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# 1-[3-Methyl-3-(2,4,6-trimethylphenyl)cyclobutyl]-2-(thiomorpholin-4-yl)ethanone

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**Key indicators**

Single-crystal X-ray study
*T* = 293 K
Mean σ(C–C) = 0.002 Å
*R* factor = 0.049
*wR* factor = 0.146
Data-to-parameter ratio = 21.4

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

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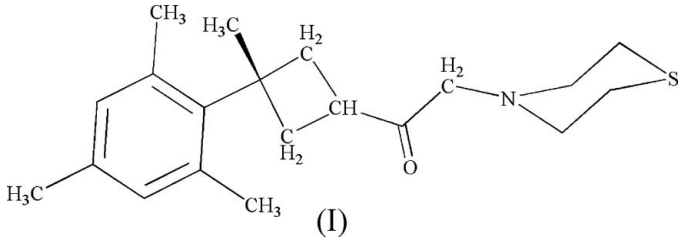
The title compound, $C_{20}H_{29}NOS$, the thiomorpholine ring adopts a chair conformation. The crystal structure is stabilized by intermolecular C—H···π interactions.

## Comment

Small and simple heterocyclic structures often exhibit complex biological properties (Matković-Čalogović *et al.*, 2003). Sulfur-containing heterocyclic scaffolds are constituents of several biologically active compounds (Mortezaei *et al.*, 1999). 3-Substituted cyclobutane carboxylic acid derivatives exhibit anti-inflammatory and antidepressant activities (Dehmlow & Schmidt, 1990). We report here the crystal structure of the title compound, (I).




(I)

Selected bond lengths and angles in (I) are given in Table 1. In the cyclobutane ring, the C4/C1/C2 plane forms a dihedral angle of 25.25 (9)° with the C2/C3/C4 plane. A survey of the geometry of cyclobutanes shows the average pucker to be 24.3° (Allen, 1984) and 23.5° (Swenson *et al.*, 1997) in acyclic substituted cyclobutane rings, and the present value is in agreement with the previous reports. The thiomorpholine ring exhibits a chair conformation. The S1···N1 distance is 3.1712 (15) Å, which is shorter than the sum of the van der Waals radii (3.35 Å; Bondi, 1964).

Analysis of the crystal packing shows that the molecules of (I) are linked by intermolecular C—H···π interactions

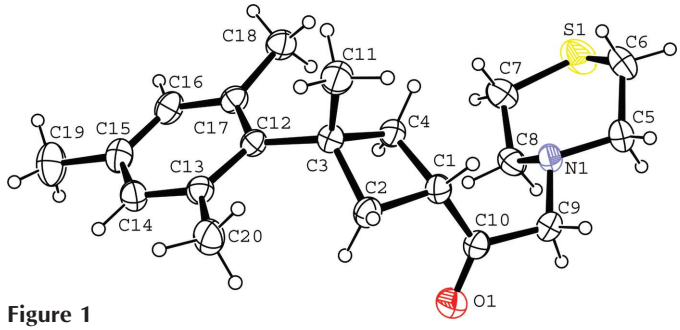



**Figure 1**
The molecular structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

(Table 2) involving the benzene ring and an H atom of the cyclobutane ring (Fig. 2).

## Experimental

Monitoring the reaction course by IR, a mixture of 1-mesityl-1-methyl-3-(2-chloro-1-oxoethyl)cyclobutane (1.323 g, 5 mmol), thiomorpholine (0.48 g, 5 mmol) and $K_2CO_3$ (0.346 g, 2.5 mmol) in absolute ethanol (50 ml) was refluxed for 10 min. The mixture was cooled to room temperature and water (200 ml) was added. The reaction mixture was then treated with diethyl ether, and the ether phase was separated and dried with $Na_2SO_4$. After the solvent had been removed under reduced pressure, compound (I) was crystallized from ethanol and was dried in air (yield 78%, m.p. 400 K). IR (KBr, $cm^{-1}$): 1698 (C═O); $^1$H NMR ($CDCl_3$, p.p.m.): $\delta$ 1.55 (*s*, 3H, –$CH_3$ on cyclobutane), 2.19 (*s*, 9H, –$CH_3$ on mesitylene), 2.34–2.87 (*m*, 12H, –$CH_2$–, thiomorpholine plus cyclobutane), 3.16 (*s*, 2H, –CO—$CH_2$—N), 3.42 (*q*, $J$ = 7.8 Hz, 1H, >CH– on cyclobutane), 6.73 (*s*, 2H, aromatics on mesitylene); $^{13}$H NMR ($CDCl_3$, p.p.m.): $\delta$ 202.24, 143.78, 135.28, 135.22, 130.64, 52.33, 45.55, 40.79, 39.54, 38.58, 36.41, 25.12, 21.60, 20.68. Calculated for $C_{20}H_{29}NOS$ (331 g $mol^{-1}$): C 72.51, H 8.76, N 4.23, S 9.67%; analysis found: C 71.94, H 8.84, N 4.39, S 9.81%.

