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# *N,N′*-Carbonyldisuccinimide

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**Key indicators**

Single-crystal X-ray study
*T* = 133 K
Mean σ(C–C) = 0.002 Å
*R* factor = 0.034
*wR* factor = 0.090
Data-to-parameter ratio = 18.5

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



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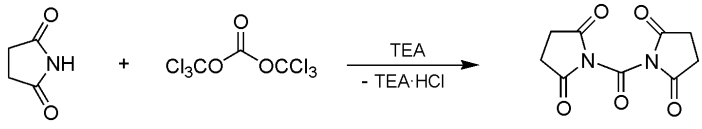
The title compound, $C_9H_8N_2O_5$, displays twofold symmetry to a good approximation. Bond lengths and angles (Å and °) at the central carbonyl group are: C=O 1.2001 (13), C—N 1.4052 (15) and 1.4139 (15), O=C—N 123.14 (12) and 123.16 (12), and N—C—N 113.70 (9). The succinimide rings subtend angles of 40.44 (6) and 37.74 (6)° with the carbonyl plane.

## Comment

*N,N′*-Carbonyldisuccinimide, (3), is a new compound that has considerable potential in organic synthesis. [Note: The title compound has the CAS registry number 158627-30-6 but is nonetheless new, to the best of our knowledge. All previous references to it have in fact involved *N,N′*-disuccinimidyl carbonate (CAS 74124-79-1).]We were motivated to prepare it by literature reports that *N*-trifluoroacetylsuccinimide displays enhanced reactivity towards N- and O-nucleophiles (Katritzky *et al.*, 1999). We are interested in compounds of higher reactivity that can be used instead of phosgene (Ryan *et al.*, 1996; Senet, 1997). In this connection, we have studied the reactivity and structures of organic carbonates such as *N,N′*-disuccinimidyl carbonate (preceding paper; Simon, Csunderlik *et al.*, 2003, and references therein; Simon, Csunderlik & Medeleanu, 2003). Here we report the synthesis (see *Experimental*) of the title compound (3) from triphosgene (2) and succinimide (1), together with its structure (see *Scheme*, TEA is triethylamine).




(1) (2) (3)

The molecule of (3) is shown in Fig. 1. It displays non-crystallographic twofold symmetry to a good approximation (r.m.s. deviation 0.022 Å), with the twofold axis passing along the bond C9=O5. The central $CN_2O$ moiety is planar (r.m.s. deviation 0.0014 Å) and subtends angles of 40.44 (6) and 37.74 (6)° with the succinimide rings. Bond lengths and angles may be regarded as normal (Table 1).

Each H atom is involved in weak C—H···O hydrogen bonds, H3*B* and H7*B* in three-centre systems. The net effect is to link the molecules in a complex three-dimensional network.

## Experimental

A solution of triphosgene (Cotarcă *et al.*, 1996; 0.107 g, 0.36 mmol) in 10 ml dichloromethane was added at 278 K to a solution of succinimide (0.2138 g, 2.16 mmol) and triethylamine (0.3 ml, 2.16 mmol) in

10 ml dichloromethane. The solution was allowed to stand at room temperature for 1 h. The precipitate was filtered off and washed with dichloromethane (2 × 2.5 ml), yielding 0.1037 g (66%) product as a white solid. Single crystals were obtained from acetonitrile. M.p. 564–566 K; IR(KBr) 1824, 1754 cm$^{-1}$ (C═O).

*Crystal data*

| | |
|---|---|
| $C_9H_8N_2O_5$ | $D_x$ = 1.608 Mg m$^{-3}$ |
| $M_r$ = 224.17 | Mo $K\alpha$ radiation |
| Monoclinic, $P2_1/c$ | Cell parameters from 5487 reflections |
| $a$ = 7.2372 (11) Å | $\theta$ = 2.6–30.4° |
| $b$ = 13.396 (2) Å | $\mu$ = 0.13 mm$^{-1}$ |
| $c$ = 9.5519 (12) Å | $T$ = 133 (2) K |
| $\beta$ = 90.01 (2)° | Tablet, colourless |
| $V$ = 926.1 (2) Å$^3$ | 0.30 × 0.15 × 0.10 mm |
| $Z$ = 4 | |

*Data collection*

| | |
|---|---|
| Bruker SMART 1000 CCD diffractometer | 2463 reflections with $I > 2\sigma(I)$ |
| $\omega$ scans | $R_{int}$ = 0.032 |
| Absorption correction: none | $\theta_{max}$ = 30.0° |
| 10780 measured reflections | $h = -10 \rightarrow 10$ |
| 2707 independent reflections | $k = -18 \rightarrow 18$ |
| | $l = -13 \rightarrow 13$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | H-atom parameters constrained |
| $R[F^2 > 2\sigma(F^2)]$ = 0.034 | $w = 1/[\sigma^2(F_o^2) + (0.0616P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $wR(F^2)$ = 0.091 | $(\Delta/\sigma)_{max} < 0.001$ |
| $S$ = 1.06 | $\Delta\rho_{max}$ = 0.36 e Å$^{-3}$ |
| 2707 reflections | $\Delta\rho_{min}$ = −0.18 e Å$^{-3}$ |
| 146 parameters | |

