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1-(3-Mesityl-3-methylcyclobutyl)-2-(pyrrolidin-1-yl)-ethan-1-one

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Key indicators

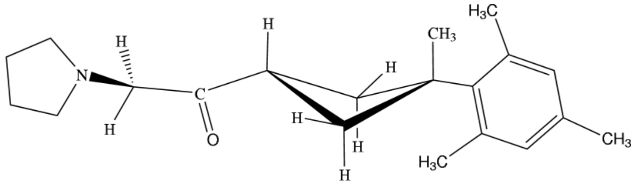
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.068
wR factor = 0.194
Data-to-parameter ratio = 16.8

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

In the title molecule, $C_{20}H_{29}NO$, the cyclobutane ring is puckered, with a dihedral angle of 19.8 (3)° between the two planes. The pyrrolidine ring adopts an envelope conformation. There are intermolecular C—H···O and C—H···π interactions.

Comment

3-Substituted cyclobutane carboxylic acid derivatives exhibit anti-inflammatory and antidepressant activities (Dehmlow & Schmidt, 1990), and liquid crystal properties (Coghi *et al.*, 1976). A recent communication showed that compounds incorporating 1,3,4-trisubstituted pyrrolidine ring scaffolds were potent CCR5 antagonists (Lynch *et al.*, 2002). The pyrrolidine ring is a structure very often encountered in the alkaloid field (Martin & Brossi, 1987). Furthermore, numerous chiral non-racemic substituted pyrrolidines and pyrrolidones are used as intermediates, chiral ligands or auxiliaries in asymmetric synthesis (Huryn *et al.*, 1991). The development of new methods for the preparation of enantiomerically pure, highly substituted pyrrolidines and pyrrolidones is then of increasing interest. Taking into account the importance of cyclobutane and pyrrolidine, we have undertaken an X-ray diffraction study of the title compound, (I).



(I)

Fig. 1 shows the molecular structure and conformation of (I), with the atomic numbering scheme. The four-atom bridge (N1/C9/C10/C5) linking the cyclobutane and pyrrolidine rings is not planar, the N1—C9—C10—C5 torsion angle being −54.1 (4)°, which corresponds to the (−)synclinal configuration. Although close to planar, the cyclobutane ring is more puckered than that in a related compound; the C8/C5/C6 plane forms a dihedral angle of 19.8 (3)° with the C6/C7/C8 plane in (I) [11.55 (3)° in the related compound; Özdemir *et al.*, 2004]. However, the bond lengths in the cyclobutane ring are similar to those in the related compound. In (I), the pyrrolidine ring is close to an envelope conformation, with atom N1 deviating by 0.257 (4) Å from the C1–C4 plane, and puckering parameters (Cremer & Pople, 1975) Q_2 = 0.408 (5) Å and φ_2 = 354.1 (7)°.

The crystal structure does not exhibit intramolecular or π–π interactions. There are, however, C—H···O and C—H···π

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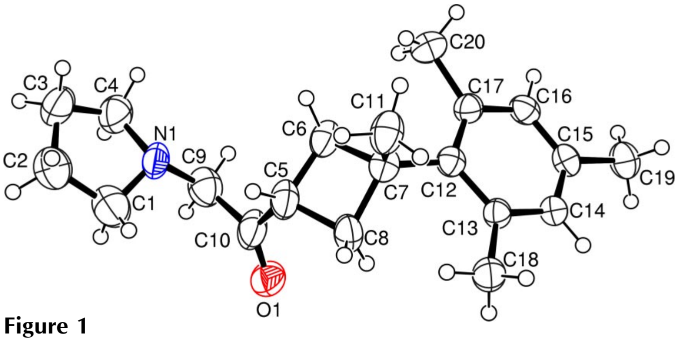

Figure 1
An *ORTEP*-3 (Farrugia, 1997) drawing of (I), showing the atomic numbering scheme. Displacement ellipsoids of non-H atoms are drawn at the 50% probability level.

intermolecular interactions (Table 2). Atom O1 and the centroid, *Cg*3, of the C12–C17 benzene ring act as a double acceptor for C—H···O and C—H···π interactions, respectively.

