Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-(Methoxycarbonyl)succinanilic acid

Shazia Anjum,[a]* M. Iqbal Choudhary,[a] Shamsher Ali,[a] Hoong-Kun Fun[b]* and Atta-ur-Rahman[a]

[a]HEJ Research Institute of Chemistry, International Centre for Chemical Sciences, University of Karachi, Karachi 75270, Pakistan, and [b]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia

Correspondence e-mail: anjumshazia@yahoo.com, hkfun@usm.my

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.042
wR factor = 0.113
Data-to-parameter ratio = 14.8

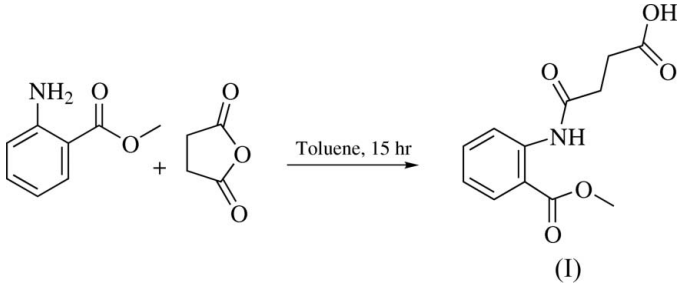
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 14 September 2005
Accepted 19 September 2005
Online 30 September 2005

The title compound, $C_{12}H_{13}NO_5$, was synthesized by the condensation of methyl anthranilate and succinic anhydride. The dihedral angle between the phenylacetamide and carboxylic acid (–C—COOH) planes is 80.47 (5)°. In the crystal structure, inversion-related molecules form an O—H···O hydrogen-bonded dimer. Adjacent dimers are interlinked by C—H···O hydrogen bonds to form a chain along [110].

Comment

Methyl(2-methoxycarbonyl)succinate, a natural aromatic amide isolated from the methanolic extract of *Jolyna laminarioides*, has shown potent chymotrypsin inhibitory activity (Atta-ur-Rahman *et al.*, 1997). Therefore, we have synthesized our desired analogue, (I), in 85% yield, by a one-step condensation of methyl anthranilate and succinic anhydride (see scheme). We report here the structure of (I).



(I)

Molecules of the title compound, (I), have normal bond lengths (Allen *et al.*, 1987). The C6—N1—C7 bond angle

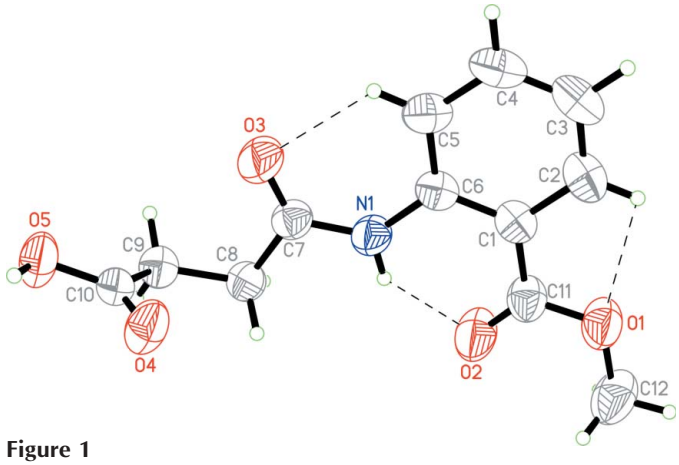


Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. Dashed lines indicate intramolecular hydrogen bonds.

[130.13 (11)°] is larger than the value of 126.69 (10)° observed in *N*,*N*′-diphenylsuccinimide (Anjum *et al.*, 2005). The N1—C7 and C8—C9 bonds are *trans* with respect to the C7—C8 bond for steric reasons, the N1—C7—C8—C9 torsion angle being 175.77 (11)°.

The phenylacetamide moiety is planar to within ±0.018 (1) Å. The C9—C10—O4—O5 and C11—C12—O1—O2 planes form dihedral angles of 80.47 (5) and 9.38 (9)°, respectively, with the phenylacetamide moiety. This orientation is influenced by intramolecular C—H···O and N—H···O interactions, namely N1—H1*N*···O2, C5—H5···O3 and C2—H2···O1 (Table 1). As seen in Fig. 1, each of these interactions generates rings of graph-set motif *S*(5) or *S*(6) (Bernstein *et al.*, 1995).

