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Key indicators

Single-crystal X-ray study
 $T = 120$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.035
 wR factor = 0.078
Data-to-parameter ratio = 9.9For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.**(1*R**,2*R**)-Di-*tert*-butyl *N,N'*-(cyclohexane-1,2-diyl)-
dicarbamate**

The title compound, $\text{C}_{16}\text{H}_{30}\text{N}_2\text{O}_4$, was synthesized as part of ongoing studies into enantioselective recognition. The molecule sits on a twofold axis and forms ladders *via* $\text{N}-\text{H}\cdots\text{O}$ hydrogen-bond pairs.

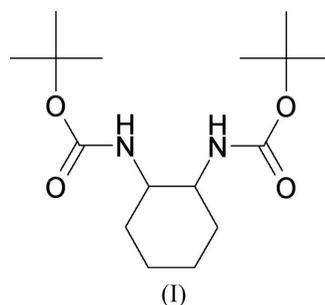
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Comment

(1*R**,2*R**)-Di-*tert*-butyl *N,N'*-(cyclohexane-1,2-diyl)dicarbamate, (I), was synthesized as part of our ongoing studies into enantioselective recognition (Botana *et al.*, 2001; Rossi *et al.*, 2002; Kyne *et al.*, 2001). The synthesis of new chiral receptors is a major challenge for chemists since it is very difficult to predict all the factors contributing to the binding process between a host and a guest in solution (Beer *et al.*, 1999). Furthermore, the use of cheap and readily available building blocks for the construction of enantioselective receptors is of fundamental importance from an industrial point of view. To that aim, compound (I), with its two chiral centres and its amidic H atoms, is an appealing intermediate for the synthesis of more complicated structures, which may be able to discriminate between two enantiomers of a racemic mixture.



In the crystal structure, the molecule is disposed about a twofold crystallographic axis. The cyclohexane ring adopts a chair conformation, with methylcarbamic acid *tert*-butyl ester groups hanging down below to form a V-shaped molecule in which the NH groups point in opposite directions. This arrangement aids the formation of hydrogen-bonded ladders (Fig.2) that extend along the *c* direction *via* $\text{N}-\text{H}\cdots\text{O}$ hydrogen-bond pairs. When viewed down the *c* axis, the hydrogen-bonded ladders arrange themselves in a close-packed manner such that the 'Vs' line up, all pointing in the same direction (Fig. 3).

Experimental

(1*S*,2*S*)-1,2-Diphenyl-1,2-ethylenediamine-L-tartaric acid (1.6 g, 4.41 mmol) was dissolved in 1*M* K_2CO_3 (20 ml). A solution of di-*tert*-

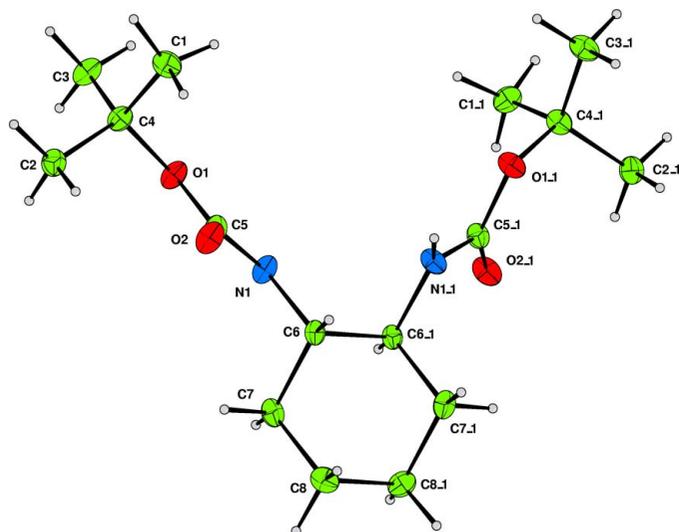


Figure 1
View of the structure of (I), showing the atomic numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 35% probability level, and H atoms are drawn with arbitrary radii. [Symmetry code: ($_1$) $-x + 1, y, -z$.]

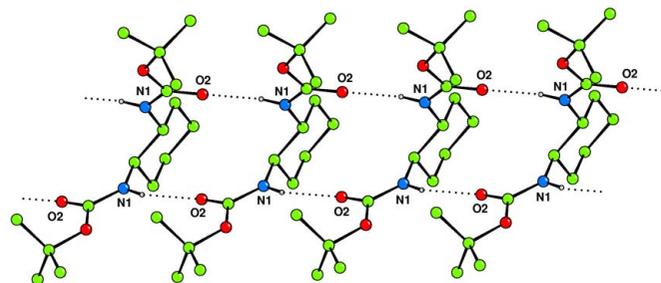


Figure 2
Part of one of the hydrogen-bonded ladders extending along c . Hydrogen bonds are shown as dotted lines. Only those H atoms involved in classical hydrogen bonds have been included.

