Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

6-Chloro-2-diethylamino-4-[(1,1,3,3-tetramethylbutyl)amino]-1,3,5-triazine

Jin-Peng Wang,[a]* Fei Wen,[a] Qing-Ming Zang[a] and Shuai Liu[b]

[a]College of Pharmaceuticals & Biotechnology, Tianjin University, Tianjin 300072, People's Republic of China, and [b]School of Chemical Engineering and Technology, Tianjin University, Tianjin 300072, People's Republic of China
Correspondence e-mail: dengyicoo@yahoo.com.cn

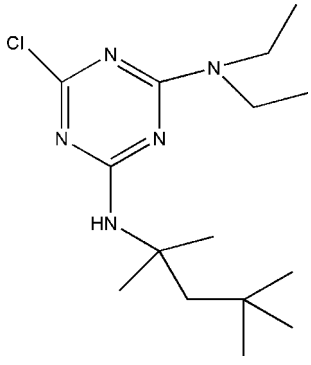
Received 14 May 2007; accepted 16 May 2007

Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.003 Å; R factor = 0.045; wR factor = 0.108; data-to-parameter ratio = 17.5.

The crystal structure of the title compound, $C_{15}H_{28}ClN_5$, is stabilized by intermolecular N—H···N hydrogen bonds.

Related literature

For related literature, see: Borzatta & Carrozza (1991); Deng *et al.* (2006); Kaiser & Thurston (1951).



Experimental

Crystal data

$C_{15}H_{28}ClN_5$
M_r = 313.87
Monoclinic, $C2/c$
a = 19.239 (3) Å
b = 7.9613 (12) Å
c = 24.105 (4) Å
β = 102.684 (5)°
V = 3602.0 (10) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.22 mm^{-1}
T = 113 (2) K
0.32 × 0.22 × 0.20 mm

Data collection

Rigaku Saturn diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.935, T_{max} = 0.958
14117 measured reflections
3524 independent reflections
2993 reflections with $I > 2\sigma(I)$
R_{int} = 0.051

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.108
S = 1.10
3524 reflections
201 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4···N3^{i}	0.85 (2)	2.25 (2)	3.098 (2)	172 (2)

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

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *CrystalStructure* (Rigaku/MSC, 2005).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2369).

References

Borzatta, V. & Carrozza, P. (1991). European Patent EP 0 462 069.
Bruker (1997). *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Deng, Y., Wang, X.-J., Wen, F., Wang, L. & Zhang, Y. (2006). *Acta Cryst.* E**62**, o5207–o5208.
Kaiser, D. W. & Thurston, J. T. (1951). *J. Am. Chem. Soc.* **73**, 2984–2986.
Rigaku/MSC (2005). *CrystalClear* and *CrystalStructure*. Rigaku/MSC, The Woodlands, Texas, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

supplementary materials

Acta Cryst. (2007). E**63**, o3017 [**doi:10.1107/S1600536807024099**]

6-Chloro-2-diethylamino-4-[(1,1,3,3-tetramethylbutyl)amino]-1,3,5-triazine

J.-P. Wang, F. Wen, Q.-M. Zang and S. Liu

Comment

The title compound (Fig. 1) is an important intermediate for the synthesis of hindered light stabilizers (Borzatta & Carrozza, 1991). These compounds containing a triazine ring are widely used (Deng *et al.*, 2006).

The triazine ring is essentially planar with an r.m.s. deviation of 0.013 Å. The molecules are linked by intermolecular N—H···N hydrogen bonds.

Experimental

The title compound was prepared according to the method of Kaiser & Thurston (1951). 2,4,6-Trichloro-1,3,5-triazine (40.0 g, 0.217 mol) was dissolved in toluene (120 ml) and then cooled to 278 K. With stirring, a solution of 2,4,4-trimethylpentan-2-amine (27.5 g, 0.213 mol) in toluene (50 ml) was then added dropwise to the mixture over a period of 0.5 h. Then a solution of Na_2CO_3 (23.02 g, 0.217 mol) in water (50 ml) was then added dropwise for 0.5 h. The mixture was stirred at 273–278 K for a further 3 h. Diethylamine(15.84 g, 0.217 mol) was and solid Na_2CO_3 (23.02 g, 0.217 mol) were added to the mixture, maintaining the temperature at 338 k for 5 h. The organic layer was washed with water and then concentrated *in vacuo*. The title compound (54.50 g) was obtained in powder form in a yield of 80.0%. Crystals of (I) were obtained by slow evaporation of a solution of methanol (m.p. 427–429 K).