*Crystal data*

| | |
|---|---|
| $C_{20}H_{29}NOS$ | $D_x$ = 1.175 Mg $m^{-3}$ |
| $M_r$ = 331.50 | Mo $K\alpha$ radiation |
| Monoclinic, $P2_1/n$ | Cell parameters from 1811 reflections |
| $a$ = 13.4594 (10) Å | $\theta$ = 1.5–27.9° |
| $b$ = 9.8855 (5) Å | $\mu$ = 0.18 $mm^{-1}$ |
| $c$ = 14.0878 (11) Å | $T$ = 293 (2) K |
| $\beta$ = 91.080 (6)° | Plate, colourless |
| $V$ = 1874.1 (2) Å$^3$ | 0.78 × 0.52 × 0.06 mm |
| $Z$ = 4 | |

*Data collection*

| | |
|---|---|
| Stoe IPDS-II diffractometer | 2988 reflections with $I > 2\sigma(I)$ |
| $\varphi$ scans | $R_{int}$ = 0.044 |
| Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002) | $\theta_{max}$ = 27.9° |
| $T_{min}$ = 0.888, $T_{max}$ = 0.991 | $h = -17 \rightarrow 17$ |
| 17351 measured reflections | $k = -12 \rightarrow 12$ |
| 4451 independent reflections | $l = -18 \rightarrow 18$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | H-atom parameters constrained |
| $R[F^2 > 2\sigma(F^2)]$ = 0.049 | $w = 1/[\sigma^2(F_o^2) + (0.0744P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $wR(F^2)$ = 0.146 | $(\Delta/\sigma)_{max}$ = 0.001 |
| $S$ = 1.17 | $\Delta\rho_{max}$ = 0.32 e Å$^{-3}$ |
| 4451 reflections | $\Delta\rho_{min}$ = −0.47 e Å$^{-3}$ |
| 208 parameters | |

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| S1—C6 | 1.790 (3) | N1—C5 | 1.457 (2) |
| S1—C7 | 1.790 (2) | N1—C9 | 1.458 (2) |
| O1—C10 | 1.211 (2) | N1—C8 | 1.462 (2) |
| | | | |
| C6—S1—C7 | 96.80 (10) | O1—C10—C1 | 122.29 (15) |
| N1—C9—C10 | 112.71 (13) | C1—C10—C9 | 118.03 (14) |
| | | | |
| C2—C1—C10—O1 | 8.7 (2) | N1—C9—C10—O1 | 134.26 (17) |
| C4—C1—C10—C9 | 87.57 (18) | N1—C9—C10—C1 | −47.9 (2) |

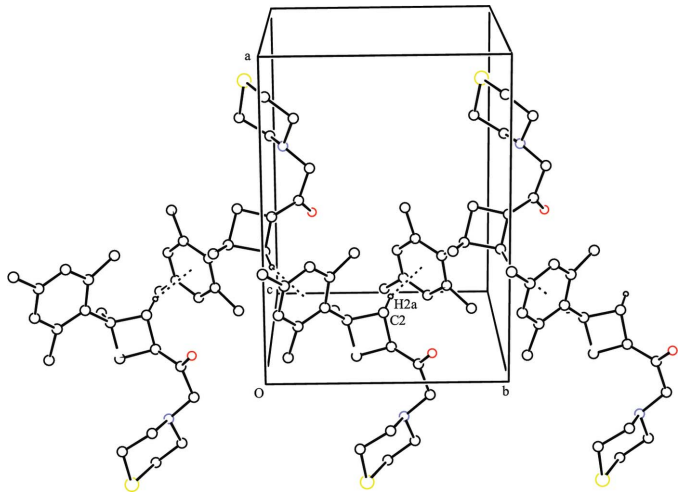



**Figure 2**
Part of the crystal packing of (I), showing C—H···$\pi$ interactions (dashed lines); only the H atoms involved in the interactions are shown.

**Table 2**
Hydrogen-bond geometry (Å, °).

*Cg*1 is the centroid of the benzene ring.

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C2—H2*A*···*Cg*1$^{i}$ | 0.97 | 2.68 | 3.612 (2) | 162 |

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

The H atoms were placed in calculated positions with C—H distances in the range 0.93–0.98 Å and refined using a riding model. The $U_{iso}$(H) values were constrained to be 1.2 (1.5 for methyl groups) times $U_{eq}$ of the carrier atom.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

## References


Allen, F. H. (1984). *Acta Cryst.* B**40**, 64–72.
Bondi, A. (1964). *J. Phys. Chem.* **68**, 441–451.
Dehmlow, E. V. & Schmidt, S. (1990). *Liebigs Ann. Chem.* pp. 411–413.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Matković-Čalogović, D., Popović, Z., Tralić-Kulenović, V., Racanè, L. & Karminski-Zamola, G. (2003). *Acta Cryst.* C**59**, o190–o191.
Mortezaei, R., Ida, S. & Campbell, D. A. (1999). *Mol. Divers.* 4, 143–148.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Stoe & Cie (2002). *X-AREA* (Version 1.18) and *X-RED32* (Version 1.04). Stoe & Cie, Darmstadt, Germany.
Swenson, D. C., Yamamoto, M. & Burton, D. J. (1997). *Acta Cryst.* C**53**, 1445–1447.