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6-(4-Chlorobenzyl)-3-phenyl-1,2,4,5-tetrazine

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In the title compound, $C_{15}H_{11}ClN_4$, the tetrazine ring is twisted with respect to the chlorobenzene plane, with a dihedral angle of 69.4 (3)°. Weak C—H···N hydrogen bonding between neighboring molecules helps to stabilize the crystal structure.

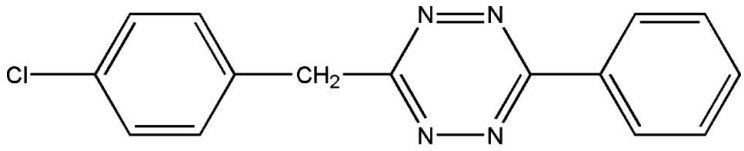
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.077
wR factor = 0.233
Data-to-parameter ratio = 16.4

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

Comment

1,2,4,5-Tetrazine derivatives possess a wide spectrum of antiviral and antitumor properties and have been widely used in pesticides and herbicides (Sauer, 1996). In a continuation of our work on the structure–activity relationships of 1,2,4,5-tetrazine derivatives (Hu *et al.*, 2004; 2005), we have prepared the title compound, (I), and determined its crystal structure.



(I)

The molecule structure of (I) is illustrated in Fig. 1. The tetrazine ring is coplanar with the C14-phenyl ring [dihedral angle 2.46 (3)°], but twisted with respect to the C8-benzene ring [dihedral angle 69.4 (3)°]. Weak C—H···N hydrogen bonding occurs between neighboring molecules (Table 1), which helps to stabilize the crystal structure.

Experimental

With sulfur (1.0 g) as catalyst, 85% hydrazine hydrate (10 ml, 170 mmol) was added dropwise to an anhydrous ethanol solution (15 ml) of 4-chlorobenzyl cyanide (50 mmol) and benzonitrile (50 mmol) at 295 K. After refluxing for 3 h, the mixture was cooled to room temperature and the resulting solid product was filtered off. The solid product was then dissolved in diethyl ether (15 ml) and oxidized by sodium nitrite (14 mmol) and acetic acid (14 mmol) over

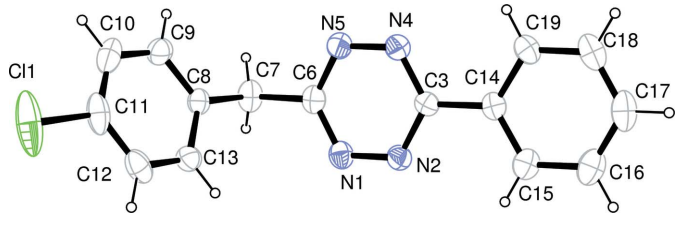


Figure 1
The molecular structure of (I), shown with 30% probability displacement ellipsoids (arbitrary spheres for H atoms).

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a period of 2 h to afford the product as purple crystals which were purified by preparative thin-layer chromatography over silica gel PF254 (2 mm) (cyclohexane–dichloromethane, 1:1) to give red single crystals of (I). The solid product was dissolved in tetrahydrofuran–anhydrous ethanol (4:1 *v*/*v*) and the solution evaporated gradually at room temperature to afford single crystals of (I).

Crystal data

$C_{15}H_{11}ClN_4$
M_r = 282.73
Monoclinic, $P2_1/c$
a = 6.246 (2) Å
b = 16.671 (6) Å
c = 14.173 (4) Å
β = 112.661 (12)°
V = 1361.9 (8) Å^3
Z = 4
D_x = 1.379 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.28 mm^{-1}
T = 293 (2) K
Prism, red
0.25 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
6707 measured reflections
2976 independent reflections
1595 reflections with $I > 2\sigma(I)$
R_{int} = 0.061
θ_{max} = 27.2°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.077
$wR(F^2)$ = 0.233
S = 0.96
2976 reflections
182 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1387P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.058 (9)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C13—H13···N5^{i}	0.93	2.55	3.295 (4)	137

Symmetry code: (i) $x-1, y, z$.

H atoms were placed in calculated positions, with C—H = 0.93 (aromatic) or 0.97 Å (methylene), and refined in riding mode, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; 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, 1999); software used to prepare material for publication: *SHELXL97*.

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