

4-*tert*-Butyl-1-(1-naphthyl)cyclohexene

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Key indicators

Single-crystal X-ray study

$T = 293$ K

Mean $\sigma(\text{C}-\text{C}) = 0.006$ Å

R factor = 0.063

wR factor = 0.259

Data-to-parameter ratio = 15.5

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

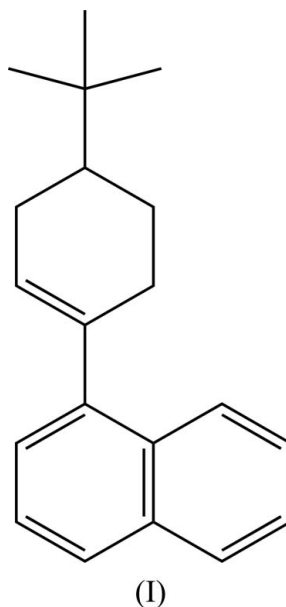
In the title compound, $\text{C}_{20}\text{H}_{24}$, the central cyclohexene ring is in a distorted half-chair conformation. The crystal structure is stabilized by weak van der Waals interactions.

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Comment

Recently, biological studies have been performed on 1-phenylcyclohexene. *In vitro* metabolites of the compound isolated from rat liver homogenates were identified to be various oxygenated products of synthetic value. This indicates the importance of 1-phenylcyclohexene and its analogue, (I), in biological studies (Cook *et al.*, 1984). In addition, alkenes undergo co-halogenation reactions to afford bifunctional compounds which serve as potential synthons towards the synthesis of various heterocyclic compounds (Rodriguez & Dulcere, 1993). We were interested in investigating some of the structural features of (I) which may alter the regio/stereoselectivity in addition reactions. In particular, we hoped to investigate the rotameric preference of the naphthyl ring system attached to one of the olefinic C atoms in (I).



The slightly distorted half-chair conformation of the cyclohexene ring (Fig. 1) is confirmed by the puckering analysis [$q_2 = 0.328$ (5) Å, $\varphi_2 = 29$ (1)° and $q_3 = 0.268$ (4) Å; Cremer & Pople, 1975]. The r.m.s. deviation from the naphthyl ring plane is 0.0197 Å.

The crystal structure of (I) is stabilized solely by weak van der Waals interactions (Fig. 2), with no evidence of significant C—H... π or π — π stacking interactions.

Experimental

A solution of 4-*tert*-butylcyclohexanone (2.6 ml) in dry diethyl ether (7.5 ml) was added gradually with stirring to a solution of naphthylmagnesiumbromide, prepared from magnesium turnings (0.6 g) and 1-bromonaphthalene (3 ml) in dry diethyl ether (15 ml). The Grignard complex was stirred for a further 2 h and then decomposed with ice-cold water. The ethereal layer was decanted, dried over anhydrous sodium sulfate, and the solvent evaporated to yield 4-*tert*-butyl-1-naphthylcyclohexanol. Crude 4-*tert*-butyl-1-naphthylcyclohexanol (4 g) was heated over a water bath for about 10 minutes with 6 ml of a mixture of 20% sulfuric acid/acetic acid with occasional swirling of the reaction flask. The reaction mixture was poured into an excess of water. The organic layer was extracted with diethyl ether, and then washed repeatedly with water, sodium bicarbonate solution and again with water. It was dried over anhydrous sodium sulfate, filtered and evaporated to afford 4-*tert*-butyl-1-(1-naphthyl)cyclohexene which was purified by column chromatography on silica gel with petroleum ether (333–353 K) as eluent. Crystals suitable for the X-ray crystallographic investigation were obtained from the resulting solution (yield: 65%). Elemental analysis calculated: C 90.85, H 9.15; found: C 90.75, H 9.19%.

