4)
O6—C17	1.358 (4)	C8—C9	1.414 (4)
O6—C3'	1.443 (4)	C9—C10	1.386 (4)
O7—C19	1.329 (5)	C1'—C2'	1.545 (4)
O7—C4'	1.441 (4)	C2'—C3'	1.527 (4)
O8—C13	1.198 (4)	C3'—C4'	1.505 (5)
O9—C15	1.190 (5)	C13—C14	1.466 (5)
O10—C17	1.185 (4)	C15—C16	1.484 (5)
O11—C19	1.208 (6)	C17—C18	1.497 (5)
C1—N	1.307 (4)	C19—C20	1.480 (7)
C6—O2—C11	118.1 (3)	C5—C10—C9	120.4 (3)
C7—O3—C12	117.5 (2)	C5—C10—C4	121.5 (3)
C13—O4—C1'	116.7 (2)	C9—C10—C4	118.1 (3)
C15—O5—C2'	117.8 (3)	O4—C1'—C1	109.8 (2)
C17—O6—C3'	115.7 (2)	O4—C1'—C2'	106.3 (2)
C19—O7—C4'	117.7 (4)	C1—C1'—C2'	114.4 (3)
N—C1—C9	119.7 (3)	O5—C2'—C3'	107.0 (2)
N—C1—C1'	116.7 (3)	O5—C2'—C1'	106.8 (2)
C9—C1—C1'	123.6 (2)	C3'—C2'—C1'	112.4 (3)
O1—N—C1	123.7 (3)	O6—C3'—C4'	107.2 (2)
O1—N—C3	115.6 (2)	O6—C3'—C2'	109.7 (3)
C1—N—C3	120.7 (3)	C4'—C3'—C2'	113.5 (3)
N—C3—C4	110.9 (3)	O7—C4'—C3'	108.1 (3)
C10—C4—C3	110.0 (2)	O8—C13—O4	121.6 (3)
C10—C5—C6	120.7 (3)	O8—C13—C14	127.0 (3)
O2—C6—C5	124.7 (3)	O4—C13—C14	111.4 (3)
O2—C6—C7	116.0 (3)	O9—C15—O5	123.2 (3)
C5—C6—C7	119.3 (3)	O9—C15—C16	126.4 (4)
O3—C7—C8	124.3 (3)	O5—C15—C16	110.4 (4)
O3—C7—C6	115.6 (3)	O10—C17—O6	122.5 (3)
C8—C7—C6	120.0 (2)	O10—C17—C18	126.1 (3)
C7—C8—C9	120.6 (3)	O6—C17—C18	111.4 (3)
C10—C9—C8	119.0 (2)	O11—C19—O7	123.6 (4)
C10—C9—C1	119.3 (2)	O11—C19—C20	125.7 (4)
C8—C9—C1	121.7 (2)	O7—C19—C20	110.7 (5)
C9—C1—N—O1	178.4 (3)	C6—C5—C10—C4	179.6 (3)
C1'—C1—N—O1	−3.7 (5)	C1—C9—C10—C5	176.8 (3)
C9—C1—N—C3	0.9 (5)	C8—C9—C10—C4	179.3 (3)
C1'—C1—N—C3	178.9 (3)	C1—C9—C10—C4	−2.3 (5)
O1—N—C3—C4	145.0 (3)	C3—C4—C10—C5	147.9 (3)
C1—N—C3—C4	−37.3 (5)	C3—C4—C10—C9	−33.0 (4)
N—C3—C4—C10	51.2 (4)	N—C1—C1'—O4	−145.3 (3)
C7—C8—C9—C1	−177.3 (3)	C9—C1—C1'—O4	32.5 (4)
N—C1—C9—C10	20.5 (5)	N—C1—C1'—C2'	95.3 (3)
C1'—C1—C9—C10	−157.3 (3)	C9—C1—C1'—C2'	−86.8 (3)
N—C1—C9—C8	−161.1 (3)	C1—C1'—C2'—O5	−79.4 (3)
C1'—C1—C9—C8	21.1 (5)	C1—C1'—C2'—C3'	163.6 (2)

