organic papers

3,5-Bis(4-fluorophenyl)-1-propyl-1,3,5-triaza-cyclohexane

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Key indicators

Single-crystal X-ray study
$T = 150$ K
Mean σ(C–C) = 0.004 Å
R factor = 0.070
wR factor = 0.172
Data-to-parameter ratio = 14.8

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

The heterocyclic ring of the title compound, $C_{18}H_{21}F_2N_3$, adopts a chair conformation with two 4-fluorophenyl groups in axial and one propyl group in an equatorial position. The torsion angles around the N—C bonds in the triazacyclohexane are in the range 55.8 (3)–60.2 (3)°.

Comment

Conformational effects of non-bonding electrons are a feature of heterocyclic chemistry (Riddel, 1980). The heterocyclic nucleus in the 1,3,5-triazacyclohexane is expected to adopt a chair conformation and four distinct types of substituent orientation can be postulated, *viz*. *eee*, *eea*, *eaa* and *aaa*, where *e* = equatorial and *a* = axial; all four conformers have axial repulsions involving substituents and/or lone pairs of electrons on the N atoms. Several conformations of 1,3,5-trialkyl-1,3,5-triazacyclohexanes in solution have been investigated by dipole moment measurement and NMR spectroscopy, the former approach suggesting varying amounts of *eee*, *eea* and *eaa* conformers. X-ray studies of the 1,3,5-tricyclohexyl compound established that it adopts the *eea* conformation in the crystal structure (Bouchemma *et al.*, 1988). The various 1,3,5-triaryl-1,3,5-triazacyclohexanes adopt the diaxial–equatorial orientation of substituents in the solid state, thus avoiding 1,3-diaxial lone-pair repulsions (Giumanini *et al.*, 1985; Gilardi *et al.*, 2003; Bouchemma *et al.*, 1989, 1990).

(I)
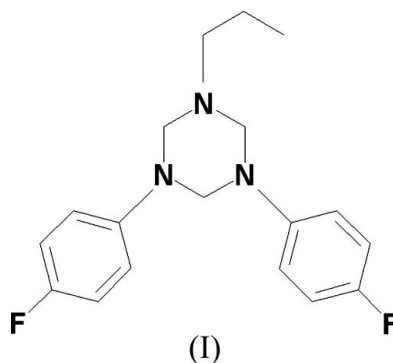
In the present work, a new derivate, (I), of triazacyclohexane is reported. The molecular geometry and the atom-numbering scheme of (I) are shown in Fig. 1. The 1,3,5-triazacyclohexane ring takes on the chair conformation which is typical of these rings (Gilardi *et al.*, 2003).

The structure of a very similar compound, *viz*. 1-propyl-3,5-bis(2-fluorophenyl)-1,3,5-triazacyclohexane, (II), has been reported (Latreche *et al.*, 2006). In both (I) and (II) the heterocyclic rings adopt chair conformations, with two

fluorophenyl substituents situated in axial positions, and a third (propyl) equatorial. The crystal structure is stabilized by van der Waal interactions and consists of alternating layers along the *b* axis, at $c = \frac{1}{4}$ and $c = \frac{3}{4}$.

Experimental

The title compound was obtained by mixing a 2:1:1 stoichiometric ratio of propylamine and 4-fluoroaniline with formalin in ethanol (25 ml) at 293 K. The resulting solution was evaporated on a rotary evaporator to dryness and the white residue was crystallized from cyclohexane.

Crystal data

$C_{18}H_{21}F_2N_3$	$V = 811.35\ (4)\ \text{Å}^3$
$M_r = 317.38$	$Z = 2$
Triclinic, $P\bar{1}$	$D_x = 1.299\ \text{Mg m}^{-3}$
$a = 6.0610\ (2)\ \text{Å}$	Mo $K\alpha$ radiation
$b = 10.0150\ (3)\ \text{Å}$	$\mu = 0.09\ \text{mm}^{-1}$
$c = 13.5200\ (4)\ \text{Å}$	$T = 150\ (2)\ \text{K}$
$\alpha = 91.4440\ (10)^\circ$	Prism, colourless
$\beta = 92.4060\ (10)^\circ$	$0.40 \times 0.25 \times 0.10\ \text{mm}$
$\gamma = 98.1090\ (17)^\circ$	

Data collection

Nonius KappaCCD diffractometer	3081 independent reflections
φ scans, and ω scans with κ offsets	2711 reflections with $I > 2\sigma(I)$
Absorption correction: none	$R_{int} = 0.049$
13114 measured reflections	$\theta_{max} = 25.8^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0293P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.070$	$+ 1.3984P]$
$wR(F^2) = 0.172$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.15$	$(\Delta/\sigma)_{max} < 0.001$
3081 reflections	$\Delta\rho_{max} = 0.30\ \text{e Å}^{-3}$
208 parameters	$\Delta\rho_{min} = -0.28\ \text{e Å}^{-3}$
H-atom parameters constrained	Extinction correction: *SHELXL97*
	Extinction coefficient: none

All H atoms were located in Fourier maps but introduced in calculated positions and treated as riding on their parent C atom, with C—H = 0.98 (methyl), 0.95 (aromatic) and 0.99 Å (methylene), and with $U_{iso}(H) = 1.5U_{eq}$(methyl C) or $1.2U_{eq}$(aromatic and methylene C atoms).

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR2002* (Burla *et al.*, 2003); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).
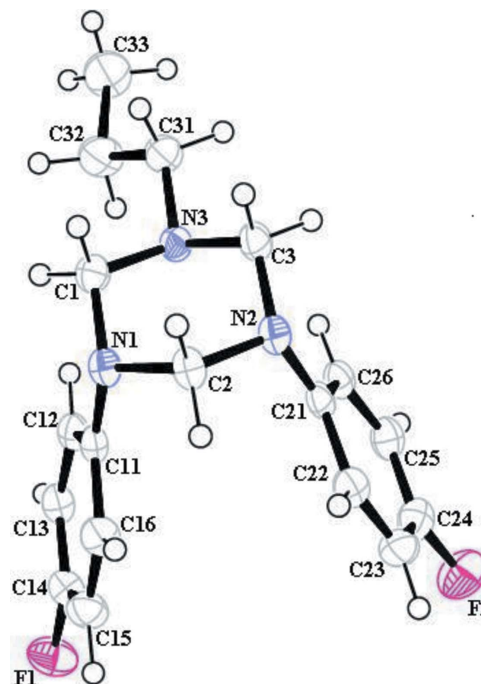
Figure 1
The molecular structure of the title compound, with the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

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