Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Dipropyl 3,6-bis(4-chlorophenyl)-1,2-dihydro-1,2,4,5-tetrazine-1,2-dicarboxylate

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Received 17 May 2005
Accepted 31 May 2005
Online 17 June 2005

Key indicators

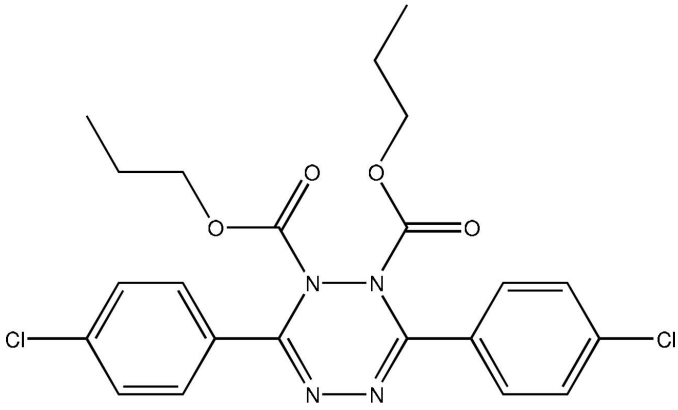
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.041
wR factor = 0.134
Data-to-parameter ratio = 14.6

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

The title compound, $C_{22}H_{22}Cl_2N_4O_4$, was prepared from propyl chloroformate and 3,6-bis(4-chlorophenyl)-1,2-dihydro-1,2,4,5-tetrazine. The six-membered 1,2-dihydro-1,2,4,5-tetrazine ring has a twist conformation.

Comment

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



(I)

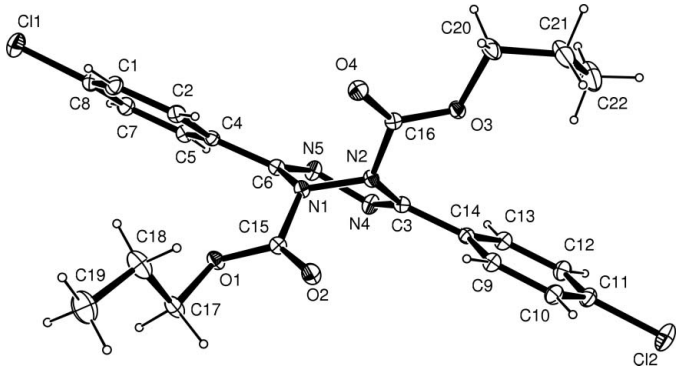


Figure 1
The molecular structure of (I); displacement ellipsoids are drawn at the 30% probability level.

The molecular structure of (I) is illustrated in Fig. 1. Atoms C3, N4, N5 and C6 are not coplanar, showing deviations of ±0.1320 (15) Å from the mean plane. However, the substituted N atoms, N1 and N2, are displaced from the C3/N4/N5/C6 mean plane much more significantly, *viz*. by 0.320 (5) and −0.338 (4) Å, respectively, thus indicating a twist conformation of the 1,2-dihydro-1,2,4,5-tetrazine ring.

Experimental

The title compound was prepared according to the procedure reported by Rao & Hu (2003). A solution of the compound in ethanol was concentrated gradually at room temperature to afford colorless parallelepiped-shaped crystals (m.p. 387–388 K).

Crystal data

$C_{22}H_{22}Cl_2N_4O_4$
M_r = 477.34
Monoclinic, $C2/c$
a = 25.824 (13) Å
b = 13.962 (3) Å
c = 15.861 (4) Å
β = 124.93 (4)°
V = 4689 (4) Å^3
Z = 8
D_x = 1.352 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 11.5–13.0°
μ = 0.31 mm^{-1}
T = 298 (2) K
Parallelepiped, colorless
0.40 × 0.30 × 0.30 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.894, T_{max} = 0.911
4694 measured reflections
4222 independent reflections
2787 reflections with $I > 2\sigma(I)$
R_{int} = 0.017
θ_{max} = 25.2°
$h = 0 \rightarrow 30$
$k = -1 \rightarrow 16$
$l = -18 \rightarrow 15$
3 standard reflections
frequency: 60 min
intensity decay: 0.3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.134
S = 1.03
4222 reflections
290 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0608P)^2 + 4.8707P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.55 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0029 (3)

H atoms were included in calculated positions (C—H distances of 0.93 Å for benzene, 0.96 Å for methyl and 0.97 Å for the rest of the C—H bonds) and refined using a riding model. Their isotropic displacement parameters were set equal to 1.2 (or 1.5 for methyl H atoms) times the equivalent isotropic displacement parameters of their parent atoms.

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* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

The authors are grateful to the National Natural and Scientific Foundation (grant No. 20272053) for financial support.

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