Data collection, cell refinement and data reduction were performed using Kuma diffractometer software. The (0kl) reflections were used for the calculation of R_{int}. The structure was solved using SHELXS86 (Sheldrick, 1990) and refined using SHELXL93 (Sheldrick, 1994). The H atoms were positioned according to geometric criteria. The Flack parameter

[−0.1 (3)] and former experimental evidence indicated that the choice of absolute configuration was correct. Molecular graphics were prepared using ORTEPII (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1162). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Two Heterocyclic 1,2,4,5-Tetrazines

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Abstract

The title compounds, 6-bromo-1,4-dihydro-1,4-di(o-tolyl)-1,2,4,5-tetrazin-3(2H)-one, $C_{16}H_{15}BrN_4O$, and 3,6-dibromo-1,4-dihydro-1,4-bis(p-methoxyphenyl)-1,2,4,5-tetrazine, $C_{16}H_{14}Br_2N_4O_2$, were prepared from 4-bromo-3-(o-tolyl)sydnone and 4-bromo-3-(p-methoxyphenyl)sydnone, respectively, in thf under ultrasonic irradiation, and identified with IR, ^{1}H NMR, mass spectrum and elemental analyses. The heterocyclic rings of both title compounds appear to lack aromatic character, as judged from the bond lengths.

Comment

Sydnone is a typical *meso*-ionic compound with unique properties (Stewart, 1964; Ohta & Kato, 1969; Ueng, Lee, Wang & Yeh, 1985). Although the sydnone ring has some aromatic character, sydnone compounds sometimes decompose during reaction. In the present work, we tried to synthesize bis-sydnone from 4-halosydnones by an Üllmann reaction under ultrasonic irradiation. The sydnone ring decomposed to give tetrazine derivatives when the reaction was carried out in thf solution, but decomposition did not occur in *N,N*-dimethylformamide medium. Compound (1), 6-bromo-1,4-dihydro-1,4-di(*o*-tolyl)-1,2,4,5-tetrazin-3(2*H*)-one, was prepared from 4-bromo-3-(*o*-tolyl)sydnone, and compound (2), 3,6-dibromo-1,4-di(*p*-methoxyphenyl)-1,4-dihydro-1,2,4,5-tetrazine, from 4-bromo-3-(*p*-methoxyphenyl)sydnone. In order to confirm the structure and study the bond character of the title compounds, the X-ray single-crystal structure determinations were undertaken.

(I)

(II)
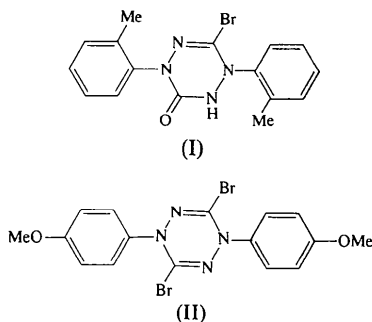
The molecular structures are shown in Fig. 1. The C(1)—O(1) length [1.224 (5) Å] in the heterocycle ring of compound (1) is within the typical C=O double-bond range (1.23 ± 1 Å). The N(2)—C(2) length [1.257 (5) Å], which is close to the N=C double-bond value (Ueng, Wang & Yeh, 1987), is shorter than the other three N—C bond lengths [N(4)—C(1) 1.360 (5), N(1)—C(1) 1.375 (5), N(3)—C(2) 1.378 (5) Å]. The N(1)—N(2) and N(3)—N(4) bond lengths [1.426 (4) and 1.418 (5) Å, respectively] have single-bond character (N—N single bond 1.44 ± 4 Å).