Crystal data

$C_{20}H_{24}$	$Z = 8$
$M_r = 264.39$	$D_x = 1.079 \text{ Mg m}^{-3}$
Orthorhombic, <i>Pccn</i>	Mo $K\alpha$ radiation
$a = 23.8908 (18) \text{ \AA}$	$\mu = 0.06 \text{ mm}^{-1}$
$b = 16.1042 (11) \text{ \AA}$	$T = 293 (2) \text{ K}$
$c = 8.4622 (6) \text{ \AA}$	Block, colourless
$V = 3255.8 (4) \text{ \AA}^3$	$0.24 \times 0.21 \times 0.17 \text{ mm}$

Data collection

Nonius MACH3 diffractometer	1197 reflections with $I > 2\sigma(I)$
ω - 2θ scans	$R_{\text{int}} = 0.015$
Absorption correction: ψ scan	$\theta_{\text{max}} = 25.0^\circ$
(North <i>et al.</i> , 1968)	3 standard reflections
$T_{\text{min}} = 0.972$, $T_{\text{max}} = 0.990$	frequency: 60 min
3000 measured reflections	intensity decay: none
2850 independent reflections	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.1398P)^2 + 0.6525P]$
$R[F^2 > 2\sigma(F^2)] = 0.063$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.259$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.01$	$\Delta\rho_{\text{max}} = 0.35 \text{ e \AA}^{-3}$
2850 reflections	$\Delta\rho_{\text{min}} = -0.15 \text{ e \AA}^{-3}$
184 parameters	
H-atom parameters constrained	

Table 1

Selected torsion angles ($^\circ$).

C7—C2—C3—C4	−54.3 (5)	C6—C5—C8—C17	−63.5 (5)
C3—C2—C7—C6	40.1 (5)		

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93–0.98 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{parent atom})$.

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: *SHELXTL/PC* (Bruker, 2000); program(s) used to refine structure: *SHELXTL/PC*;

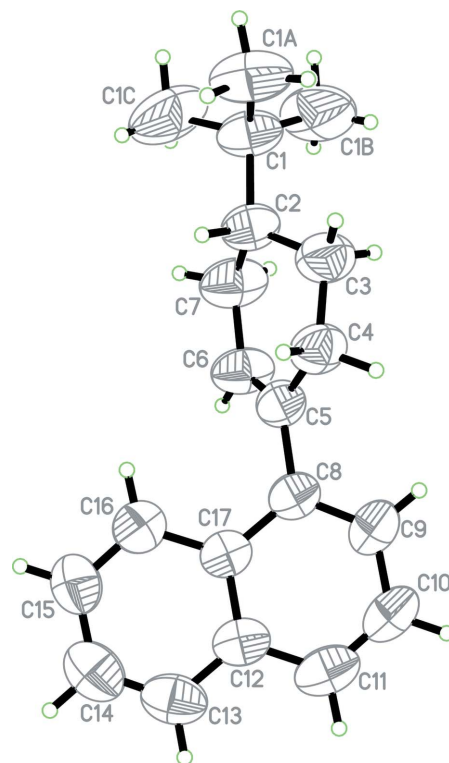


Figure 1

The molecular structure of the title compound, (I), showing the atom numbering scheme and 50% probability displacement ellipsoids.

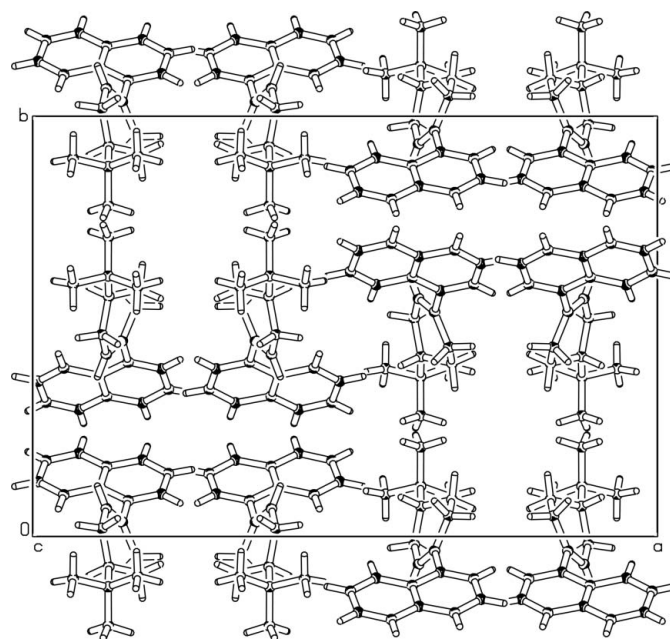


Figure 2

Packing diagram of (I), viewed down the *c* axis.

molecular graphics: *SHELXTL/PC* and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL/PC*.

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