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5-(*N*,*N*-Diethylamino)-4,6-diphenyl-1,2,3-triazine

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.035
wR factor = 0.087
Data-to-parameter ratio = 8.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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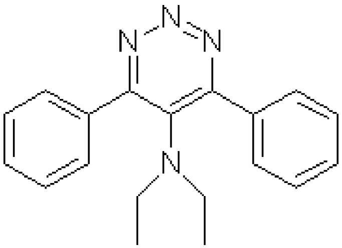
In the title compound, $C_{19}H_{20}N_4$, the dihedral angles formed by the planes of the triazine ring and the two phenyl substituents are 51.54 (1) and 49.27 (1)°. The phenyl rings form a dihedral angle of 80.81 (1)°. All bond lengths and angles are normal. The structure is stabilized by van der Waals interactions.

Comment

The derivatives of 1,2,3-triazine are an important class of heterocyclic compounds useful in organic synthesis and as insecticides, pharmaceuticals and dyestuffs. Their chemistry has been reviewed several times since 1956 (Erickson, 1956; Neunhoeffer & Wiley, 1978; Kobylecki & Mckillop, 1976). Their importance in organic synthesis is due to the fact that they can react as dienes on inverse-demand Diels–Alder cycloadditions with electron-rich dienophiles (Prieto *et al.*, 2002). The many applications of these compounds as pharmaceuticals and selective herbicides, which are usually applied as pre- and post-emergent weed control agents to improve the quality of agricultural products (Masquelin *et al.*, 1998), are a result of the wide range of biological activity associated with this interesting heteroaromatic ring system (Tsai *et al.*, 1998). The Cambridge Structural Database (CSD; Version 5.25 and updates; Allen, 2002) contains eight different derivatives of 1,2,3-triazine [refcodes KOTCIF (Yamaguchi *et al.*, 1992), PIKDAO (Eichhorn *et al.*, 1993), PINWEO (Bopp *et al.*, 1994), TMPTAZ (Oeser & Schiele, 1972), VOBNAB and VOBNAB01 (Yamaguchi, Itoh, Okada, Ohsawa & Matsumura, 1991), VOBPAD, VOBPEH and VOBPEH01 (Yamaguchi, Itoh, Okada & Ohsawa, 1991), and UHUJIQ (Bau *et al.*, 1998)]. Recently, we also published the structure of 5-(*N*,*N*-diethylamino)-4,6-tris(4-fluorophenyl)-1,2,3-triazine (Matsumoto *et al.*, 2002) and we report here the crystal structure of 5-(*N*,*N*-diethylamino)-4,6-diphenyl-1,2,3-triazine, (I).



(I)

A perspective view of the 4,6-diphenyl-5-(*N*,*N*-diethylamin)-1,2,3-triazine molecule is shown in Fig. 1. This molecule is built up from an essentially planar triazine ring, two

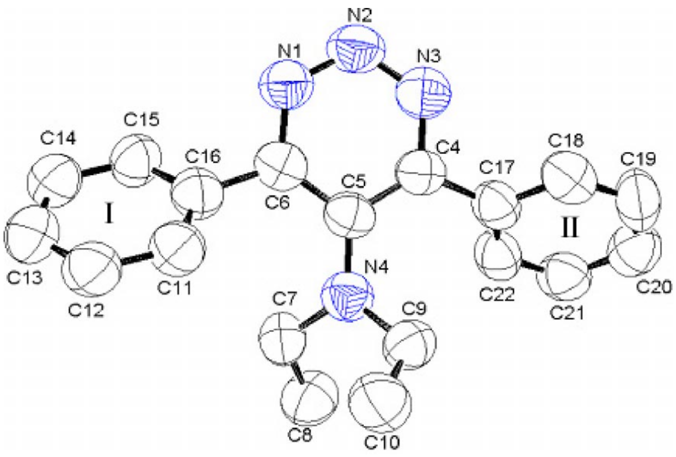


Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted.

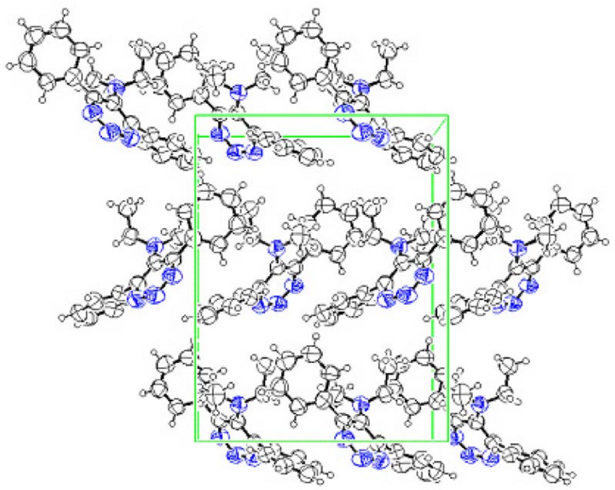


Figure 2
The crystal packing, viewed along the *b* axis.

