

Ying Chen, Haitao Zhang* and
Lina ZhouSchool of Chemical Engineering and Tech-
nology, Tianjin University, Tianjin 300072,
People's Republic of ChinaCorrespondence e-mail:
cheny_79@yahoo.com.cn

Key indicators

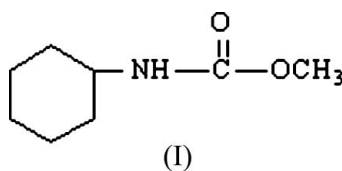
Single-crystal X-ray study
 $T = 293\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$
 R factor = 0.038
 wR factor = 0.105
Data-to-parameter ratio = 10.8For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Methyl *N*-cyclohexylcarbamate

The structure of the title compound, $\text{C}_8\text{H}_{15}\text{NO}_2$, is normal and contains a six-membered ring with a chair conformation. The crystal structure is stabilized by intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds.

Received 4 August 2006
Accepted 6 August 2006

Comment

The title compound, (I), is an important carbamate compound, which serves as an intermediate in the preparation of *N*-alicyclic isocyanates (Chen *et al.*, 2005). It was first reported in 1978 for the nitrosation reaction of 5-cyclohexyl- and 5-hexylhydantonic acids (Lutsenko *et al.*, 1978). Since then, its IR and proton NMR spectra (Hiegel & Hogenauer, 2005) and mass spectrum (Selva *et al.*, 2005) have been reported but, until now, its single-crystal structure has remained undetermined.



The molecular structure of (I) is shown in Fig. 1. The six-membered ring adopts a chair conformation. The $\text{C6}-\text{N1}-\text{C7}$ angle of $123.34(17)^\circ$ is suggestive of sp^2 hybridization for the N atom. An intermolecular hydrogen bond (Table 1) links the molecules into infinite chains (Fig. 2).

Experimental

Cyclohexylamine (10 ml) was reacted with dimethyl carbonate (75 ml) over an alkaline catalyst under atmospheric pressure. The reaction mixture was stirred under reflux for 3 h. The excess dimethyl carbonate was removed by vacuum evaporation at 298 K. The product was purified by column chromatography and recrystallization from dimethyl carbonate. Colourless prism-like single crystals of (I)

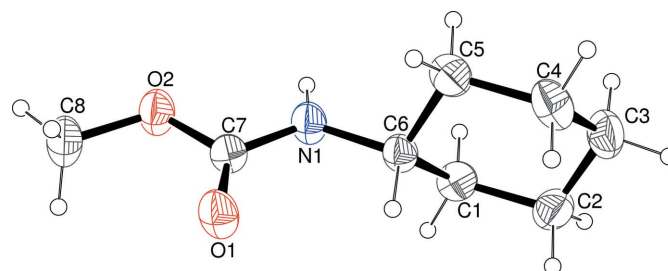


Figure 1
The molecular structure of (I), showing 30% displacement ellipsoids (arbitrary spheres for the H atoms).

were obtained by slow evaporation of a dimethyl carbonate solution at room temperature (m.p. 349.7 K).

Crystal data

$C_8H_{15}NO_2$ $Z = 4$
 $M_r = 157.21$ $D_x = 1.176 \text{ Mg m}^{-3}$
 Orthorhombic, $Pna2_1$ Mo $K\alpha$ radiation
 $a = 10.031 (2) \text{ \AA}$ $\mu = 0.08 \text{ mm}^{-1}$
 $b = 8.7863 (18) \text{ \AA}$ $T = 293 (2) \text{ K}$
 $c = 10.070 (2) \text{ \AA}$ Block, colorless
 $V = 887.6 (3) \text{ \AA}^3$ $0.26 \times 0.25 \times 0.18 \text{ mm}$

Data collection

Rigaku R-AXIS RAPID IP area-detector diffractometer 8264 measured reflections
 1079 independent reflections
 885 reflections with $I > 2\sigma(I)$
 ω scans $R_{int} = 0.032$
 Absorption correction: multi-scan (SADABS; Sheldrick, 1997) $\theta_{max} = 27.5^\circ$
 $T_{min} = 0.979, T_{max} = 0.985$