Planarity can be quantified by χ^2 which is defined as $\Sigma \Delta d_i^2/(\sigma_x^2 + \sigma_y^2 + \sigma_z^2)$, where Δd_i is the distance of the atom from the least-squares plane and σ_x, σ_y and σ_z are the standard deviations of the coordinates x, y and z, respectively, for each atom. The planarity coefficient of the two phenyl groups of compound (1) are 3.9 [C(3)–C(8)] and 0.5 [C(10)–C(15)]. Hence the coeffiencient for the heterocyclic ring, $\chi^2 = 9095.5$ [Δd: N(1) 0.239 (4); N(3) 0.214 (4); C(2) −0.083 (5); C(1) −0.103 (5); N(2) −0.168 (4); N(4) −0.176 (5) Å] indicates significant departures from planarity. The distances of Br, O(1), C(3) and C(10)
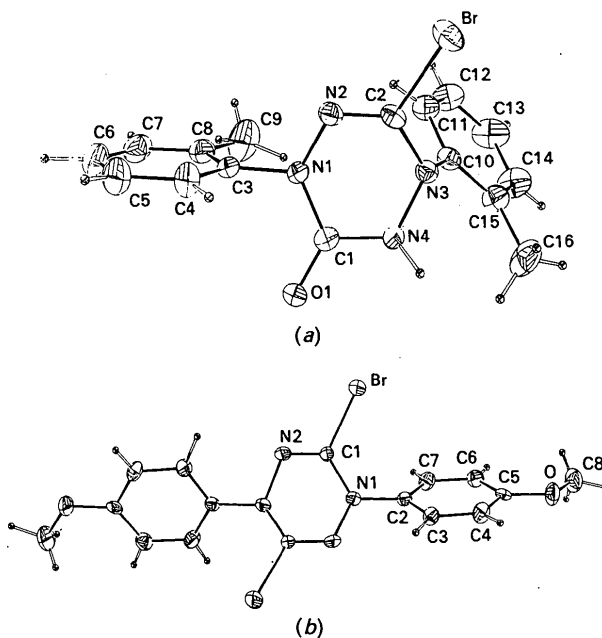

Fig. 1. Molecular structures of (*a*) compound (1) and (*b*) compound (2).

from the least-squares plane are −0.545 (6), −0.314 (6), 0.3077 (7) and 1.500 (6) Å, respectively, *i.e.* the two phenyl groups are on the same side of the least-squares plane, and Br and O(1) are on the other side. With the dihedral angle between N(1)—N(2)—C(2)—Br ($\chi^2 = 23.8$) and N(1)—C(1)[=O(1)]—N(4) ($\chi^2 = 0.4$) being 33.7 (3)°, this shows that the interaction between the carbonyl group and the C(2)=N(2) double bond is negligible. From the bond-length and planarity data, the bonds of the six-membered ring may be described as localized C(2)=N(2) and C(1)= O(1) double bonds.

The N(2)—C(1) length [1.247 (9) Å] of compound (2) is also shorter than the N(1)—C(1) length [1.370 (9) Å]. The N(1)—N(2) length [1.443 (8) Å], which is slightly longer than the corresponding bond in compound (1), shows typical single-bond character. The ring is again not very planar, $\chi^2 = 3477.7$, with Br and C(2) 0.464 (7) and −0.04 (1) Å from the least-squares plane, respectively. Therefore, the heterocyclic ring in compound (2), as in compound (1), lacks aromatic character and the bonds may be described as localized C(1)=N(2) double and N(1)—N(2) single bonds.

There is little π interaction between the phenyl ring and the heterocyclic ring owing to the large dihedral angle between them in compounds (1) and (2) [88.6 (2) and 71.3 (2)° in (1); 62.0 (3)° in (2)]. The bond lengths and angles of the tolyl and methoxyphenyl groups are normal. Both the title compounds have no intermolecular contacts of structural significance.

Experimental

Compound (1) was prepared by adding 4-bromo-3-(*o*-tolyl)sydnone (0.64 g, 2.5 mmol) to tetrahydrofuran (10 cm³), oscillating the solution and irradiating with ultrasonic radiation (20 kHz, 225 W) for 4 h in a water bath at room temperature. After reducing to about 5 cm³, the solution was allowed to stand for 1 d, yielding colourless crystals, which were filtered and washed with 95% alcohol, and dried; m.p. 449–450 K, yield 0.20 g (44%). Analysis: found, C 53.30, H 4.28, N 15.58, Br 22.17%; calculated for $C_{16}H_{15}BrN_4O$, C 53.47, H 4.21, N 15.63, Br 22.24%. IR (KBr) ν_{max}: 3190, *m* (N—H), 1683, *s* (C=O) and 1590 cm⁻¹, *m* (C=N). ¹H NMR δ_H(DMSO-d₆): 9.84 (1*H*, *s*, N—H); 7.45–7.25 (8*H*, *m*, 2C₆H₄); 2.42 (3*H*, *s*, Ar—CH₃) and 2.20 (3*H*, *s*, Ar—CH₃). Mass *m/z* (%): 360 (*M*⁺ + 2, 69), 358 (*M*⁺, 71), 198 (13), 196 (13), 133 (26) and 91 (100). The compound was recrystallized from dichloromethane. The density D_m was measured by flotation in CHCl₃/CCl₄.

