

3,6-Bis(4-chlorophenyl)-1-isobutyryl-1,4-dihydro-1,2,4,5-tetrazine

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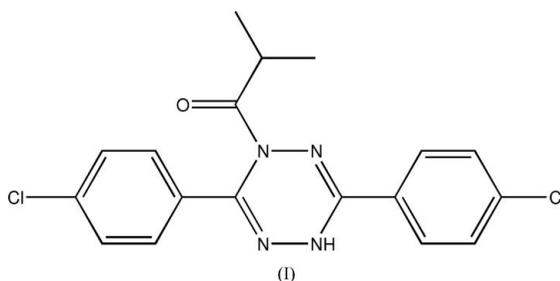
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Key indicators

Single-crystal X-ray study
 $T = 298$ K
Mean $\sigma(\text{C}-\text{C}) = 0.009$ Å
 R factor = 0.040
 wR factor = 0.121
Data-to-parameter ratio = 8.8For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.The title compound, $\text{C}_{18}\text{H}_{16}\text{Cl}_2\text{N}_4\text{O}$, was prepared from isobutyric anhydride and 3,6-bis(4-chlorophenyl)-1,2-dihydro-1,2,4,5-tetrazine. The central six-membered ring has a boat conformation.

Comment

1,2,4,5-Tetrazine derivatives have a high potential for biological activity and possess a wide range of antiviral and antitumor properties. These derivatives have been widely used in the production of pesticides and herbicides (Sauer, 1996). As part of our continuing interest in the structure–activity relationship of 1,2,4,5-tetrazine derivatives (Hu *et al.*, 2002, 2004), we have isolated the product, (I), of the reaction of isobutyric anhydride and 3,6-bis(4-chlorophenyl)-1,2-dihydro-1,2,4,5-tetrazine, as yellow crystals suitable for X-ray analysis.The molecular structure of (I) is illustrated in Fig. 1. Atoms N2, C3, N5 and C6 are coplanar, with the largest deviation from the plane being -0.024 (3) Å for atom N5. Atoms N1 and N4 deviate from this plane by 0.413 (8) and 0.344 (8) Å, respectively, indicating a boat conformation.There is an $\text{N}-\text{H}\cdots\text{O}$ hydrogen interaction, building a zigzag chain parallel to the a axis (Table 1 and Fig. 2).

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. 472–474 K).

Crystal data

 $\text{C}_{18}\text{H}_{16}\text{Cl}_2\text{N}_4\text{O}$
 $M_r = 375.25$
Orthorhombic, $P2_12_12_1$
 $a = 7.268$ (4) Å
 $b = 11.813$ (1) Å
 $c = 21.282$ (3) Å
 $V = 1827.2$ (10) Å³
 $Z = 4$
 $D_x = 1.364$ Mg m⁻³Mo $K\alpha$ radiation
Cell parameters from 25 reflections
 $\theta = 10.1\text{--}10.8^\circ$
 $\mu = 0.37$ mm⁻¹
 $T = 298$ (2) K
Prism, yellow
0.25 × 0.25 × 0.20 mm

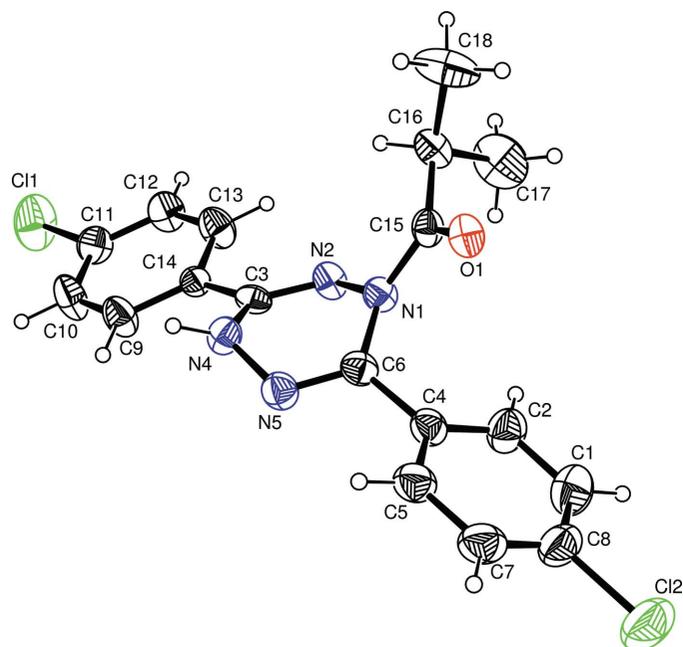


Figure 1
The structure of (I), showing the atom-labeling scheme. Ellipsoids are drawn at the 30% probability level.