Refinement

H atoms bonded to C were positioned geometrically (C—H=0.98–0.99 Å), and refined as riding with U_{iso}(H)=1.2U_{eq}(C) or 1.5$_{eq}$(methyl groups). The methyl groups were allowed to rotate but not to tip. The H atom bonded to N was freely refined.

Figures

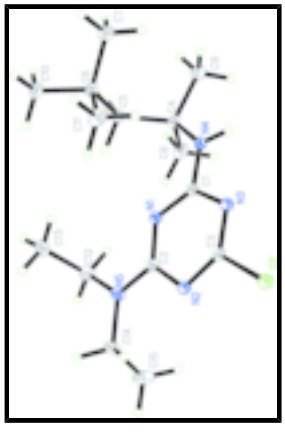


Fig. 1. A view of the molecular of (I). Displacement ellopsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

6-chloro-2-diethylamino-4-[(1,1,3,3-tetramethylbutyl)amino] −1,3,5-triazine

Crystal data

$C_{15}H_{28}ClN_5$	D_x = 1.158 Mg m^{-3}
M_r = 313.87	Melting point: 94-96 K
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71070 Å
a = 19.239 (3) Å	Cell parameters from 3245 reflections
b = 7.9613 (12) Å	θ = 2.8–25.0º
c = 24.105 (4) Å	μ = 0.22 mm^{-1}
β = 102.684 (5)º	*T* = 113 (2) K
V = 3602.0 (10) Å^3	Prism, colorless
Z = 8	0.32 × 0.22 × 0.20 mm
F_{000} = 1360	

Data collection

Rigaku Saturn diffractometer	2993 reflections with *I* > 2s(*I*)
Radiation source: rotating anode	R_{int} = 0.051
Monochromator: confocal	θ_{max} = 26.0º
T = 113(2) K	θ_{min} = 1.7º
ω scans	*h* = −23→23
Absorption correction: multi-scan (CrystalClear; Rigaku/MSC, 2005)	*k* = −9→9
T_{min} = 0.935, T_{max} = 0.958	*l* = −29→29
14117 measured reflections	Standard reflections: ?
3524 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.045	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.108	$w = 1/[\sigma^2(F_o^2) + (0.051P)^2 + 0.7372P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.10	$(\Delta/\sigma)_{max} < 0.001$
3524 reflections	$\Delta\rho_{max}$ = 0.28 e Å^{-3}
201 parameters	$\Delta\rho_{min}$ = −0.27 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.61905 (2)	0.77094 (6)	0.160423 (18)	0.02578 (15)
N1	0.40684 (7)	0.54339 (18)	0.10629 (6)	0.0192 (3)
N2	0.50602 (7)	0.69474 (18)	0.08423 (6)	0.0197 (3)
N3	0.50886 (7)	0.61285 (18)	0.18027 (6)	0.0195 (3)
N4	0.41177 (8)	0.48292 (19)	0.20162 (7)	0.0211 (3)
N5	0.40728 (8)	0.62277 (19)	0.01429 (6)	0.0245 (4)
C1	0.44167 (9)	0.5478 (2)	0.16095 (7)	0.0183 (4)
C2	0.44069 (9)	0.6192 (2)	0.06958 (7)	0.0199 (4)
C3	0.53402 (9)	0.6820 (2)	0.13897 (7)	0.0186 (4)
C4	0.33961 (9)	0.4098 (2)	0.19432 (7)	0.0234 (4)
C5	0.32706 (9)	0.2567 (2)	0.15346 (7)	0.0205 (4)
H5A	0.3246	0.3022	0.1149	0.025*
H5B	0.2788	0.2143	0.1538	0.025*
C6	0.37619 (9)	0.0995 (2)	0.15906 (7)	0.0238 (4)
C7	0.38462 (11)	0.0073 (3)	0.21581 (8)	0.0365 (5)
H7A	0.4065	−0.1027	0.2132	0.055*
H7B	0.3377	−0.0079	0.2247	0.055*
H7C	0.4150	0.0735	0.2459	0.055*
C8	0.28372 (9)	0.5440 (2)	0.17013 (9)	0.0315 (5)
H8A	0.2920	0.6445	0.1941	0.047*
H8B	0.2360	0.4999	0.1694	0.047*
H8C	0.2875	0.5730	0.1314	0.047*
C9	0.33090 (11)	0.3647 (3)	0.25456 (8)	0.0351 (5)
H9A	0.3712	0.2952	0.2735	0.053*
H9B	0.2864	0.3022	0.2520	0.053*
H9C	0.3296	0.4679	0.2765	0.053*
C10	0.33932 (11)	−0.0216 (2)	0.11220 (8)	0.0339 (5)
H10A	0.3692	−0.1212	0.1121	0.051*
H10B	0.3321	0.0346	0.0752	0.051*
H10C	0.2931	−0.0555	0.1194	0.051*
C11	0.45047 (10)	0.1395 (3)	0.14874 (9)	0.0360 (5)
H11A	0.4762	0.2119	0.1793	0.054*
H11B	0.4456	0.1974	0.1122	0.054*

