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(*S*)-*trans*-Cyclohexane-1,2-dicarboximide

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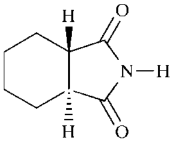
The molecule of the title compound, $C_8H_{11}NO_2$, contains a strained bicyclic system with a significantly twisted imide chromophore. The five-membered ring fragment containing the imide function is strongly puckered and adopts a half-chair conformation. The six-membered ring has a slightly distorted chair conformation. The molecules are joined by strong N—H$\cdots$O and weak C—H$\cdots$O hydrogen bonds into infinite chains.

Comment

The title compound, (I), can be considered as an example of a strongly twisted succinimide moiety and imide chromophore as a result of *trans*-fusion of the six- and five-membered rings. Compound (I) was synthesized, together with some optically active succinic anhydrides and imides of known absolute configuration, in order to study the chiro-optical properties of five-membered ring compounds with C_2 and C_{2v} local symmetry of the chromophores (Połoński, 1988).

It has been shown that twisting of the chromophores profoundly influences the circular dichroism spectra of cyclic anhydrides and imides (Połoński, 1988; Połoński *et al.*, 1993). Since the molecular geometry parameters of (I) were known only from semi-empirical MNDO (modified neglect of diatomic overlap) calculations (Połoński *et al.*, 1993), we decided to determine the structure of the *S*,*S*-enantiomer of (I) by single-crystal X-ray analysis, and we present the results of this analysis here.



(I)

A view of the molecule of (I) with the atom-labelling scheme is shown in Fig. 1 and torsion angles are given in Table 1. A search of the Cambridge Structural Database (CSD, Version 5.23; Allen & Kennard, 1993) revealed that monocyclic as well as bicyclic succinimide and dithiosuccinimide derivatives exhibit various degrees of puckering of the five-membered ring, with the puckering amplitude (Cremer & Pople, 1975) being as high as 0.269 Å in tetramethyldithiosuccinimide (CSD refcode NINTAF; Ratajczak-Sitarz *et al.*, 1996) or 0.285 Å in 1,6-dimethyl-8-hydroxy-8-azabicyclo[4.3.0]-nonane-7,9-dione (CSD refcode DENVAT; Hengjin *et al.*, 1985), and only 0.021 Å in succinimide (Fronczek, 1995). The puckering of the succinimide fragment of (I) is very similar to that of NINTAF and DENVAT, with a puckering amplitude of 0.289 (2) Å and a phase angle of 268.6 (4)° (266.8 and 272.5° for NINTAF and DENVAT, respectively), indicating a half-chair conformation of the five-membered ring. The six-membered ring has a slightly distorted chair conformation, with absolute values of the endocyclic torsion angles in the range 53.0 (2)–65.4 (2)°.

Molecules of (I) are connected into infinite polar chains in the crystal *via* a strong N2—H2$\cdots$O1($y - 1, x, -z$) hydrogen bond and two weak C—H$\cdots$O interactions (Table 2). There is a striking similarity between the one-dimensional aggregate structure of this chiral imide and the one-dimensional networks formed by the achiral molecules of 3,6-dithia-3,4,5,6-tetrahydrophthalimide (DTTHP; Kirfel *et al.*, 1975) and 3,4,5,6-tetrahydrophthalimide (THP; Kirfel, 1975) (Fig. 2).

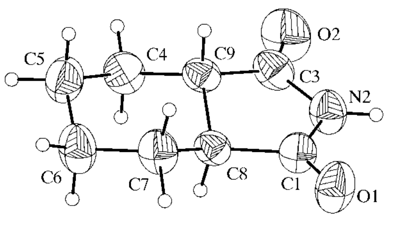
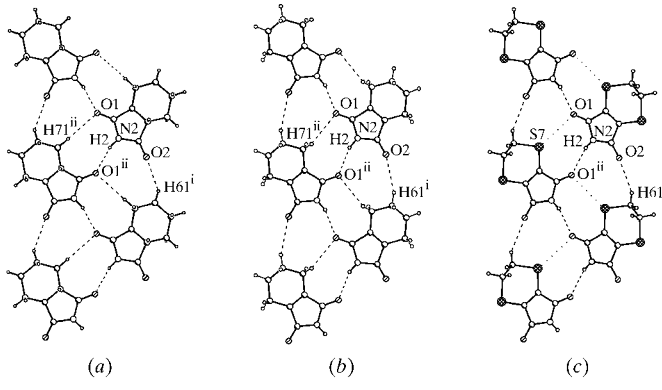


Figure 1
A view of the structure of (I) with 50% probability displacement ellipsoids. H atoms are shown as small spheres of arbitrary radii.