Experimental

A mixture of 1-phenyl-1-mesityl-3-(2-chloro-1-oxoethyl)cyclobutane (5.29 g, 20 mmol) and pyrrolidine (2.874 g, 40 mmol) in dry benzene (50 ml) was refluxed with continuous stirring. The course of the reaction was monitored by IR spectroscopy. The product was formed after about half an hour. After cooling to room temperature and filtration of the pyrrolidine salt, benzene was removed under reduced pressure through a rotary evaporator. The oily product was treated with diethyl ether and dried over magnesium sulfate. Shiny crystals of (I) suitable for X-ray analysis were obtained from a diethyl ether solution by deep freezing at 253 K (yield 78%, m.p. 412 K). IR (KBr): 1724 cm^{-1} (C═O). ^{1}H NMR (DMSO-d_6, p.p.m.): 1.59 (*s*, 3H, CH_3 on cyclobutane), 1.85 (*s*, 3H, *p*-CH_3 on mesitylene), 2.24–2.74 (*br*, 10H, *o*-CH_3 on mesitylene plus –CH_2– on pyrollidine), 2.78–3.04 (*br*, 8H, –CH_2—N—CH_2– plus –CH_2– in cyclobutane), 3.31 (*q*, *j* = 7.8 Hz, 1H, >CH–), 3.58 (*s*, 2H, –CO—CH_2—N), 6.77 (*s*, 2H, aromatics on mesitylene).

Crystal data

$C_{20}H_{29}NO$
$M_r = 299.44$
Tetragonal, $P4_2/n$
$a = 21.2110\ (9)$ Å
$c = 7.8940\ (4)$ Å
$V = 3551.6\ (3)$ Å^3
$Z = 8$
$D_x = 1.120$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 18 614 reflections
$\theta = 1.4–26.0°$
$\mu = 0.07$ mm^{-1}
$T = 293\ (2)$ K
Prism, yellow
0.42 × 0.29 × 0.20 mm

Data collection

Stoe IPDS-II diffractometer
ω scans
Absorption correction: none
23 972 measured reflections
3390 independent reflections
1642 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.068$
$\theta_{\rm max} = 26.0°$
$h = -26 \rightarrow 26$
$k = -26 \rightarrow 26$
$l = -9 \rightarrow 8$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.068$
$wR(F^2) = 0.194$
$S = 1.01$
3390 reflections
202 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0947P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.74$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.24$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0042 (12)

Table 1
Selected geometric parameters (Å, °).

O1—C10	1.186 (4)	C7—C12	1.525 (4)
N1—C1	1.401 (5)	C7—C11	1.534 (4)
N1—C9	1.450 (4)	C7—C8	1.554 (4)
N1—C4	1.561 (5)	C9—C10	1.572 (5)
C5—C10	1.498 (5)	C13—C18	1.508 (4)
C5—C6	1.527 (5)	C15—C19	1.521 (4)
C5—C8	1.540 (4)	C17—C20	1.521 (4)
C6—C7	1.565 (4)		
C1—N1—C9	114.5 (3)	C5—C6—C7	90.4 (3)
C1—N1—C4	105.5 (3)	C12—C7—C11	111.2 (2)
C9—N1—C4	108.3 (3)	C11—C7—C8	111.9 (3)
N1—C1—C2	104.7 (3)	C11—C7—C6	111.3 (2)
C1—C2—C3	106.1 (4)	C8—C7—C6	86.9 (2)
C4—C3—C2	106.7 (3)	C5—C8—C7	90.3 (3)
C3—C4—N1	98.0 (3)	O1—C10—C5	122.9 (3)
C6—C5—C8	88.8 (2)	O1—C10—C9	119.0 (3)
N1—C9—C10—C5	−54.1 (4)		

Table 2
Hydrogen-bonding geometry (Å, °).

*Cg*3 is the centroid of the C12–C17 benzene ring.

$D—\mathrm{H}\cdots A$	$D—\mathrm{H}$	$\mathrm{H}\cdots A$	$D\cdots A$	$D—\mathrm{H}\cdots A$
$\mathrm{C6—H6}B\cdots\mathrm{O1}^{\mathrm{i}}$	0.97	2.65	3.594 (4)	164
$\mathrm{C9—H9}A\cdots\mathrm{O1}^{\mathrm{i}}$	0.97	2.69	3.450 (5)	136
$\mathrm{C9—H9}B\cdots Cg3^{\mathrm{ii}}$	0.97	3.05	3.994 (4)	165
$\mathrm{C11—H11}A\cdots Cg3^{\mathrm{iii}}$	0.96	2.96	3.818 (3)	149

Symmetry codes: (i) $-x, 1 - y, 1 - z$; (ii) $-x, -y, 2 - z$; (iii) $-\frac{1}{2} - y, x, \frac{1}{2} - z$.

H atoms were positioned geometrically and treated as riding, with C—H bond lengths of 0.93–0.98 Å, and with $U_{\rm iso}(\mathrm{H}) = 1.2U_{\rm eq}(\mathrm{C})$ [$1.5U_{\rm eq}(\mathrm{C})$ for methyl]. In the final difference Fourier map, the maximum residual density was located 1.27 Å from atom H1*A*, and the minimum was 0.26 Å from H18*C*.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED*32 (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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