H11C	0.4771	0.0348	0.1481	0.054*
C12	0.33647 (9)	0.5470 (3)	−0.00460 (8)	0.0300 (5)
H12A	0.3111	0.6037	−0.0398	0.036*
H12B	0.3087	0.5652	0.0249	0.036*
C13	0.34036 (10)	0.3595 (3)	−0.01590 (8)	0.0347 (5)
H13A	0.3705	0.3402	−0.0432	0.052*
H13B	0.2923	0.3163	−0.0316	0.052*
H13C	0.3607	0.3012	0.0198	0.052*
C14	0.43836 (10)	0.7093 (3)	−0.02854 (8)	0.0296 (5)
H14A	0.4250	0.6482	−0.0651	0.036*
H14B	0.4910	0.7075	−0.0162	0.036*
C15	0.41314 (12)	0.8903 (3)	−0.03733 (10)	0.0466 (6)
H15A	0.3610	0.8926	−0.0490	0.070*
H15B	0.4337	0.9421	−0.0669	0.070*
H15C	0.4284	0.9527	−0.0017	0.070*
H4	0.4351 (11)	0.508 (3)	0.2348 (9)	0.035 (6)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0212 (2)	0.0333 (3)	0.0230 (2)	−0.01011 (19)	0.00524 (18)	−0.00033 (19)
N1	0.0187 (7)	0.0192 (8)	0.0200 (7)	−0.0009 (6)	0.0049 (6)	0.0003 (6)
N2	0.0186 (7)	0.0212 (8)	0.0197 (8)	−0.0037 (6)	0.0052 (6)	−0.0005 (6)
N3	0.0181 (7)	0.0217 (8)	0.0197 (7)	−0.0034 (6)	0.0063 (6)	−0.0006 (6)
N4	0.0221 (8)	0.0231 (8)	0.0196 (8)	−0.0078 (6)	0.0075 (7)	−0.0040 (7)
N5	0.0226 (8)	0.0296 (9)	0.0198 (8)	−0.0035 (7)	0.0012 (6)	0.0034 (7)
C1	0.0191 (8)	0.0154 (9)	0.0214 (9)	0.0002 (7)	0.0068 (7)	−0.0017 (7)
C2	0.0208 (9)	0.0178 (9)	0.0211 (9)	0.0025 (7)	0.0047 (7)	0.0015 (7)
C3	0.0174 (8)	0.0172 (9)	0.0217 (9)	−0.0025 (7)	0.0053 (7)	−0.0020 (7)
C4	0.0228 (9)	0.0214 (10)	0.0292 (10)	−0.0062 (7)	0.0128 (8)	−0.0035 (8)
C5	0.0183 (9)	0.0202 (9)	0.0234 (9)	−0.0036 (7)	0.0054 (7)	−0.0002 (7)
C6	0.0256 (9)	0.0174 (9)	0.0277 (10)	0.0009 (7)	0.0045 (8)	0.0002 (7)
C7	0.0431 (12)	0.0282 (11)	0.0349 (11)	0.0011 (9)	0.0013 (9)	0.0065 (9)
C8	0.0214 (9)	0.0255 (11)	0.0519 (13)	−0.0031 (8)	0.0169 (9)	−0.0056 (9)
C9	0.0405 (12)	0.0365 (12)	0.0352 (11)	−0.0153 (10)	0.0235 (9)	−0.0073 (9)
C10	0.0417 (12)	0.0213 (10)	0.0363 (11)	0.0024 (9)	0.0032 (9)	−0.0047 (9)
C11	0.0289 (11)	0.0265 (11)	0.0555 (14)	0.0060 (9)	0.0159 (10)	0.0012 (10)
C12	0.0207 (9)	0.0400 (12)	0.0257 (10)	−0.0045 (9)	−0.0027 (8)	0.0029 (9)
C13	0.0312 (11)	0.0452 (13)	0.0278 (11)	−0.0127 (10)	0.0064 (9)	−0.0057 (9)
C14	0.0291 (10)	0.0395 (12)	0.0197 (9)	−0.0024 (9)	0.0040 (8)	0.0068 (9)
C15	0.0500 (14)	0.0439 (14)	0.0475 (14)	0.0003 (11)	0.0143 (11)	0.0218 (11)

