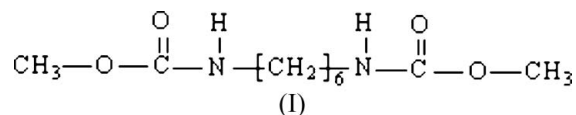


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Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
 R factor = 0.046
 wR factor = 0.127
Data-to-parameter ratio = 12.6For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Dimethyl *N,N'*-(hexane-1,6-diyl)dicarbamateThe molecule of the title compound, $\text{C}_{10}\text{H}_{20}\text{N}_2\text{O}_4$, lies on an inversion centre. The crystal structure is stabilized by intermolecular $\text{N}-\text{H}\cdots\text{O}$ classical hydrogen bonds, which pack the molecules along the short [100] axis.Received 20 October 2006
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Comment

The title molecule, (I), is an important carbamate compound, which serves as an intermediate in the preparation of *N*-aliphatic isocyanates (Wang *et al.*, 2006). It was first reported in 1967 for the synthesis of alkyl (6-isocyanatohexyl)-carbamates and dialkyl hexane-1,6-dicarbamates (Sinyavskii *et al.*, 1967). Since then, its ^1H NMR spectrum has been reported (Degner & Hannebaum, 1987) but, until now, its single-crystal structure has remained undetermined.The molecular structure of (I) is shown in Fig. 1. It exhibits a classical aliphatic chain, similar to that found in normal paraffin hydrocarbons, with an inversion centre located at the mid-point of the $\text{C}5-\text{C}5^i$ bond [symmetry code: (i) $1-x, -y, -z$]. The $\text{C}2-\text{N}1-\text{C}3$ angle of $120.59(12)^\circ$ is suggestive of sp^2 hybridization for the N atom. Intermolecular hydrogen bonding involving the amine NH groups as donor and the carbonyl O atoms as acceptor (Table 1) link the molecules into infinite chains (Fig. 2) running along the short [100] axis.

Experimental

Hexane-1,6-diamine (10 ml) was reacted with dimethyl carbonate (75 ml) over an alkaline catalyst at atmospheric pressure. The reaction mixture was refluxed for 3 h. The excess of dimethyl carbonate was removed by evaporation under reduced pressure at 298 K. The crude product was purified by column chromatography and recrystallized from dimethyl carbonate. Colourless flake-like single crystals of (I) were obtained by slow evaporation of a dimethyl carbonate solution at 298 K (m.p. 385.2 K).

Crystal data

 $\text{C}_{10}\text{H}_{20}\text{N}_2\text{O}_4$
 $M_r = 232.28$
Monoclinic, $P2_1/n$
 $a = 5.0290(10)$ Å
 $b = 23.674(5)$ Å
 $c = 5.2550(11)$ Å
 $\beta = 93.56(3)^\circ$
 $V = 624.4(2)$ Å³ $Z = 2$
 $D_x = 1.235$ Mg m⁻³
Mo $K\alpha$ radiation
 $\mu = 0.10$ mm⁻¹
 $T = 293(2)$ K
Plate, colourless
 $0.32 \times 0.22 \times 0.09$ mm

Data collection

Rigaku R-AXIS RAPID IP
diffractometer
 ω scans
Absorption correction: multi-scan
(*ABSCOR*; Higashi, 1995)
 $T_{\min} = 0.970$, $T_{\max} = 0.991$

6072 measured reflections
1432 independent reflections
1023 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.070$
 $\theta_{\text{max}} = 27.5^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.046$
 $wR(F^2) = 0.127$
 $S = 1.04$
1432 reflections
114 parameters
Only H-atom coordinates refined

$w = 1/[\sigma^2(F_o^2) + (0.0521P)^2 + 0.0675P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.13 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.16 \text{ e \AA}^{-3}$
Extinction correction: *SHELXL97*
Extinction coefficient: 0.055 (16)

Table 1

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1D\cdots O2^{\text{ii}}$	0.855 (19)	2.116 (19)	2.9664 (17)	172.9 (17)

Symmetry code: (ii) $x - 1, y, z$.

All H atoms were found in a difference map and refined with free coordinates. Isotropic displacement parameters for H atoms were fixed at $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{carrier atom})$ for methylene CH_2 and amine NH groups, and at $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for the methyl group. Methyl H atoms were found to be disordered over two sites, due to rotation about the $\text{O1}-\text{C1}$ σ bond. Their site occupancy factors were constrained to sum to unity and converged to 0.53 (4) and 0.47 (4).

Data collection: *RAPID-AUTO* (Rigaku/MSK, 2004); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSK, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997a); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997a); molecular graphics: *SHELXTL* (Sheldrick, 1997b); software used to prepare material for publication: *SHELXL97*.

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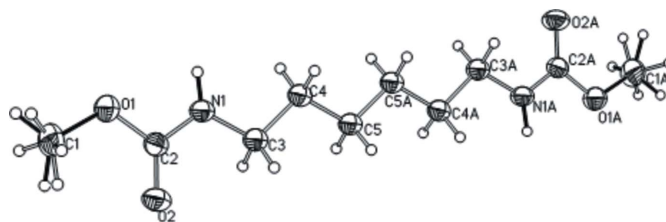


Figure 1

The molecular structure of (I), showing 30% displacement ellipsoids (arbitrary spheres for the H atoms). Both disorder components are shown. [Symmetry code: (A) $1 - x, -y, -z$.]

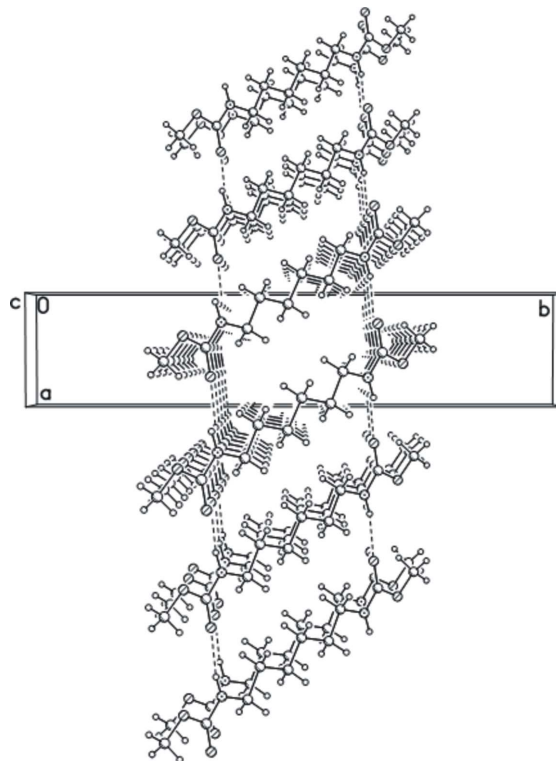


Figure 2

The molecular packing of (I), viewed along [001]. Dashed lines indicate hydrogen bonds.

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