Compound (2) was prepared in a similar way to compound (1), except that the solvent was purified before use. 4-Bromo-3-(*p*-methoxyphenyl)sydnone (0.68 g, 2.5 mmol) yielded colourless crystals; m.p. 495–496 K, yield 0.14 g (25%). Analysis: found, C 42.25, H 3.16, N 12.25, Br 35.40%; calculated for $C_{16}H_{14}Br_2N_4O_2$, C 42.32, H 3.11, N 12.34, Br 35.19%. IR (KBr) ν_{max}: 1590, 1578 cm⁻¹, *m* (C=N). ¹H NMR δ_H(DMSO-d₆): 7.33 (4*H*, *d*, *J* = 90 Hz, Ar-2′,6′,2″,6″-H); 7.00 (4*H*, *d*, *J* = 90 Hz, Ar-3′,5′,3″,5″-H) and 3.79 (6*H*, *s*, 2 OCH₃). Mass *m/z* (%): 456 (*M*⁺ + 4, 39), 454 (*M*⁺ + 2, 100), 452 (*M*⁺, 45), 214 (25), 212 (24), 147 (87), 133 (48) and 107 (54). The compound was recrystallized from methanol. The density D_m was measured by flotation in CHCl₃/CHBr₃.

Compound (1)

Crystal data

$C_{16}H_{15}BrN_4O$
$M_r = 359.2$
Triclinic
$P\bar{1}$
$a = 8.217\ (3)$ Å
$b = 8.477\ (5)$ Å
$c = 12.045\ (3)$ Å
$\alpha = 99.16\ (3)°$
$\beta = 104.23\ (2)°$
$\gamma = 95.41\ (5)°$
$V = 795.0$ Å³
$Z = 2$
$D_x = 1.50$ Mg m⁻³
$D_m = 1.51\ (3)$ Mg m⁻³

Mo *Kα* radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 8.515–12.29°$
$\mu = 2.56$ mm⁻¹
$T = 298$ K
$0.70 \times 0.45 \times 0.30$ mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
θ–2θ scans [width (0.70 + 0.35tanθ)°]
Absorption correction:
ψ scans
$T_{min} = 0.900$, $T_{max} = 0.998$
3004 measured reflections
2791 independent reflections
1937 observed reflections
[$I > 1.5\sigma(I)$]

$R_{int} = 0.010$
$\theta_{max} = 24.9°$
$h = -9 \rightarrow 9$
$k = 0 \rightarrow 10$
$l = -14 \rightarrow 14$
3 standard reflections
frequency: 120 min
intensity variation: <3%

Refinement

Refinement on *F*
$R = 0.044$
$wR = 0.026$
$S = 1.77$
1937 reflections
245 parameters
Weighting scheme from counting statistics
$(\Delta/\sigma)_{max} = 0.737$

$\Delta\rho_{max} = 0.330$ e Å⁻³
$\Delta\rho_{min} = -0.290$ e Å⁻³
Extinction correction: secondary
Extinction coefficient: $1.8\ (1) \times 10^{-6}$
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å²) *for* (1)

