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Ethyl 3,6-diphenyl-1,4-dihydro-1,2,4,5-tetrazine-1-carboxylate

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Key indicators

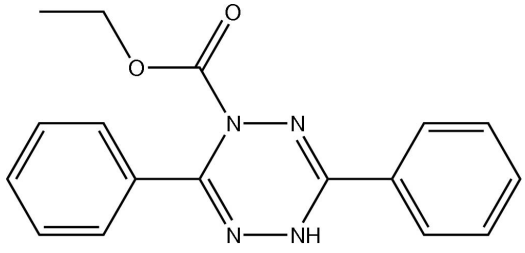
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.034
wR factor = 0.098
Data-to-parameter ratio = 7.0

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

The title compound, $C_{17}H_{16}N_4O_2$, was prepared from ethyl chloroformate and 3,6-diphenyldihydro-1,2,4,5-tetrazine. The central six-membered ring has an asymmetric boat conformation.

Comment

1,2,4,5–Tetrazine derivatives have a high potential for biological activity, possessing a wide range of antiviral and antitumor properties, and these derivatives have been widely used in pesticides and herbicides (Sauer, 1996). In a continuation of our work on the structure-activity relationship of 1,2,4,5-tetrazine derivatives (Hu *et al.*, 2002, 2004), we have obtained a yellow crystalline compound as the product of the reaction of ethyl chloroformate and 3,6-diphenyldihydro-1,2,4,5-tetrazine. The structural identity of our product, (I), was resolved using single-crystal X-ray diffraction.



(I)

The molecular structure of (I) is illustrated in Fig. 1. In (I), atoms N2, C3, N5 and C6 are coplanar within 0.035 Å, and atoms N1 and N4 deviate from the plane by 0.454 (2) and

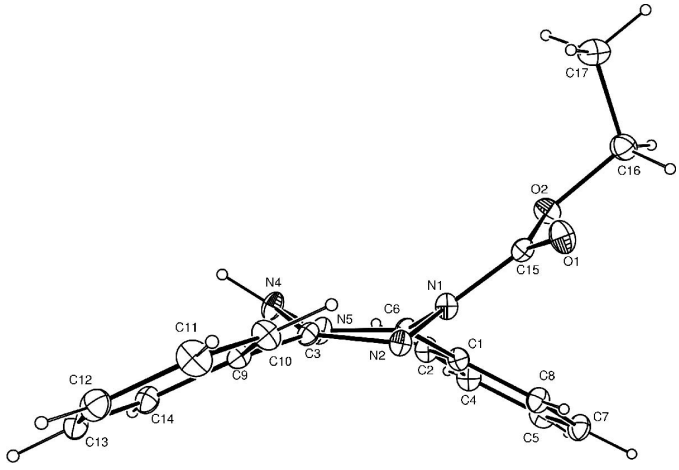


Figure 1
The structure of (I), shown with 30% probability displacement ellipsoids.

0.354 (2) Å, respectively, indicating an asymmetric boat conformation.

Experimental

The title compound was prepared according to the procedure of Rao & Hu (2004). A solution of the compound in ethanol was concentrated gradually at room temperature to afford yellow prisms (m.p. 411–413 K).

Crystal data

$C_{17}H_{16}N_4O_2$
$M_r = 308.34$
Orthorhombic, $Pna2_1$
$a = 10.175$ (2) Å
$b = 9.219$ (2) Å
$c = 16.814$ (4) Å
$V = 1577.2$ (6) Å^3
$Z = 4$
$D_x = 1.298$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 11.5$–12.5°
$\mu = 0.09$ mm^{-1}
$T = 293$ (2) K
Prism, yellow
0.35 × 0.30 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.970$, $T_{max} = 0.983$
1633 measured reflections
1468 independent reflections
1159 reflections with $I > 2\sigma(I)$
$R_{int} = 0.046$
$\theta_{max} = 25.2^\circ$
$h = -1 \rightarrow 12$
$k = 0 \rightarrow 11$
$l = -20 \rightarrow 0$
3 standard reflections
frequency: 60 min
intensity decay: 0.3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.098$
$S = 1.04$
1468 reflections
210 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0519P)^2 + 0.3521P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0283 (14)

H atoms were included in calculated positions and refined using a riding model. H atoms were given isotropic displacement parameters equal to 1.2 (or 1.5 for methyl H atoms) times U_{eq} of their parent atoms and C—H distances were set to 0.96 Å for those bonded to methyl and 0.97 Å for the remainder, while N—H distances were set to 0.86 Å. In the absence of significant anomalous dispersion effects, Friedel pairs were averaged.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Version 1.05; Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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