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1,1-Dimethylethyl *N*-propanoylcarbamate

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The title compound, $C_8H_{15}NO_3$, has been obtained as a by-product of an epoxidation reaction. The molecules are linked by nearly symmetrical, bifurcated N—H···O hydrogen bonds, forming infinite chains parallel to the *c* axis.

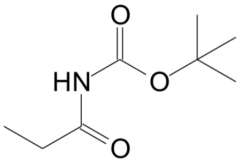
Key indicators

Single-crystal X-ray study
T = 123 K
Mean σ(C–C) = 0.003 Å
R factor = 0.044
wR factor = 0.111
Data-to-parameter ratio = 16.0

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

Comment

The title compound, (I), has been obtained in trace amounts as an unexpected by-product in the epoxidation of 1,1-dimethylethyl 1(*S*)-ethylprop-2-enylcarbamate with 3-chloroperoxybenzoic acid. It has been prepared previously by different routes (Tanaka *et al.*, 1988; Meffre *et al.*, 1996).



(I)

The molecular structure of (I) is shown in Fig. 1. The bond angles O1—C1—N1, O1—C1—C2, O2—C4—O3 and O2—C4—N1 are larger than 120° and range from 123.4 (2) to 126.3 (2)° (Table 1). The two carbonyl groups C1═O1 and C4═O2, as well as their attached atoms C2, N1, and O3, are nearly in a plane, as seen from torsion angles C4—N1—C1—C2 and C1—N1—C4—O3 (Table 1).

The molecular arrangement in the crystal structure is characterized by the presence of a nearly symmetrical bifur-

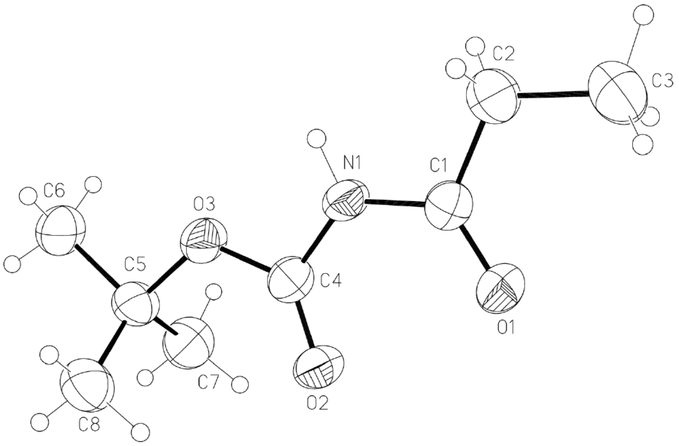


Figure 1
The molecular structure of (I), showing the atom-numbering scheme and displacement ellipsoids at the 50% probability level for the non-H atoms.

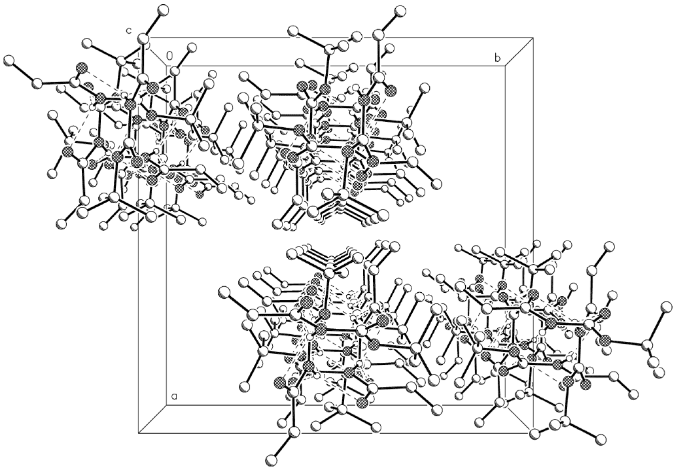


Figure 2
The crystal structure of (I) projected along the *c* axis, showing the packing of the infinite chains. H atoms have been omitted, except for the carbamate H atom.

cated intermolecular hydrogen bond (Table 2), forming infinite chains parallel to the *c* axis (Fig. 2). In these chains, four molecules build a repeating unit (Fig. 3). According to Steiner (2002), this hydrogen bond can be categorized as moderately strong. The crystal structure of a related compound, *viz.* 2(*S*)-*N*-*tert*-butoxycarbonylamino-*N*-methoxy-*N*-methylbutanamide (Sawatzki *et al.*, 2002), in which the amide carbonyl O atom of (I) is replaced by an *N*-methoxy-*N*-methylamide group, shows an entirely different network of hydrogen bonds.