(*a*) (*b*) (*c*)

Figure 2
A comparision of the one-dimensional hydrogen-bonded networks in the crystal structures of hydrogenated phthalimide derivatives with approximately flat condensed ring systems; (*a*) (I) [symmetry codes: (i) $x - 1, y - 1, z$; (ii) $y - 1, x, -z$], (*b*) THP [symmetry codes: (i) $x, y, 1 + z$; (ii) $x, -1 - y, \frac{1}{2} + z$], and (*c*) DTTHP [symmetry codes: (i) $x, y, 1 + z$; (ii) $x, -\frac{1}{2} - y, \frac{1}{2} + z$].

The latter two compounds are less strained than (I), but their bicyclic skeletons are significantly flattened, due to the C8═C9 double bond. Similar to (I), their polar one-dimensional networks are stabilized by an N—H···O hydrogen bond and C—H···O interactions. However, one of the short C—H···O contacts in DTTHP is substituted by an S···O contact of 3.480 Å. Substantial differences in the packing modes of these one-dimensional aggregates are observed at the higher level of the structure organization, leading to different space-group symmetries in each case [$P4_12_12$ for (I), *Pmca* for THP and $P2_1/c$ for DTTHP].

Experimental

Optically active (I) was prepared as described previously by Połoński (1988). Crystals of (I) can be grown from various polar and non-polar organic solvents as square plates with well developed {001} faces, but due to serious crystal defects, they are not suitable for X-ray diffraction studies. However, after many crystallization attempts, we were able to choose, with the help of a polarizing microscope, one good quality single crystal which had the form of a {100} plate and gave a satisfactory diffraction pattern.

Crystal data

$C_8H_{11}NO_2$
M_r = 153.18
Tetragonal, $P4_12_12$
a = 5.903 (1) Å
c = 45.164 (4) Å
V = 1573.8 (3) Å^3
Z = 8
D_x = 1.292 Mg m^{-3}
Cu *Kα* radiation
Cell parameters from 46 reflections
θ = 8–51°
μ = 0.77 mm^{-1}
T = 293 (2) K
Plate, colourless
0.5 × 0.5 × 0.2 mm

Data collection

Kuma KM-4 four-circle diffractometer
ω/2*θ* scans
1956 measured reflections
1660 independent reflections
1563 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.051
$\theta_{\rm max}$ = 80.1°
h = 0 → 7
k = 0 → 7
l = 0 → 57
3 standard reflections every 100 reflections intensity decay: 1.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.125$
S = 1.07
1660 reflections
145 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0825P)^2 + 0.2028P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max}$ = 0.14 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.13 e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997)
Extinction coefficient: 0.0029 (8)

Table 1
Selected torsion angles (°).

C8—C1—N2—C3	10.5 (2)	C6—C7—C8—C9	59.7 (2)
C1—N2—C3—C9	8.8 (2)	N2—C3—C9—C8	−23.7 (2)
C9—C4—C5—C6	−53.0 (2)	C5—C4—C9—C8	58.4 (2)
C4—C5—C6—C7	53.5 (3)	C1—C8—C9—C3	29.0 (2)
C5—C6—C7—C8	−54.3 (2)	C7—C8—C9—C4	−65.4 (2)
N2—C1—C8—C9	−24.8 (2)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O1$^{\rm i}$	0.87 (3)	2.11 (3)	2.933 (2)	159 (2)
C7—H71···O1$^{\rm ii}$	1.02 (3)	2.70 (3)	3.658 (2)	157 (2)
C6—H61···O2$^{\rm iii}$	0.90 (3)	2.54 (3)	3.308 (2)	144 (2)

Symmetry codes: (i) $y-1, x, -z$; (ii) $y, 1+x, -z$; (iii) $1+x, 1+y, z$.

The enantiomorphous space group $P4_12_12$ was assigned based on the known absolute configuration of (I). H atoms were located in a difference Fourier map. Their positional and isotropic displacement parameters were included in the refinement; refined C—H distances were in the range 0.90 (3)–1.07 (3) Å.

Data collection: *KM*-4 *Software* (Kuma, 1991); cell refinement: *KM*-4 *Software*; data reduction: *KM*-4 *Software*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *Stereochemical Workstation* (Siemens, 1989); software used to prepare material for publication: *SHELXL*97.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FA1000). Services for accessing these data are described at the back of the journal.

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