attached phenyl rings (I and II) and one diethylamino group, which are not coplanar with the triazine ring. The N atom of the diethylamino group lies 0.073 (2) Å above the plane formed by the triazine and the phenyl ring C atoms; C16 (ring I) and C17 (ring II) lie 0.094 (2) Å above and 0.186 (2) Å below this plane, respectively. The angle between the mean plane of phenyl ring I and the triazine mean plane is 51.54 (1)°, while the angle between the plane of ring II and the triazine plane is 49.27 (1)°. The angle between the two phenyl rings is 80.81 (1)°. The values of the bond lengths and valence angles lie in the usual ranges for similar structures included in the CSD.

Molecules pack in the cell, forming sheets parallel to the *ac* plane (Fig. 2). The crystal structure is stabilized by van der Waals interactions.

Experimental

Diphenylcyclopropenone, used as a starting material, was prepared according to the procedure of Breslow & Posner (1973). To a solution of diphenylcyclopropenone (0.606 g, 3 mmol) in dry dichloromethane (15 ml) under a nitrogen atmosphere was added triethyloxonium tetrafluoroborate (0.624 g, 3.3 mmol). The resulting solution was stirred for 1 h at room temperature. A solution of diethylamine (0.22 g, 3 mmol) in dichloromethane (10 ml) was then added *via* syringe. The reaction mixture was kept at room temperature for another 1 h, diethyl ether (70 ml) was added and the mixture was concentrated to 20 ml. The precipitated white solid was collected on a funnel and dried, giving 1-(*N*,*N*-diethylamino)-2,3-diphenylcyclopropenium tetrafluoroborate (0.78 g, 72%). To a suspension of this salt (0.69 g, 2 mmol) in dichloromethane (30 ml) was added sodium azide (0.39 g, 6 mmol), and the mixture was further stirred at room temperature for 24 h. The solvent was then evaporated, leaving a yellow solid which was shaken with benzene; a white solid which remained after shaking was filtered off and the benzene solution was evaporated again, leaving the crude product. Crystallization from chloroform/ethanol (1:1) afforded yellow crystals (0.4 g, 65%). Analysis calculated for $C_{19}H_{20}N_4$: C 74.97, H 6.62, N 18.41%; found: C 74.92, H 6.64, N 18.19%. M.p. 481–482 K. IR (KBr disk): 2971, 2842, 2776, 2467, 2036, 1468, 1440, 1053; ^{1}H NMR ($CDCl_3$, 200 MHz): δ 0.93 (*t*, *J* = 7 Hz, 6H), 2.79 (*q*, *J* = 7 Hz, 4H), 7.47–7.51 (*m*, 6H), 7.67–7.72 (*m*, 4H); ^{13}C NMR ($CDCl_3$, 54.6z): δ 12.7, 46.0, 128.2, 128.7, 129.5, 136.7, 138.0, 152.6; MS (70 eV) *m*/*z*: 304 (100) (*M*+), 276 (43), 173 (59), 158 (5), 104 (5).

Crystal data

$C_{19}H_{20}N_4$
$M_r = 304.39$
Orthorhombic, $Pca2_1$
$a = 12.535$ (3) Å
$b = 7.856$ (2) Å
$c = 16.253$ (3) Å
$V = 1600.5$ (6) Å^3
$Z = 4$
$D_x = 1.263$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 20 reflections
$\theta = 10$–16°
$\mu = 0.08$ mm^{-1}
$T = 293$ (2) K
Needle, clear pale yellow
0.50 × 0.06 × 0.02 mm

Data collection

Burevestnik DARCH-1 diffractometer
ω–2θ scans
Absorption correction: part of the refinement model (ΔF) (*DIFABS*; Walker & Stuart, 1983)
$T_{min} = 0.957$, $T_{max} = 0.998$
3080 measured reflections
1894 independent reflections

1563 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$
$\theta_{max} = 27.4°$
$h = 0 \rightarrow 16$
$k = 0 \rightarrow 10$
$l = -10 \rightarrow 20$
3 standard reflections every 100 reflections
intensity decay: 2.3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.088$
$S = 1.06$
1894 reflections
213 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0374P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.006$
$\Delta\rho_{max} = 0.11$ e Å^{-3}
$\Delta\rho_{min} = -0.14$ e Å^{-3}

H atoms were treated as riding (C—H = 0.93–0.97 Å). The two free variables for U_{iso}(H) were refined according to *SHELXL97* (Sheldrick, 1997). In the absence of significant anomalous dispersion effects, Friedel pairs were merged.

Data collection: DARCH software; cell refinement: DARCH software; data reduction: DARCH software; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *PLATON* (Spek, 2000); software used to prepare material for publication: *SHELXL97*.

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