Experimental

Glassware was flame-dried under an argon atmosphere and allowed to cool. The starting material, 1,1-dimethylethyl 1(*S*)-ethylprop-2-enylcarbamate, was prepared from 2(*S*)-*N*-*tert*-butoxycarbonylamino-*N*-methoxy-*N*-methylbutanamide (Sibi, 1993; Sawatzki *et al.*, 2002). In brief, this Weinreb amide was reduced to the aldehyde by lithium aluminium hydride, followed by a Wittig reaction with methylene triphenylphosphoranylidene to the starting material according to a published procedure (Campbell *et al.*, 1998). The starting material (4.77 g, 25.8 mmol) was dissolved in a mixture of dichloromethane (260 ml) and a 0.5 *M* aqueous solution of sodium hydrogencarbonate (240 ml). After cooling to 273 K, 70% 3-chloroperoxybenzoic acid (*m*CPBA) (19.08 g, 77.4 mmol) was added in small portions. The reaction mixture was stirred at room temperature for 1 h, cooled to 273 K again, and 70% *m*CPBA (19.08 g, 77.4 mmol) was added. After stirring for another hour, the same amount of *m*CPBA was added and stirring was continued overnight. The organic and the aqueous phases were separated. The organic phase was subsequently washed with a 1 *M* solution of sodium hydroxide, with water, and dried over sodium sulfate. After evaporation of the solvent, the resulting residue was purified by column chromatography on silica gel using hexane/ethyl acetate (5:1) as eluent. The major product was separated, and one fraction containing a substance eluting with $R_F = 0.23$ was further analysed. On slow removal of the solvent, a few colourless crystals of (I) were obtained, which were suitable for X-ray analysis. Spectroscopic data were in accordance with literature data (Tanaka *et al.*, 1988; Meffre *et al.*, 1996).

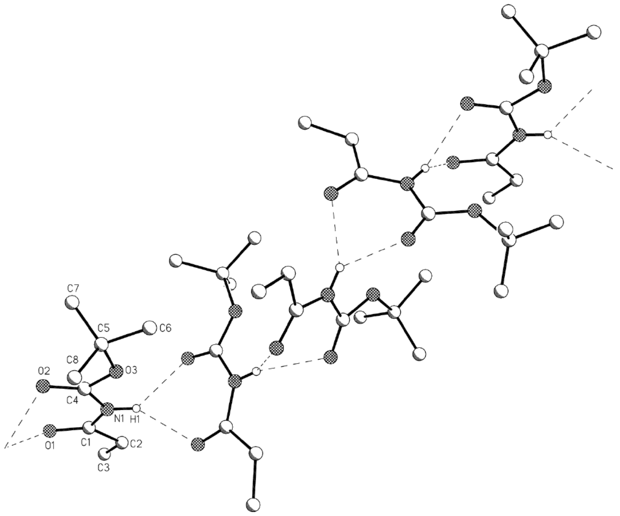


Figure 3
Enlarged excerpt of the crystal packing, showing that four molecules constitute a repeating unit. H atoms have been omitted, except for the carbamate H atom.

Crystal data

$C_8H_{15}NO_3$
$M_r = 173.21$
Tetragonal, $I4_1/a$
$a = 16.5136$ (9) Å
$c = 14.9106$ (8) Å
$V = 4066.1$ (4) Å^3
$Z = 16$
$D_x = 1.132$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 4015 reflections
$\theta = 1$–25°
$\mu = 0.09$ mm^{-1}
$T = 123$ (2) K
Plate, colourless
0.30 × 0.10 × 0.05 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans with 1° frames
Absorption correction: none
13978 measured reflections
1792 independent reflections
928 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.078$
$\theta_{\rm max} = 25.0°$
$h = -19 \rightarrow 12$
$k = -19 \rightarrow 19$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.111$
$S = 0.89$
1792 reflections
112 parameters

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0547P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.16$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.15$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.212 (2)	N1—C1	1.376 (3)
O2—C4	1.207 (2)	N1—C4	1.377 (3)
O3—C4	1.343 (2)	C1—C2	1.496 (3)
O1—C1—N1	123.5 (2)	O2—C4—O3	126.3 (2)
O1—C1—C2	123.4 (2)	O2—C4—N1	125.7 (2)
N1—C1—C2	113.1 (2)	O3—C4—N1	108.03 (19)
C4—N1—C1—O1	−3.9 (4)	C1—N1—C4—O2	3.3 (4)
C4—N1—C1—C2	176.0 (2)	C1—N1—C4—O3	−176.36 (19)
O1—C1—C2—C3	10.4 (3)	C4—O3—C5—C8	66.2 (2)
N1—C1—C2—C3	−169.5 (2)	C4—O3—C5—C7	−58.9 (3)
C5—O3—C4—O2	−0.7 (3)	C4—O3—C5—C6	−176.10 (19)
C5—O3—C4—N1	178.97 (17)		

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

$D-\mathrm{H}\cdots A$	$D-\mathrm{H}$	$\mathrm{H}\cdots A$	$D\cdots A$	$D-\mathrm{H}\cdots A$
$\mathrm{N1-H1}\cdots\mathrm{O1}^{\mathrm{i}}$	0.87 (2)	2.18 (2)	2.943 (2)	146.8 (19)
$\mathrm{N1-H1}\cdots\mathrm{O2}^{\mathrm{i}}$	0.87 (2)	2.26 (2)	2.933 (2)	134.8 (18)

Symmetry code: (i) $\frac{5}{4}-y, \frac{3}{4}+x, z-\frac{1}{4}$.

The carbamate H atom was located in a difference Fourier map and the coordinates were refined freely, with $U_{\mathrm{iso}}(\mathrm{H}) = 1.2U_{\mathrm{eq}}(\mathrm{N})$. Other H atoms were treated as riding, with C—H = 0.98–0.99 Å and $U_{\mathrm{iso}}(\mathrm{H}) = 1.2U_{\mathrm{eq}}(\mathrm{CH_2})$ or $1.5U_{\mathrm{eq}}(\mathrm{CH_3})$.

Data collection: *COLLECT* (Nonius, 1997–2000); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL-NT* (Sheldrick, 2001); software used to prepare material for publication: *SHELXL*97.

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