Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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

Mark E. Light,* Andrea Ragusa and Jeremy D. Kilburn

School of Chemistry, University of Southampton, Highfield, Southampton SO17 1BJ, England

Correspondence e-mail: light@soton.ac.uk

Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.003 Å
R factor = 0.035
wR factor = 0.078
Data-to-parameter ratio = 9.9

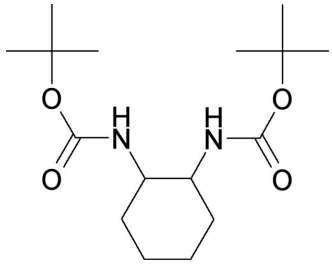
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 16 May 2005
Accepted 25 May 2005
Online 31 May 2005

The title compound, $C_{16}H_{30}N_2O_4$, was synthesized as part of ongoing studies into enantioselective recognition. The molecule sits on a twofold axis and forms ladders *via* N—H···O hydrogen-bond pairs.

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.



(I)

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* N—H···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*-

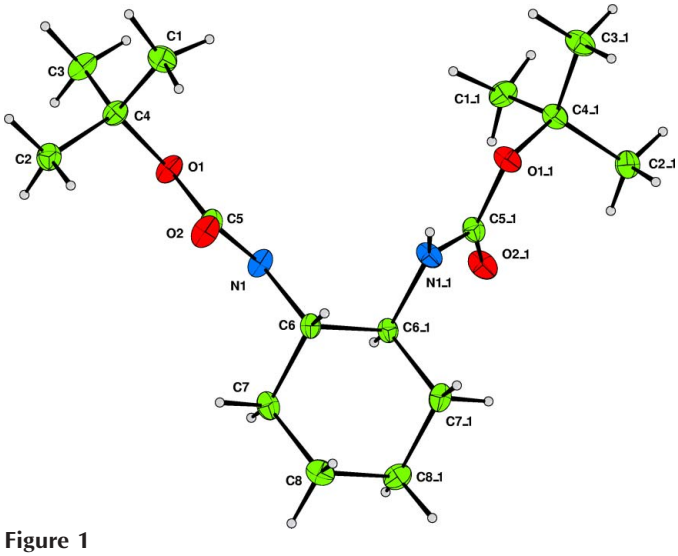


Figure 1
View of the structure of (I), showing the atomic numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 35% probability level, and H atoms are drawn with arbitrary radii. [Symmetry code: (_1) $-x+1, y, -z$.]

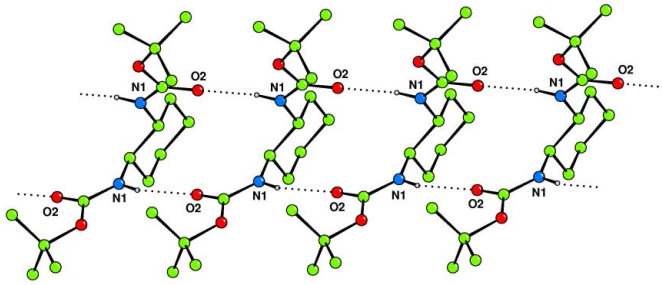


Figure 2
Part of one of the hydrogen-bonded ladders extending along *c*. Hydrogen bonds are shown as dotted lines. Only those H atoms involved in classical hydrogen bonds have been included.

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 m*M* solution of the product in dimethyl sulfoxide (DMSO, 1 ml). M.p. 493–495 K. ^{1}H 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*, CH_2CH_2CH); ^{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 $[M+Na]^+$; HRMS (ES^+) Calculated for $C_{16}H_{31}N_2O_4^+$: 315.2278; found: 315.2282. Analysis calculated for $C_{16}H_{30}N_2O_4$: C 61.12, H 9.62, N 8.91%; found: C 61.12, H 9.64, N 8.98%.

Crystal data

$C_{16}H_{30}N_2O_4$
M_r = 314.42
Monoclinic, *C*2
a = 18.856 (4) Å
b = 9.3110 (19) Å
c = 5.183 (1) Å
β = 101.04 (3)°
V = 893.1 (3) Å^3
Z = 2
D_x = 1.169 Mg m^{-3}
Mo *K*α radiation
Cell parameters from 982 reflections
θ = 2.9–27.5°
μ = 0.08 mm^{-1}
T = 120 (2) K
Slab, pale yellow
0.20 × 0.12 × 0.03 mm

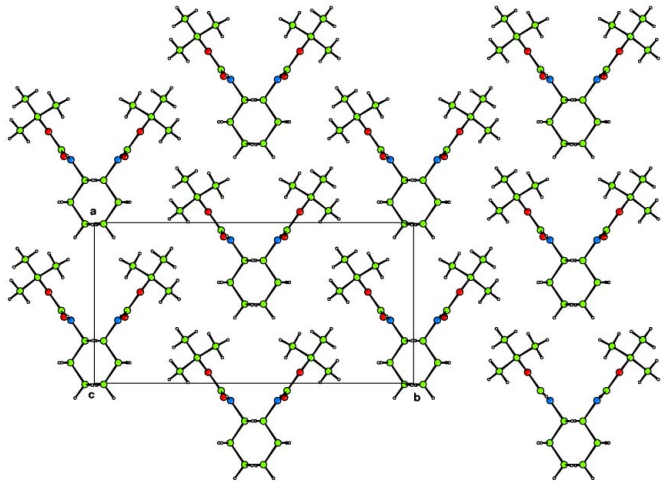


Figure 3
A packing diagram viewed down *c*, showing the arrangement of the V-shaped molecules.

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_{int} = 0.045
θ_{max} = 27.5°
h = −23 → 24
k = −12 → 12
l = −6 → 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 $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.006
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.018 (5)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H99···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 C—H = 0.98 Å and U_{iso}(H) = 1.5U_{eq}(C) for CH_3, C—H = 0.99 Å and U_{iso}(H) = 1.2U_{eq}(C) for CH_2, and C—H = 1.00 Å and U_{iso}(H) = 1.2U_{eq}(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).

The authors thank the EPSRC for funding the crystallographic facilities.

References

Beer, P. D., Gale, P. A. & Smith, D. K. (1999). *Supramolecular Chemistry*. Oxford University Press.
Botana, E., Ongeri, S., Arienzo, R., Demarcus, M., Frey, J. G., Piarulli, U., Potenza, D., Gennari, C. & Kilburn, J. D. (2001). *Chem. Commun.* **15**, 1358–1359.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Hooft, R. (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
Kyne, G. M., Light, M. E., Hursthouse, M. B., de Mendoza, J. & Kilburn, J. D. (2001). *J. Chem. Soc. Perkin Trans. 1*, pp. 1258–1263.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Rossi, S., Kyne, G. M., Turner, D. L., Wells, N. J. & Kilburn, J. D. (2002). *Angew. Chem. Int. Ed.* **41**, 4233–4236.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (2003). *SADABS*. Version 2.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Watkin, D. M., Pearce, L. & Prout, C. K. (1993). *CAMERON*. Chemical Crystallography Laboratory, University of Oxford, England.