4-[(1,3-Dioxo-2-azaspiro[4.5]decan-2-yl)-amino]benzoic acid

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In the structure of the title compound, $C_{16}H_{18}N_2O_4$, intermolecular N—H···O [N···O = 2.866 (3) Å] and O—H···O [O···O = 2.611 (3) Å] hydrogen bonds link molecules into a two-dimensional framework. The crystal structure is further stabilized by weak intermolecular C—H···O interactions.

Comment

In the course of our investigation on packing motifs in succinimides (Obniska *et al.*, 2005; Dzierżawska-Majewska *et al.*, 2006), we have determined the structure of the title compound, (I), where the carboxyl substituent on the benzene ring may be a useful hydrogen-bond donor/acceptor. A view of (I) is shown in Fig. 1. The bicyclic skeleton consists of succinimide and spirocyclohexane attached at atom C3. The five-membered imide ring adopts a pseudo-half-chair conformation, while the cyclohexane ring is in a chair form. The general conformation of the molecule can be characterized by the torsion angles Φ1 (C2—N1—N2—C21) and Φ2 (C5—N1—N2—C21) which have values of −84.6 (3) and 113.9 (2)°, respectively. In all *N*-aminophenyl-substituted succinimides there are at least four distinct phenyl-ring orientations, distinguishable by the values of these two torsion angles. The conformation of (I) is different from that of *N*-aminophenyl-2-azaspiro[4.5]decane-1,3-dione [Φ1 = −112.3 (4)° and Φ2 = 74.8 (4)°] and *N*-aminophenyl-2-azaspiro[4.4]nonane-1,3-dione [Φ1 = −112.8 (7)° and Φ2 = 84.1 (6)°], which are analogous to similar structures with unsubstituted phenyl rings (Obniska *et al.*, 2005).

(I)
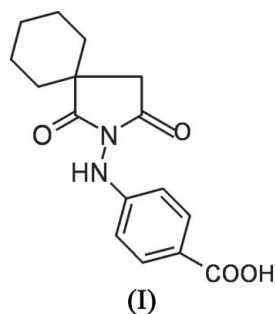
In the crystal structure, molecules form the centrosymmetric hydrogen-bonded dimers expected for carboxylic acids (Table 1). In addition, intermolecular N—H···O hydrogen bonds connect molecules into a two-dimensional framework (Fig. 2) and weak C—H···O interactions complete the formation of a three-dimensional network (Table 1). Similar
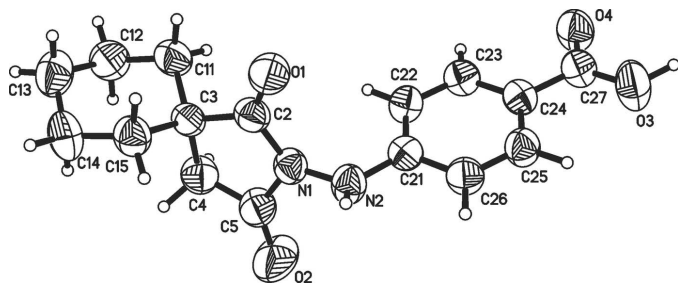
Figure 1
The molecular structure of the title compound, showing 50% probability displacement ellipsoids. H atoms are drawn as small spheres.

Figure 2
Part of the crystal structure of (I), showing the hydrogen-bond interactions as dotted lines.

types of hydrogen bonding have been observed in a structure we have already studied (Obniska *et al.*, 2005). It should be noted that in *N*-benzylsuccinimide structures (with a CH_2 group replacing atom N2), molecules are connected only by means of C—H···O hydrogen bonds (Dzierżawska-Majewska *et al.*, 2006).
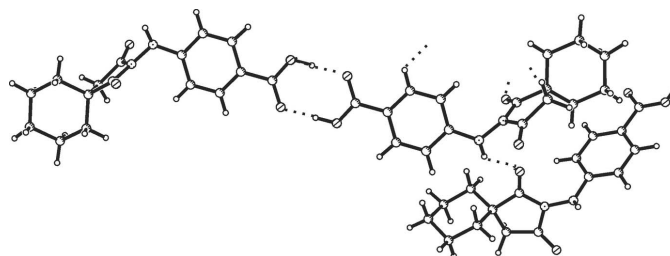
Experimental

4-Hydrazinebenzoic acid (0.01 mol) was dissolved in water (20 ml), and then 1-carboxy-1-cyclohexane-1-acetic acid (0.01 mol) was gradually added. The mixture was heated in an oil bath with simultaneous distillation of water. After the complete removal of water, the temperature of the reaction mixture was raised to 453 K and maintained at this level for 1.5 h. The crude products were recrystallized from ethanol and single crystals of the title compound were obtained by slow evaporation of an ethanol solution [yield: 74%; m.p. 499–501 K; R_F (S_1) = 0.12, (S_2) = 0.64]. ^{1}H NMR (CDCl$_3$): δ 1.33–1.80 (*m*, 10H, cyclohexane), 2.71 (*s*, 2H, succinimide), 3.50–3.57 (*q*, 1H, NH), 6.70–6.72 (*m*, 2H, arom.), 7.82–7.90 (*m*, 2H, aromatic), 11.02 (rough *s*, 1H, COOH).

Crystal data

$C_{16}H_{18}N_2O_4$	$D_x = 1.358$ Mg m^{-3}
$M_r = 302.32$	Cu $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 930
$a = 18.740\ (4)$ Å	reflections
$b = 6.1600\ (12)$ Å	$\theta = 12.3$–43.6°
$c = 25.637\ (5)$ Å	$\mu = 0.82$ mm^{-1}
$\beta = 92.54\ (3)$°	$T = 293\ (2)$ K
$V = 2956.6\ (10)$ Å^3	Plate, colorless
$Z = 8$	$0.3 \times 0.2 \times 0.05$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer	2777 independent reflections
ω scans	2073 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan	$R_{int} = 0.034$
(*SADABS*; Sheldrick, 2003)	$\theta_{max} = 70.1$°
$T_{min} = 0.784$, $T_{max} = 0.940$	$h = -22 \rightarrow 22$
15719 measured reflections	$k = -7 \rightarrow 7$
	$l = -30 \rightarrow 31$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0671P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.056$	$+ 2.2235P]$
$wR(F^2) = 0.150$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} < 0.001$
2777 reflections	$\Delta\rho_{max} = 0.22$ e Å^{-3}
200 parameters	$\Delta\rho_{min} = -0.30$ e Å^{-3}
H-atom parameters constrained	

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1^i	0.91	2.10	2.866 (3)	141
O3—H3···O4^{ii}	0.99	1.62	2.611 (3)	171
C4—H4B···O4^{iii}	1.03	2.59	3.380 (3)	133
C23—H23···O2^{iv}	0.94	2.41	3.249 (3)	149
C25—H25···O3	0.98	2.41	2.773 (3)	101

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

All H atoms were located in difference Fourier maps and included in the refinement in these as-found positions in a riding-model approximation, with U_{iso}(H) = U_{eq}(C or N).

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2003); software used to prepare material for publication: *SHELXTL*.

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