butyl dicarbonate (2.77 g, 12.7 mmol) in ethanol (40 ml) was added and the mixture was stirred at room temperature for 17 h. The solvents were removed *in vacuo* and the residue was dissolved in water to yield the product as a pale-yellow precipitate (1.3 g, 94%). The crystal for structure determination was obtained by slow evaporation of a 0.05 mM solution of the product in dimethyl sulfoxide (DMSO, 1 ml). M.p. 493–495 K. ^1H NMR (400 MHz, DMSO- d_6): δ 7.71 (2H, *m*, NH), 3.62 (2H, *m*, CH), 1.81 (2H, *m*, CHHCH), 1.66 (2H, *m*, CHHCH), 1.24 (18H, *s*, CH_3), 1.17 (4H, *m*, $\text{CH}_2\text{CH}_2\text{CH}$); ^{13}C NMR (100 MHz, DMSO- d_6): δ 155.2 (0), 78.2 (0), 52.3 (1), 31.6 (2), 28.3 (3), 24.2 (2); m/z (ES^+) 337.2 [$M+\text{Na}$] $^+$; HRMS (ES^+) Calculated for $\text{C}_{16}\text{H}_{31}\text{N}_2\text{O}_4^+$: 315.2278; found: 315.2282. Analysis calculated for $\text{C}_{16}\text{H}_{30}\text{N}_2\text{O}_4$: C 61.12, H 9.62, N 8.91%; found: C 61.12, H 9.64, N 8.98%.

Crystal data

$\text{C}_{16}\text{H}_{30}\text{N}_2\text{O}_4$
 $M_r = 314.42$
Monoclinic, $C2$
 $a = 18.856$ (4) Å
 $b = 9.3110$ (19) Å
 $c = 5.183$ (1) Å
 $\beta = 101.04$ (3)°
 $V = 893.1$ (3) Å 3
 $Z = 2$

$D_x = 1.169$ Mg m $^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 982 reflections
 $\theta = 2.9$ – 27.5°
 $\mu = 0.08$ mm $^{-1}$
 $T = 120$ (2) K
Slab, pale yellow
0.20 × 0.12 × 0.03 mm

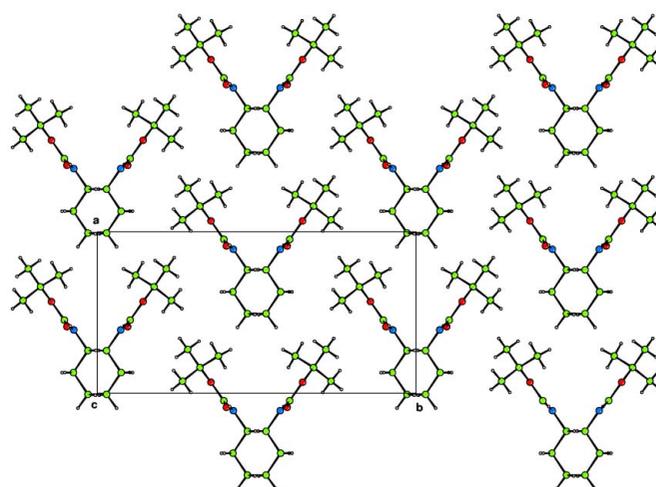


Figure 3
A packing diagram viewed down c , showing the arrangement of the V-shaped molecules.

Data collection

Nonius KappaCCD diffractometer
 φ and ω scans
Absorption correction: multi-scan
(*SADABS*; Sheldrick, 2003)
 $T_{\min} = 0.984$, $T_{\max} = 0.998$
3861 measured reflections
1069 independent reflections

966 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.045$
 $\theta_{\text{max}} = 27.5^\circ$
 $h = -23 \rightarrow 24$
 $k = -12 \rightarrow 12$
 $l = -6 \rightarrow 6$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.035$
 $wR(F^2) = 0.078$
 $S = 1.06$
1069 reflections
108 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0274P)^2 + 0.2768P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.006$
 $\Delta\rho_{\text{max}} = 0.16$ e Å $^{-3}$
 $\Delta\rho_{\text{min}} = -0.15$ e Å $^{-3}$
Extinction correction: *SHELXL97*
Extinction coefficient: 0.018 (5)

Table 1

Hydrogen-bond geometry (Å, °).