In the crystal structure, centrosymmetrically related molecules are linked by O5—H1O5···O4^{i} intermolecular hydrogen bonds to form a dimeric pair (symmetry code as in Table 1). These hydrogen bonds form an $R_2^2(8)$ ring motif. The adjacent dimers are interlinked by C9—H9*B*···O2^{ii} hydrogen bonds to form a chain along [110]. These hydrogen bonds form an $R_2^2(18)$ ring motif (Fig. 2). A C—H···π interaction involving atom H8*B* and the C1–C6 ring is observed, with atom H8*B* separated from the centroid (*Cg*1) of the ring by 2.58 Å (Table 1).

Experimental

Succinic anhydride (1.0 g, 0.01 mol) was added to methyl anthranilate (0.5 g, 0.003 mol) in a round-bottomed flask containing dry toluene (50 ml). The reaction mixture was then refluxed for 15 h using a Dean–Stark trap. The reaction mixture was quickly filtered and left for crystallization at room temperature, resulting in colourless crystals of compound (I) (1.01 g, yield 85%, m.p. 539–540 K).

Crystal data

$C_{12}H_{13}NO_5$
M_r = 251.23
Monoclinic, $C2/c$
a = 18.301 (2) Å
b = 12.1788 (15) Å
c = 10.8967 (14) Å
β = 93.001 (2)°
V = 2425.3 (5) Å^3
Z = 8
D_x = 1.376 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5412 reflections
θ = 2.0–26.5°
μ = 0.11 mm^{-1}
T = 293 (2) K
Block, colourless
0.42 × 0.27 × 0.24 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.956, T_{max} = 0.975
6736 measured reflections
2498 independent reflections
2230 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 26.5°
$h = -22 \rightarrow 22$
$k = -15 \rightarrow 14$
$l = -12 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.113
S = 1.04
2498 reflections
169 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0551P)^2 + 1.0027P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Extinction correction: *SHELXTL* (Sheldrick, 1997)
Extinction coefficient: 0.0070 (10)

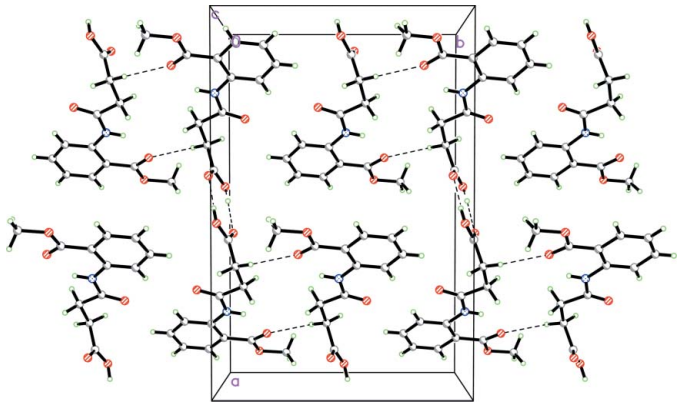


Figure 2
Part of the crystal structure of (I), showing $R_2^2(8)$ and $R_2^2(18)$ ring motifs. Dashed lines indicate hydrogen bonds.

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

*Cg*1 is the centroid of ring C1–C6.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N···O2	0.86	1.94	2.6534 (17)	139
O5—H1O5···O4^{i}	0.87 (2)	1.80 (2)	2.6657 (15)	173 (2)
C2—H2···O1	0.93	2.36	2.700 (2)	101
C5—H5···O3	0.93	2.27	2.881 (2)	123
C9—H9*B*···O2^{ii}	0.97	2.42	3.3184 (19)	154
C8—H8*B*···*Cg*1^{iii}	0.97	2.58	3.4470 (15)	149

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

Atom H1O5 was located in a difference Fourier map and refined isotropically. All other H atoms were positioned geometrically and allowed to ride on their parent atoms, with N—H = 0.86 Å and C—H = 0.93–0.97 Å, and with U_{iso}(H) = 1.2 or 1.5U_{eq}(C,N). A rotating-group model was used for the methyl group.

Data collection: *SMART* (Siemens, 1997); cell refinement: *SAINT* (Siemens, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

Financial support from the Higher Education Commission, Government of Pakistan, is gratefully acknowledged. AUR, SA and HKF also thank the Malaysian Government and Universiti Sains Malaysia for Scientific Advancement Grant Allocation (SAGA) grant No. 304/PFIZIK/653003/A118.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–S19.
Anjum, S., Choudhary, M. I., Ali, S., Fun, H.-K. & Atta-ur-Rahman (2005). *Acta Cryst.* E**61**, o3001–o3002.
Atta-ur-Rahman, Choudhary, M. I., Majeed, A., Ghani, U., Shabir, M. & Shameel, M. (1997). *Phytochemistry*, **46**, 1215–1218.
Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.

Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Siemens (1997). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.