Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N,N′-Disuccinimidyl carbonate

Monika Simon,[a] Carol Csunderlik,[a] Peter G. Jones,[b]* Ion Neda[c] and Andreea Tarta-Micle[a]

[a]Department of Organic Chemistry, Polytechnical University of Timisoara, Bocsa 6, 1900 Timisoara, Romania, [b]Institut für Anorganische und Analytische Chemie, Technische Universität Braunschweig, Postfach 3329, 38023 Braunschweig, Germany, and [c]InnoChemTech GmbH, Hagenring 30, 38106 Braunschweig, Germany

Correspondence e-mail: p.jones@tu-bs.de

Key indicators

Single-crystal X-ray study
T = 133 K
Mean σ(C–C) = 0.002 Å
R factor = 0.027
wR factor = 0.073
Data-to-parameter ratio = 9.3

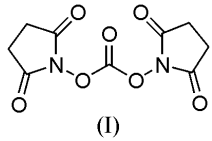
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 4 April 2003
Accepted 7 April 2003
Online 23 April 2003

The title compound, $C_9H_8N_2O_7$, possesses crystallographic twofold symmetry and displays an *s-cis–s-cis* conformation of the carbonate group. Bond lengths and angles (Å and °) involving this group are: C═O 1.175 (3), C—O 1.3669 (17), O—N 1.3876 (15); O═C—O 127.67 (8) and O—C—O 104.67 (17). The succinimide ring subtends an angle of 73.92 (4)° with the carbonate plane.

Comment

We are interested in the structures and synthetic applications of organic carbonates and have recently published the structures of bis(*o*-nitrophenyl) carbonate and bis(*p*-nitrophenyl) carbonate (Simon *et al.*, 2003*a*,*b*). Introductory material is presented in the first of these publications. Here we present the structure of *N,N′*-disuccinimidyl carbonate, (I), a versatile reagent for the preparation of active esters in peptide chemistry (Pearson & Roush, 2001; Ogura *et al.*, 1979; Gooßen & Ghosh, 2001), and a substitute for phosgene in reactions with various nucleophiles (Takeda & Ogura, 1982; Halstrøm & Kovács, 1986; Ghosh *et al.*, 1992; Boeden *et al.*, 1998).



(I)

The molecule of (I) is shown in Fig. 1. It displays crystallographic twofold symmetry, with the twofold axis (symmetry code: $-x$, $1-y$, z) passing along the bond C5═O4. The carbonate moiety displays the usual *s-cis–s-cis* conformation (*cf.* torsion angles in Table 1). The N atom is essentially coplanar with the carbonate moiety, lying only 0.0174 (3) Å out of the exact carbonate plane. The succinimide ring is almost planar (r.m.s. deviation 0.040 Å) and subtends an interplanar angle of 73.92 (4)° to the carbonate group.

Bond lengths and angles of the carbonate moiety (Table 1) may be considered normal. A search of the Cambridge Structural Database (Allen, 2002) revealed no hit for the moiety N—O—C(═O)—O—N; our earlier paper (Simon *et al.*, 2003*a*) briefly presents database results for diaryl carbonates.

The molecular packing involves two weak C—H···O hydrogen bonds (Table 2), the effect of which is to link the molecules to form two interpenetrating networks, one of which is shown in Fig. 2. Short intermolecular contacts O1···C4^{i} [2.991 (2) Å; symmetry code: (i) $-\frac{1}{4}+x$, $\frac{5}{4}-y$, $-\frac{1}{4}+z$] and O1···O4^{ii} [2.975 (2) Å; symmetry code: (ii) $\frac{1}{4}+x$, $\frac{5}{4}-y$, $\frac{1}{4}+z$] are also observed. These are not shown in Fig. 2.

Experimental

Triphosgene (bis(trichloromethyl) carbonate) was treated with six equivalents of *N*-hydroxysuccinimide and six equivalents of tri-*n*-butylamine in tetrahydrofuran (Pereira *et al.*, 1998). The product was recrystallized from acetonitrile.

Crystal data

$C_9H_8N_2O_7$
$M_r = 256.17$
Orthorhombic, *Fdd*2
$a = 13.239$ (2) Å
$b = 14.629$ (2) Å
$c = 10.3595$ (12) Å
$V = 2006.3$ (5) Å^3
$Z = 8$
$D_x = 1.696$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2400 reflections
$\theta = 2.8$–$30.5°$
$\mu = 0.15$ mm^{-1}
$T = 133$ (2) K
Flattened octahedron, colourless
0.3 × 0.3 × 0.2 mm

Data collection

Bruker SMART 1000 CCD diffractometer
ω scans
Absorption correction: none
3941 measured reflections
773 independent reflections
723 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 30.0°$
$h = -9 \rightarrow 18$
$k = -20 \rightarrow 20$
$l = -13 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.027$
$wR(F^2) = 0.073$
$S = 1.06$
773 reflections
83 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0522P)^2 + 0.2064P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—O1	1.2058 (19)	C5—O3	1.3669 (17)
C1—N	1.3882 (18)	N—O3	1.3876 (15)
C5—O4	1.175 (3)		
O4—C5—O3	127.67 (8)	O3—N—C4	121.08 (12)
O3—C5—O3^{iii}	104.67 (17)	C1—N—C4	116.95 (12)
O3—N—C1	121.53 (11)	C5—O3—N	109.27 (11)
O3^{iii}—C5—O3—N	−179.24 (12)	C1—N—O3—C5	69.98 (15)

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

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2A···O1^{iv}	0.99	2.60	3.5334 (19)	156
C2—H2B···O2^{v}	0.99	2.47	3.429 (2)	164

Symmetry codes: (iv) $\frac{1}{2}-x, \frac{3}{2}-y, z$; (v) $\frac{1}{2}-x, 1-y, z-\frac{1}{2}$.