$D\text{---}H\cdots A$	$D\text{---}H$	$H\cdots A$	$D\cdots A$	$D\text{---}H\cdots A$
$\text{N1---H99}\cdots\text{O2}^i$	0.84 (2)	2.20 (3)	2.996 (2)	160 (2)

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

In the absence of significant anomalous dispersion effects, Friedel pairs were merged. All C-bound H atoms were located in a difference Fourier map, and were placed in calculated positions and treated as riding on their parent atoms, with $\text{C---H} = 0.98$ Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for CH_3 , $\text{C---H} = 0.99$ Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for CH_2 , and $\text{C---H} = 1.00$ Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for CH. The single H atom on the N atom was freely refined.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *CAMERON* (Watkin *et al.*, 1993); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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supporting information

Acta Cryst. (2005). E61, o1956–o1958 [https://doi.org/10.1107/S1600536805016715]

(1*R,2*R**)-Di-*tert*-butyl *N,N'*-(cyclohexane-1,2-diyl)dicarbamate**

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(1*R,2*R**)-Di-*tert*-butyl *N,N'*-(cyclohexane-1,2-diyl)dicarbamate***Crystal data*

$C_{16}H_{30}N_2O_4$

$M_r = 314.42$

Monoclinic, *C*2

Hall symbol: C 2y

$a = 18.856$ (4) Å

$b = 9.3110$ (19) Å

$c = 5.183$ (1) Å

$\beta = 101.04$ (3)°

$V = 893.1$ (3) Å³

$Z = 2$

$F(000) = 344$

$D_x = 1.169$ Mg m⁻³

Mo *K*α radiation, $\lambda = 0.71073$ Å

Cell parameters from 982 reflections

$\theta = 2.9$ – 27.5 °

$\mu = 0.08$ mm⁻¹

$T = 120$ K

Slab, pale yellow

$0.20 \times 0.12 \times 0.03$ mm

Data collection

Nonius KappaCCD
diffractometer

Radiation source: Bruker Nonius FR591
Rotating Anode

10cm confocal mirrors monochromator

Detector resolution: 9.091 pixels mm⁻¹

φ and ω scans

Absorption correction: multi-scan
(SADABS; Sheldrick, 2003)

$T_{\min} = 0.984$, $T_{\max} = 0.998$

3861 measured reflections

1069 independent reflections

966 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.045$

$\theta_{\max} = 27.5$ °, $\theta_{\min} = 4.0$ °

$h = -23$ → 24

$k = -12$ → 12

$l = -6$ → 6

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.035$

$wR(F^2) = 0.078$

$S = 1.06$

1069 reflections

108 parameters

1 restraint

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: inferred from
neighbouring sites

H atoms treated by a mixture of independent
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0274P)^2 + 0.2768P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.006$

$\Delta\rho_{\max} = 0.16$ e Å⁻³

$\Delta\rho_{\min} = -0.15$ e Å⁻³

Extinction correction: SHELXL97,

$F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.018 (5)

Special details

Experimental. *SADABS* was used to perform the Absorption correction Parameter refinement on 3374 reflections reduced $R(\text{int})$ from 0.1021 to 0.0430 Ratio of minimum to maximum apparent transmission: 0.825366 The given T_{\min} and T_{\max} were generated using the *SHELX SIZE* command

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.37974 (12)	−0.2307 (2)	0.5011 (4)	0.0317 (5)
H1A	0.4177	−0.2702	0.4166	0.048*
H1B	0.3579	−0.3083	0.5870	0.048*
H1C	0.4007	−0.1593	0.6326	0.048*
C2	0.26751 (12)	−0.0748 (3)	0.4139 (4)	0.0297 (5)
H2A	0.2930	−0.0054	0.5409	0.045*
H2B	0.2396	−0.1406	0.5032	0.045*
H2C	0.2348	−0.0235	0.2744	0.045*
C3	0.28359 (12)	−0.2712 (3)	0.1034 (4)	0.0319 (5)
H3A	0.2484	−0.2233	−0.0331	0.048*
H3B	0.2586	−0.3398	0.1981	0.048*
H3C	0.3190	−0.3222	0.0218	0.048*
C4	0.32203 (11)	−0.1598 (2)	0.2946 (4)	0.0249 (5)
C5	0.39673 (10)	0.0463 (2)	0.2217 (4)	0.0237 (4)
C6	0.47084 (9)	0.2357 (2)	0.0855 (3)	0.0211 (4)
H6	0.4955	0.2347	0.2743	0.025*
C7	0.42526 (11)	0.3717 (2)	0.0363 (4)	0.0281 (5)
H7A	0.3983	0.3715	−0.1474	0.034*
H7B	0.3896	0.3722	0.1537	0.034*
C8	0.47087 (12)	0.5068 (2)	0.0837 (4)	0.0297 (5)
H8A	0.4938	0.5128	0.2719	0.036*
H8B	0.4395	0.5922	0.0400	0.036*
N1	0.42652 (9)	0.1078 (2)	0.0353 (3)	0.0252 (4)
O1	0.35630 (8)	−0.06813 (17)	0.1227 (2)	0.0271 (4)
O2	0.40471 (8)	0.08750 (17)	0.4484 (3)	0.0318 (4)
H99	0.4150 (12)	0.083 (3)	−0.122 (5)	0.028 (6)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0364 (12)	0.0310 (12)	0.0280 (11)	0.0049 (10)	0.0067 (9)	−0.0021 (9)
C2	0.0277 (11)	0.0334 (11)	0.0292 (11)	−0.0006 (10)	0.0080 (8)	0.0014 (10)
C3	0.0383 (12)	0.0309 (11)	0.0274 (11)	−0.0104 (11)	0.0083 (8)	−0.0001 (9)
C4	0.0288 (10)	0.0275 (11)	0.0190 (10)	−0.0042 (9)	0.0057 (7)	0.0022 (8)
C5	0.0210 (10)	0.0296 (11)	0.0198 (10)	−0.0030 (9)	0.0021 (7)	−0.0024 (8)
C6	0.0179 (9)	0.0266 (10)	0.0186 (10)	−0.0009 (9)	0.0033 (7)	−0.0012 (8)
C7	0.0235 (10)	0.0360 (12)	0.0257 (10)	0.0038 (10)	0.0070 (8)	−0.0008 (9)