Geometric parameters (Å, °)

Cl1—C3	1.7531 (17)	C8—H8A	0.9800
N1—C1	1.341 (2)	C8—H8B	0.9800
N1—C2	1.351 (2)	C8—H8C	0.9800
N2—C3	1.315 (2)	C9—H9A	0.9800
N2—C2	1.368 (2)	C9—H9B	0.9800

N3—C3	1.319 (2)	C9—H9C	0.9800
N3—C1	1.375 (2)	C10—H10A	0.9800
N4—C1	1.344 (2)	C10—H10B	0.9800
N4—C4	1.480 (2)	C10—H10C	0.9800
N4—H4	0.85 (2)	C11—H11A	0.9800
N5—C2	1.347 (2)	C11—H11B	0.9800
N5—C12	1.468 (2)	C11—H11C	0.9800
N5—C14	1.473 (2)	C12—C13	1.522 (3)
C4—C8	1.537 (3)	C12—H12A	0.9900
C4—C9	1.540 (2)	C12—H12B	0.9900
C4—C5	1.552 (2)	C13—H13A	0.9800
C5—C6	1.557 (2)	C13—H13B	0.9800
C5—H5A	0.9900	C13—H13C	0.9800
C5—H5B	0.9900	C14—C15	1.520 (3)
C6—C7	1.529 (3)	C14—H14A	0.9900
C6—C10	1.535 (3)	C14—H14B	0.9900
C6—C11	1.537 (2)	C15—H15A	0.9800
C7—H7A	0.9800	C15—H15B	0.9800
C7—H7B	0.9800	C15—H15C	0.9800
C7—H7C	0.9800		
C1—N1—C2	115.04 (14)	C4—C8—H8C	109.5
C3—N2—C2	112.08 (13)	H8A—C8—H8C	109.5
C3—N3—C1	112.02 (14)	H8B—C8—H8C	109.5
C1—N4—C4	127.23 (15)	C4—C9—H9A	109.5
C1—N4—H4	112.0 (14)	C4—C9—H9B	109.5
C4—N4—H4	119.4 (14)	H9A—C9—H9B	109.5
C2—N5—C12	120.47 (14)	C4—C9—H9C	109.5
C2—N5—C14	121.64 (15)	H9A—C9—H9C	109.5
C12—N5—C14	117.82 (14)	H9B—C9—H9C	109.5
N1—C1—N4	120.32 (15)	C6—C10—H10A	109.5
N1—C1—N3	124.91 (14)	C6—C10—H10B	109.5
N4—C1—N3	114.76 (15)	H10A—C10—H10B	109.5
N5—C2—N1	117.63 (15)	C6—C10—H10C	109.5
N5—C2—N2	117.26 (14)	H10A—C10—H10C	109.5
N1—C2—N2	125.11 (15)	H10B—C10—H10C	109.5
N2—C3—N3	130.70 (15)	C6—C11—H11A	109.5
N2—C3—Cl1	114.51 (12)	C6—C11—H11B	109.5
N3—C3—Cl1	114.79 (13)	H11A—C11—H11B	109.5
N4—C4—C8	109.38 (14)	C6—C11—H11C	109.5
N4—C4—C9	105.84 (14)	H11A—C11—H11C	109.5
C8—C4—C9	107.93 (15)	H11B—C11—H11C	109.5
N4—C4—C5	113.30 (13)	N5—C12—C13	112.33 (16)
C8—C4—C5	107.86 (14)	N5—C12—H12A	109.1
C9—C4—C5	112.39 (14)	C13—C12—H12A	109.1
C4—C5—C6	124.73 (15)	N5—C12—H12B	109.1
C4—C5—H5A	106.1	C13—C12—H12B	109.1
C6—C5—H5A	106.1	H12A—C12—H12B	107.9
C4—C5—H5B	106.1	C12—C13—H13A	109.5
C6—C5—H5B	106.1	C12—C13—H13B	109.5