Data collection

Enraf–Nonius CAD-4
diffractometer
 $\omega/2\theta$ scans
Absorption correction: multi-scan
(*ABSCOR*; Higashi, 1995)
 $T_{\min} = 0.912$, $T_{\max} = 0.930$
2019 measured reflections
1997 independent reflections
840 reflections with $I > 2\sigma(I)$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.121$
 $S = 0.99$
1997 reflections
228 parameters
H-atom parameters constrained

$R_{\text{int}} = 0.025$
 $\theta_{\text{max}} = 25.2^\circ$
 $h = 0 \rightarrow 8$
 $k = 0 \rightarrow 14$
 $l = -1 \rightarrow 25$
3 standard reflections
frequency: 60 min
intensity decay: 0.3%

$w = 1/[\sigma^2(F_o^2) + (0.0446P)^2 + 0.4072P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.20 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.21 \text{ e } \text{\AA}^{-3}$

Table 1

Hydrogen-bond geometry (\AA , $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N4-H4\cdots O1^i$	0.86	2.31	2.851 (6)	121

Symmetry code: (i) $x - \frac{1}{2}, -y + \frac{1}{2}, -z + 1$.

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 the equivalent isotropic displacement parameters of their parent atoms and C–H distances were set at 0.93 \AA for aromatic H atoms, 0.96 \AA for methyl H atoms, and 0.97 \AA for the remainder. N–H distances were fixed at 0.86 \AA . Although there were not sufficient Friedel pairs to determine reliably the absolute structure, the values of the Flack (1983) parameter, 0.3 (2) for the reported model and 0.7 (2) for the inverted one, seem to be in favour of the former.

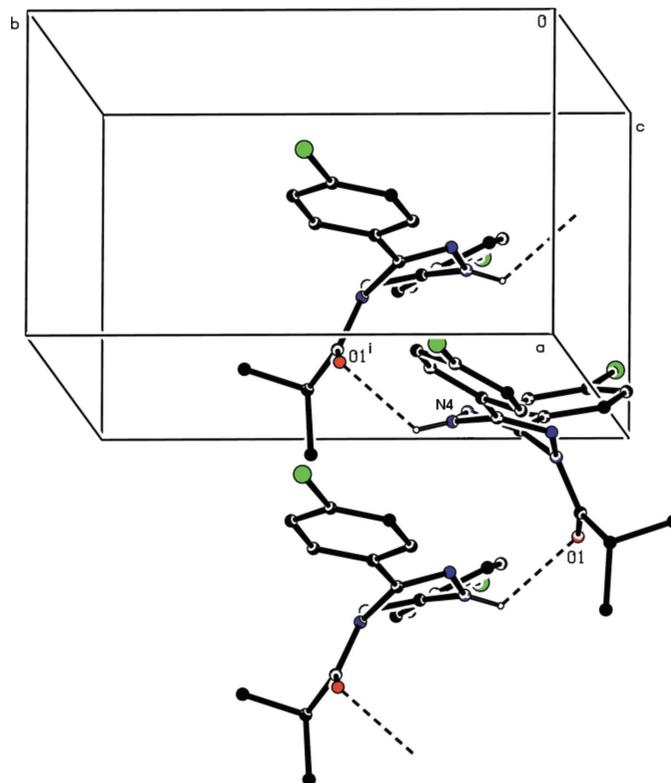


Figure 2

View showing the N–H \cdots O hydrogen bonding and the formation of the zigzag chain. For clarity, only H atoms involved in hydrogen bonding (dashed lines) are represented. [Symmetry code: (i) $x - \frac{1}{2}, \frac{1}{2} - y, 1 - z$.]

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: *ORTEP3* for Windows (Version 1.05; Farrugia, 1997) and *PLATON* (Spek, 2003; software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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