C8	0.0354 (12)	0.0293 (12)	0.0244 (11)	0.0050 (10)	0.0060 (9)	-0.0013 (9)
N1	0.0284 (9)	0.0315 (10)	0.0163 (9)	-0.0098 (8)	0.0056 (7)	-0.0051 (7)
O1	0.0333 (8)	0.0314 (8)	0.0179 (7)	-0.0104 (7)	0.0080 (6)	-0.0025 (6)
O2	0.0373 (8)	0.0411 (9)	0.0177 (7)	-0.0125 (8)	0.0072 (6)	-0.0062 (6)

Geometric parameters (Å, °)

C1—C4	1.523 (3)	C5—N1	1.336 (3)
C1—H1A	0.9800	C5—O1	1.353 (2)
C1—H1B	0.9800	C6—N1	1.450 (3)
C1—H1C	0.9800	C6—C7	1.524 (3)
C2—C4	1.519 (3)	C6—C6 ⁱ	1.539 (4)
C2—H2A	0.9800	C6—H6	1.0000
C2—H2B	0.9800	C7—C8	1.517 (3)
C2—H2C	0.9800	C7—H7A	0.9900
C3—C4	1.519 (3)	C7—H7B	0.9900
C3—H3A	0.9800	C8—C8 ⁱ	1.525 (4)
C3—H3B	0.9800	C8—H8A	0.9900
C3—H3C	0.9800	C8—H8B	0.9900
C4—O1	1.470 (2)	N1—H99	0.84 (2)
C5—O2	1.218 (2)		
C4—C1—H1A	109.5	O2—C5—O1	124.81 (18)
C4—C1—H1B	109.5	N1—C5—O1	110.32 (16)
H1A—C1—H1B	109.5	N1—C6—C7	111.44 (14)
C4—C1—H1C	109.5	N1—C6—C6 ⁱ	110.33 (13)
H1A—C1—H1C	109.5	C7—C6—C6 ⁱ	110.22 (12)
H1B—C1—H1C	109.5	N1—C6—H6	108.3
C4—C2—H2A	109.5	C7—C6—H6	108.3
C4—C2—H2B	109.5	C6 ⁱ —C6—H6	108.3
H2A—C2—H2B	109.5	C8—C7—C6	112.21 (16)
C4—C2—H2C	109.5	C8—C7—H7A	109.2
H2A—C2—H2C	109.5	C6—C7—H7A	109.2
H2B—C2—H2C	109.5	C8—C7—H7B	109.2
C4—C3—H3A	109.5	C6—C7—H7B	109.2
C4—C3—H3B	109.5	H7A—C7—H7B	107.9
H3A—C3—H3B	109.5	C7—C8—C8 ⁱ	110.84 (14)
C4—C3—H3C	109.5	C7—C8—H8A	109.5
H3A—C3—H3C	109.5	C8 ⁱ —C8—H8A	109.5
H3B—C3—H3C	109.5	C7—C8—H8B	109.5
O1—C4—C2	110.78 (16)	C8 ⁱ —C8—H8B	109.5
O1—C4—C3	102.18 (15)	H8A—C8—H8B	108.1
C2—C4—C3	110.21 (17)	C5—N1—C6	121.99 (17)
O1—C4—C1	109.87 (16)	C5—N1—H99	121.2 (16)
C2—C4—C1	112.82 (16)	C6—N1—H99	116.1 (17)

C3—C4—C1	110.47 (18)	C5—O1—C4	120.55 (14)
O2—C5—N1	124.87 (19)		

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

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

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H99 \cdots O2 ⁱⁱ	0.84 (2)	2.20 (3)	2.996 (2)	160 (2)

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