H atoms were included using a riding model, with fixed C—H bond lengths of 0.99 Å; U_{iso}(H) values were fixed at 1.2 times U_{eq} of the parent atom. The anomalous scattering was not sufficient to determine the absolute structure; Friedel opposite reflections were therefore merged.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL*97.

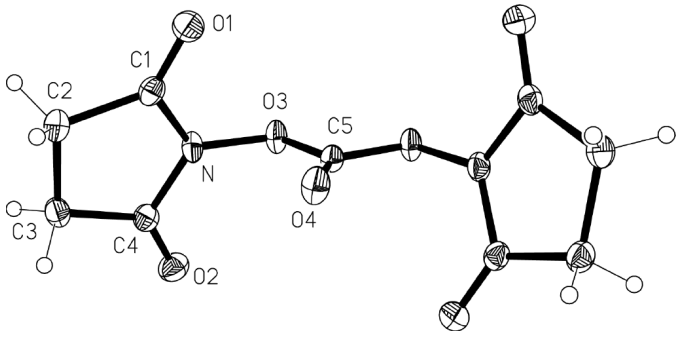


Figure 1
The molecule of the title compound in the crystal. Ellipsoids are shown at the the 50% probability level and H-atom radii are arbitrary.

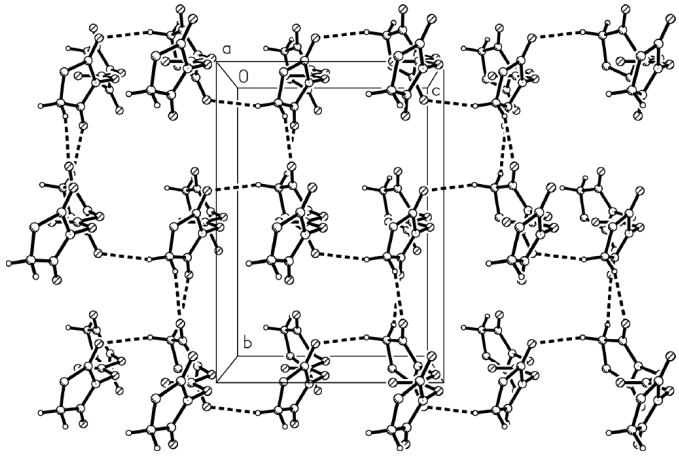


Figure 2
One of two interpenetrating networks of the title compound in the crystal. Hydrogen bonds are indicated by dashed lines. H atoms not involved in hydrogen bonding have been omitted.

Financial support from the Fonds der Chemischen Industrie and the Romanian Ministry of Education and Research, CNCSIS (grant D,19) is gratefully acknowledged. We thank Mr A. Weinkauf for technical assistance.

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Boeden, N., Bushby, R. J., Liu, Q., Evans, S. D., Jenkins, T. A., Knowles, P. F. & Miles, R. E. (1998). *Tetrahedron*, **54**, 11537–11548.
Bruker (1998). *SMART* (Version 5.0) and *SAINT* (Version 4.0). Bruker AXS Inc., Madison, Wisconsin, USA.
Ghosh, A. K., Duong, T. T., McKee, S. P. & Thompson, W. J. (1992). *Tetrahedron Lett.* **33**, 2781–2784.
Gooßen, L. J. & Ghosh, A. K. (2001). *Chem. Commun.* pp. 2084–2085.
Halstrøm, J. & Kovács, K. (1986). *Acta Chem. Scand. Ser. B*, **40**, 462–464.
Ogura, H., Kobayashi, T., Shimizu, K., Kawabe, K. & Takeda, K. (1979). *Tetrahedron Lett.* **49**, 4745–4749.
Pearson, A. J. & Roush, W. R. (2001). *Handbook of Reagents for Organic Synthesis. Activating Agents and Protecting Groups*, pp. 173–174. Chichester: John Wiley and Sons.
Pereira, D., That Hai, T. & Nelson, D. (1998). *Synth. Commun.* **28**, 4019–4024.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.

Siemens (1994). *XP*. Version 5.03. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Simon, M., Csunderlik, C., Jones, P. G., Neda, I. & Fischer, A. K. (2003*a*). *Acta Cryst.* E**59**, o688–o690.
Simon, M., Csunderlik, C., Jones, P. G., Neda, I. & Fischer, A. K. (2003*b*). *Acta Cryst.* E**59**, o691–o692.
Takeda, K. & Ogura, H. (1982). *Synth. Commun.* **12**, 213–217.