H5A—C5—H5B	106.3	H13A—C13—H13B	109.5
C7—C6—C10	107.35 (15)	C12—C13—H13C	109.5
C7—C6—C11	108.93 (16)	H13A—C13—H13C	109.5
C10—C6—C11	107.87 (16)	H13B—C13—H13C	109.5
C7—C6—C5	113.98 (15)	N5—C14—C15	112.00 (16)
C10—C6—C5	105.52 (14)	N5—C14—H14A	109.2
C11—C6—C5	112.82 (15)	C15—C14—H14A	109.2
C6—C7—H7A	109.5	N5—C14—H14B	109.2
C6—C7—H7B	109.5	C15—C14—H14B	109.2
H7A—C7—H7B	109.5	H14A—C14—H14B	107.9
C6—C7—H7C	109.5	C14—C15—H15A	109.5
H7A—C7—H7C	109.5	C14—C15—H15B	109.5
H7B—C7—H7C	109.5	H15A—C15—H15B	109.5
C4—C8—H8A	109.5	C14—C15—H15C	109.5
C4—C8—H8B	109.5	H15A—C15—H15C	109.5
H8A—C8—H8B	109.5	H15B—C15—H15C	109.5
C2—N1—C1—N4	177.07 (15)	C1—N3—C3—N2	−1.3 (3)
C2—N1—C1—N3	−4.2 (2)	C1—N3—C3—Cl1	179.02 (12)
C4—N4—C1—N1	−3.4 (3)	C1—N4—C4—C8	−60.6 (2)
C4—N4—C1—N3	177.73 (15)	C1—N4—C4—C9	−176.65 (17)
C3—N3—C1—N1	4.3 (2)	C1—N4—C4—C5	59.8 (2)
C3—N3—C1—N4	−176.88 (14)	N4—C4—C5—C6	52.9 (2)
C12—N5—C2—N1	0.3 (2)	C8—C4—C5—C6	174.13 (15)
C14—N5—C2—N1	177.13 (15)	C9—C4—C5—C6	−67.0 (2)
C12—N5—C2—N2	−178.93 (15)	C4—C5—C6—C7	58.4 (2)
C14—N5—C2—N2	−2.1 (2)	C4—C5—C6—C10	175.92 (15)
C1—N1—C2—N5	−178.17 (15)	C4—C5—C6—C11	−66.5 (2)
C1—N1—C2—N2	1.0 (2)	C2—N5—C12—C13	−85.1 (2)
C3—N2—C2—N5	−179.25 (15)	C14—N5—C12—C13	97.89 (19)
C3—N2—C2—N1	1.6 (2)	C2—N5—C14—C15	−92.2 (2)
C2—N2—C3—N3	−1.4 (3)	C12—N5—C14—C15	84.8 (2)
C2—N2—C3—Cl1	178.24 (12)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4···N3^{i}	0.85 (2)	2.25 (2)	3.098 (2)	172 (2)

Symmetry codes: (i) −*x*+1, *y*, −*z*+1/2.

Fig. 1