Refinement

Refinement on F^2 H-atom parameters constrained
 $R[F^2 > 2\sigma(F^2)] = 0.038$ $w = 1/[\sigma^2(F_o^2) + (0.0639P)^2]$
 $wR(F^2) = 0.105$ where $P = (F_o^2 + 2F_c^2)/3$
 $S = 1.16$ $(\Delta/\sigma)_{max} < 0.001$
 1079 reflections $\Delta\rho_{max} = 0.10 \text{ e \AA}^{-3}$
 100 parameters $\Delta\rho_{min} = -0.17 \text{ e \AA}^{-3}$

Table 1

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1A\cdots O1^i$	0.86	2.19	3.009 (2)	160

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

Anomalous scattering effects were negligible and Friedel equivalents were merged prior to the final refinement, thus the absolute configuration of the crystal used for data collection is indeterminate. H atoms were placed in calculated positions ($N-H = 0.86 \text{ \AA}$ and $C-H = 0.93-0.98 \text{ \AA}$) and refined as riding, with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(\text{methyl C})$.

Data collection: *RAPID-AUTO* (Rigaku, 2004); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics:

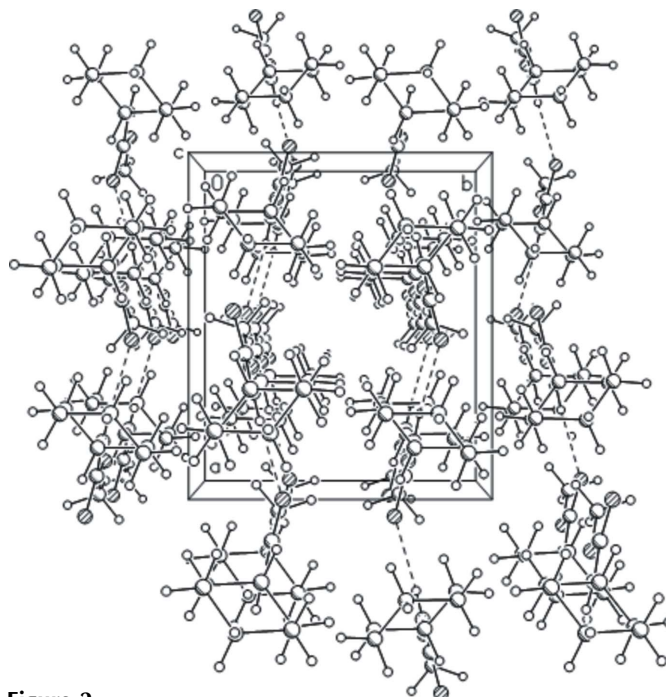


Figure 2

The molecular packing of (I), viewed along the c axis. Dashed lines indicate hydrogen bonds.

SHELXTL (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

The authors gratefully acknowledge support from Tianjin University.

References

Bruker (1998). *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
 Chen, Y., Zhao, X. & Wang, Y. (2005). *Acta Petrol. Ei Sin. (Petroleum Processing Section)*, **47**, 56–61.
 Hiegel, G. A. & Hogenauer, T. J. (2005). *Synth. Commun.* **35**, 2091–2098.
 Lutsenko, V. V., Stumbreviciute, Z. & Zimantiene, Z. (1978). *Zh. Org. Khim.* **14**, 1203–1209. (In Russian.)
 Rigaku (2004). *RAPID-AUTO* and *CrystalStructure*. Rigaku/MSC Inc., The Woodlands, Texas, USA.
 Selva, M., Tundo, P. & Perosa, A. (2005). *J. Org. Chem.* **70**, 2771–2777.
 Sheldrick, G. M. (1997). *SADABS, SHELXS97* and *SHELXL97*. University of Göttingen, Germany.