

2,6-Di-*tert*-butyl-4-[[*N*-(4,6-dichloro-1,3,5-triazin-2-yl)propylamino]methyl]phenol

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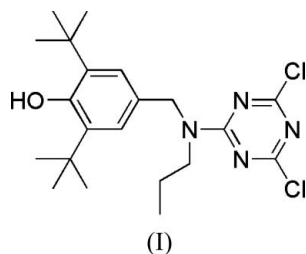
Key indicators

Single-crystal X-ray study
 $T = 294$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
Disorder in main residue
 R factor = 0.050
 wR factor = 0.149
Data-to-parameter ratio = 17.1For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The title compound, $\text{C}_{21}\text{H}_{30}\text{Cl}_2\text{N}_4\text{O}$, contains a sterically hindered phenol group. The dihedral angle between the two rings is $72.3(1)^\circ$. The propyl chain is disordered over two orientations.

Comment

Hindered phenol antioxidants are widely used in polymers and lubricants since they can protect polymers by increasing both their process stability and long-term stability against oxidative degradation (Yamazaki & Seguchi, 1997). Furthermore, amino derivatives of 2,4,6-trichloro-1,3,5-triazine are widely used as starting materials for many products, including drugs and light stabilizers (Mathias & Simanek, 1994; Manasek & Hrdlovik, 1990).



The title compound, (I), has been synthesised from 2,6-di-*tert*-butyl-4-[(propylamino)methyl]phenol and 2,4,6-trichloro-1,3,5-triazine. The bond length and angles are normal (Allen *et al.*, 1987) and compare well with those in a similar compound, *viz.* *N*-(4,6-dichloro-1,3,5-triazin-2-yl)aniline (Zeng *et al.*, 2005). The dihedral angle between the triazine ring and the benzene ring is $72.3(1)^\circ$. The propyl chain was found to be disordered over two orientations. The molecular structure is stabilized by four intramolecular hydrogen bonds (Table 1 and Fig. 1). However, atom H1 of the phenolic group is not involved in hydrogen bonding, presumably because of the steric hindrance of the *tert*-butyl groups.

Experimental

2,6-Di-*tert*-butyl-4-[(propylamino)methyl]phenol was prepared according to the method that we have reported in an earlier paper (Shu *et al.*, 2005). 2,4,6-Trichloro-1,3,5-triazine (4.61 g, 0.025 mol) and 2,6-di-*tert*-butyl-4-[(propylamino)methyl]phenol (6.8 g, 0.025 mol) were added to acetone (50 ml) and stirred at 273 K for 2 h. A solution of Na_2CO_3 (1.38 g, 0.013 mol) in water (10 ml) was then added dropwise over a period of 1 h. The reaction mixture was stirred at 273–278 K for a further 3 h. The precipitate was filtered off and the acetone was evaporated under reduced pressure. The title compound (9.89 g) was obtained in a yield of 93%. Suitable crystals (m.p. 377–

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379 K) were obtained by slow evaporation of a solution in a mixture of dichloromethane and ethyl acetate (6:1 v/v).

Crystal data

$C_{21}H_{30}Cl_2N_4O$
 $M_r = 425.39$
 Monoclinic, $P2_1/n$
 $a = 9.9134 (17) \text{ \AA}$
 $b = 19.373 (3) \text{ \AA}$
 $c = 12.378 (2) \text{ \AA}$
 $\beta = 98.037 (3)^\circ$
 $V = 2353.9 (7) \text{ \AA}^3$

$Z = 4$
 $D_x = 1.200 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 $\mu = 0.29 \text{ mm}^{-1}$
 $T = 294 (2) \text{ K}$
 Block, colourless
 $0.54 \times 0.40 \times 0.38 \text{ mm}$

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.840$, $T_{\max} = 0.895$

13049 measured reflections
 4796 independent reflections
 2364 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.044$
 $\theta_{\text{max}} = 26.5^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.050$
 $wR(F^2) = 0.150$
 $S = 1.00$
 4796 reflections
 281 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0569P)^2 + 0.7546P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.25 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.26 \text{ e \AA}^{-3}$
 Extinction correction: SHELXL97
 Extinction coefficient: 0.0197 (15)

Table 1

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C12-H12B\cdots O1$	0.96	2.32	2.974 (4)	125
$C14-H14B\cdots O1$	0.96	2.47	3.077 (4)	121
$C16-H16B\cdots O1$	0.96	2.40	3.026 (4)	122
$C17-H17B\cdots O1$	0.96	2.39	3.001 (4)	121

H1, attached to O1, was located in a difference Fourier map; it was then constrained to ride on O1 with $O-H = 1.02 \text{ \AA}$ and $U_{\text{iso}}(\text{H}) = 0.11 \text{ \AA}^2$. All other H atoms were positioned geometrically and refined using a riding model, with $C-H = 0.93-0.97 \text{ \AA}$; $U_{\text{iso}}(\text{H}) = xU_{\text{eq}}(\text{carrier atom})$, where $x = 1.5$ for methyl H and 1.2 for all other H atoms. Atoms C20, C21 and the H atoms attached to C19, C20 and C21 are disordered over two orientations; the site-occupancy factors refined to 0.603 (7) and 0.397 (7).

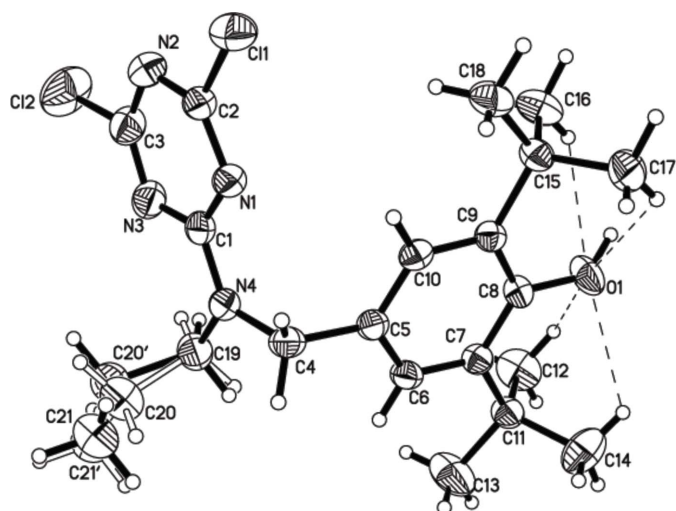


Figure 1

A view of the molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii. Both disorder components are shown. Dashed lines indicate the intramolecular hydrogen bonds.

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: SHELXTL (Bruker, 1997); software used to prepare material for publication: SHELXTL.

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