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

	x	*y*	*z*	B_{eq}
Br	0.18917 (7)	0.01393 (6)	0.23722 (5)	4.70 (3)
O(1)	0.0972 (4)	0.6424 (3)	0.1371 (2)	3.5 (2)
N(1)	0.2051 (4)	0.4940 (4)	0.2739 (3)	3.0 (2)
N(2)	0.1828 (4)	0.3345 (4)	0.2966 (3)	3.2 (2)
N(3)	0.2353 (4)	0.2581 (4)	0.1131 (3)	3.0 (2)
N(4)	0.1579 (5)	0.3917 (4)	0.0789 (3)	3.2 (2)
C(1)	0.1492 (5)	0.5182 (5)	0.1613 (3)	3.0 (2)
C(2)	0.2081 (5)	0.2314 (4)	0.2174 (4)	2.9 (2)
C(3)	0.1970 (6)	0.6175 (5)	0.3677 (4)	3.4 (2)
C(4)	0.0450 (7)	0.6365 (6)	0.3923 (4)	5.0 (3)
C(5)	0.0403 (9)	0.7596 (6)	0.4822 (6)	6.5 (4)
C(6)	0.185 (1)	0.8575 (7)	0.5455 (5)	6.2 (4)
C(7)	0.3319 (9)	0.8372 (6)	0.5217 (5)	5.5 (4)
C(8)	0.3433 (7)	0.7158 (5)	0.4330 (4)	4.1 (3)
C(9)	0.5082 (9)	0.692 (1)	0.4036 (6)	7.8 (4)
C(10)	0.4027 (5)	0.2534 (5)	0.0973 (4)	3.1 (2)
C(11)	0.5395 (7)	0.2664 (6)	0.1923 (4)	4.2 (3)
C(12)	0.6992 (7)	0.2540 (7)	0.1766 (5)	5.4 (3)
C(13)	0.7199 (7)	0.2280 (8)	0.0663 (6)	6.5 (4)
C(14)	0.5828 (8)	0.2156 (7)	-0.0287 (5)	6.0 (4)
C(15)	0.4225 (6)	0.2271 (5)	-0.0145 (4)	4.1 (3)
C(16)	0.2761 (8)	0.2116 (8)	-0.1204 (5)	6.6 (4)

Table 2. *Selected geometric parameters* (Å, °) *for* (1)

Br—C(2)	1.893 (4)	N(2)—C(2)	1.257 (5)
O(1)—C(1)	1.224 (5)	N(3)—N(4)	1.418 (5)
N(1)—N(2)	1.426 (4)	N(3)—C(2)	1.378 (5)
N(1)—C(1)	1.375 (5)	N(3)—C(10)	1.438 (5)
N(1)—C(3)	1.432 (5)	N(4)—C(1)	1.360 (5)
N(2)—N(1)—C(1)	119.0 (3)	O(1)—C(1)—N(4)	122.8 (4)
N(2)—N(1)—C(3)	114.1 (3)	N(1)—C(1)—N(4)	113.9 (4)
C(1)—N(1)—C(3)	118.7 (3)	Br—C(2)—N(2)	117.0 (3)
N(1)—N(2)—C(2)	112.0 (3)	Br—C(2)—N(3)	116.1 (3)
N(4)—N(3)—C(2)	110.6 (3)	N(2)—C(2)—N(3)	126.7 (3)
N(4)—N(3)—C(10)	114.6 (3)	N(1)—C(3)—C(4)	120.1 (4)
C(2)—N(3)—C(10)	118.4 (3)	N(1)—C(3)—C(8)	119.3 (4)
N(3)—N(4)—C(1)	119.9 (3)	N(3)—C(10)—C(11)	120.1 (4)
O(1)—C(1)—N(1)	123.3 (4)	N(3)—C(10)—C(15)	119.1 (4)

Compound (2)

Crystal data

$C_{16}H_{14}Br_2N_4O_2$
$M_r = 454$
Orthorhombic
Pbcn
$a = 18.975\ (3)$ Å
$b = 10.327\ (2)$ Å
$c = 8.569\ (3)$ Å
$V = 1679.2$ Å³
$Z = 4$
$D_x = 1.80$ Mg m⁻³
$D_m = 1.79\ (3)$ Mg m⁻³

Mo *Kα* radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 10.57–12.83°$
$\mu = 4.80$ mm⁻¹
$T = 298$ K
$0.55 \times 0.30 \times 0.30$ mm
Colourless

Data collection

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

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
137 parameters	from International Tables
Weighting scheme from counting statistics	for X-ray Crystallography (1974, Vol. IV)

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

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

	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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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