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| C9—O5 | 1.2001 (13) | C9—N2 | 1.4139 (15) |
| C9—N1 | 1.4052 (15) | | |
| O5—C9—N1 | 123.16 (12) | N1—C9—N2 | 113.70 (9) |
| O5—C9—N2 | 123.14 (12) | | |
| O5—C9—N1—C1 | 39.17 (18) | N2—C9—N1—C4 | 42.70 (15) |

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C2—H2$A$···O1$^{i}$ | 0.99 | 2.60 | 3.3530 (15) | 132 |
| C6—H6$B$···O1$^{ii}$ | 0.99 | 3.23 | 3.4198 (16) | 93 |
| C3—H3$B$···O1$^{i}$ | 0.99 | 2.58 | 3.3436 (15) | 134 |
| C3—H3$B$···O2$^{iii}$ | 0.99 | 2.54 | 3.3108 (16) | 134 |
| C7—H7$A$···O2$^{iv}$ | 0.99 | 2.60 | 3.5779 (17) | 168 |
| C2—H2$B$···O3$^{v}$ | 0.99 | 2.41 | 3.2274 (16) | 139 |
| C7—H7$B$···O3$^{vi}$ | 0.99 | 2.66 | 3.4543 (15) | 137 |
| C7—H7$B$···O4$^{vii}$ | 0.99 | 2.64 | 3.3011 (16) | 124 |
| C3—H3$A$···O4$^{viii}$ | 0.99 | 2.57 | 3.5548 (16) | 174 |
| C6—H6$A$···O4$^{ii}$ | 0.99 | 2.68 | 3.3862 (15) | 129 |

Symmetry codes: (i) $x, \frac{1}{2}-y, z-\frac{1}{2}$; (ii) $-x, \frac{1}{2}+y, \frac{1}{2}-z$; (iii) $1-x, 1-y, -z$; (iv) $x-1, y, z$; (v) $1-x, 1-y, 1-z$; (vi) $x, \frac{3}{2}-y, z-\frac{1}{2}$; (vii) $-x, 1-y, -z$; (viii) $1+x, y, z$.

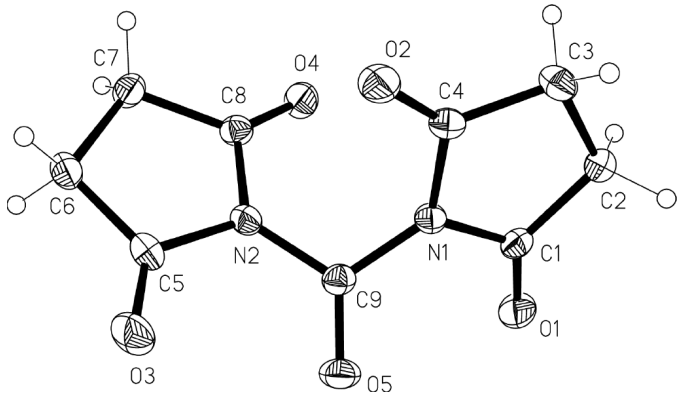



**Figure 1**
The molecule of the title compound in the crystal. Ellipsoids are shown at the 50% probability level and H-atom radii are arbitrary.

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 structure was refined as a pseudo-merohedral twin with components 0.451:0.549 (2), twin matrix (−1 0 0 / 0 −1 0 / 0 0 1).

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.

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## References

Bruker (1998). *SMART* (Version 5.0) and *SAINT* (Version 4.0). Bruker AXS Inc., Madison, Wisconsin, USA.
Cotarcă, L., Delogu, P., Nardelli, A. & Sunjic, V. (1996). *Synthesis*, **5**, 553–576.
Katritzky, A. R., Yang, B., Qiu, G. & Zhang, Z. (1999). *Synthesis*, **1**, 55–57.
Ryan, T. A., Ryan, C., Seddon, E. A. & Seddon, K. R. (1996). *Phosgene and Related Carbonyl Halides*, Vol. 1. Amsterdam: Elsevier.
Senet, J. P. (1997). *Recent Advances in Phosgene Chemistry*. Nanterre, France: Societé Nationale des Poudres et Explosifs.
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. & Tarta-Micle, A. (2003). *Acta Cryst.* E**59**, o683–o685.
Simon, M., Csunderlik, C. & Medeleanu, M. (2003). *Rev. Chim.* **54**. In the press.