

3-(3-Nitrophenylaminocarbonyl)-propionic acid: hydrogen-bonded sheets of alternating $R_2^2(8)$ and $R_6^6(36)$ rings

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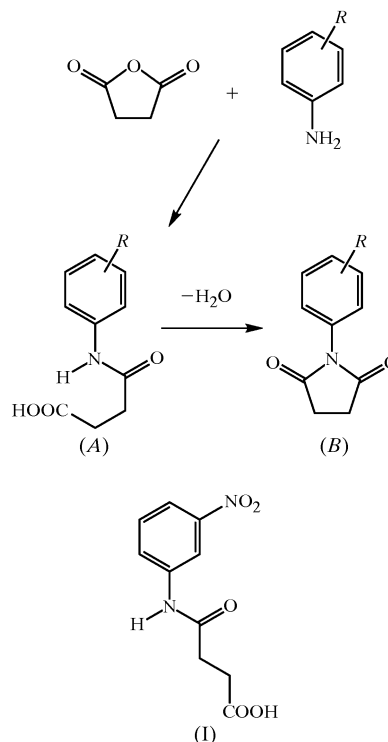
Molecules of the title compound, $C_{10}H_{10}N_2O_5$, are linked by a combination of $O-H\cdots O$ and $N-H\cdots O$ hydrogen bonds into (100) sheets containing alternating $R_2^2(8)$ and $R_6^6(36)$ rings.

Comment

The reaction of C-substituted anilines, such as nitroanilines, with succinic anhydride yields initially 3-(arylamino-carbonyl)propionic acids, (A) (see scheme), dehydration of which yields the corresponding *N*-arylsuccinimides, (B). We have recently reported the molecular and supramolecular structures of the three isomeric *N*-(nitrophenyl)succinimides (B), where $R = NO_2$ (Glidewell *et al.*, 2005). We have now prepared all three isomeric 3-(nitrophenylaminocarbonyl)propionic acids (A), where $R = NO_2$, but unfortunately only the 3-nitro isomer has provided crystals suitable for single-crystal structure determination. We report here the molecular and supramolecular structures of 3-(3-nitrophenylaminocarbonyl)propionic acid, (I).

The molecules of (I) (Fig. 1) are linked into sheets by a combination of an $N-H\cdots O=C$ hydrogen bond, forming the usual amidic $C(4)$ chain, and an $O-H\cdots O$ hydrogen bond, forming the usual centrosymmetric $R_2^2(8)$ (Bernstein *et al.*, 1995) motif characteristic of simple carboxylic acids (Table 1). Carboxyl atom O43 in the molecule at (x, y, z) acts as a hydrogen-bond donor to atom O44 in the molecule at $(1-x, 1-y, 1-z)$, so that the reference $R_2^2(8)$ dimer is centred at $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$ (Fig. 2). Amide atoms N1 at (x, y, z) and $(1-x, 1-y, 1-z)$, which form part of the dimer centred at $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$, act as hydrogen-bond donors to amide atoms O1 at $(x, \frac{1}{2}-y, -\frac{1}{2}+z)$ and $(1-x, \frac{1}{2}+y, \frac{3}{2}-z)$, respectively, which themselves form

parts of the $R_2^2(8)$ dimers centred at $(\frac{1}{2}, 0, 0)$ and $(\frac{1}{2}, 1, 1)$, respectively. Similarly, atoms O1 at (x, y, z) and $(1-x, 1-y, 1-z)$ accept hydrogen bonds from atoms N1 at $(x, \frac{1}{2}-y, \frac{1}{2}+z)$ and $(1-x, \frac{1}{2}+y, \frac{1}{2}-z)$, which are pairs of the dimers centred, respectively, at $(\frac{1}{2}, 0, 1)$ and $(\frac{1}{2}, 1, 0)$. In this manner, each dimer



is directly linked to four other dimers *via* the amidic $C(4)$ chains along [001], so forming a (100) sheet in which centrosymmetric $R_2^2(8)$ and $R_6^6(36)$ rings alternate in a chessboard fashion (Fig. 3).

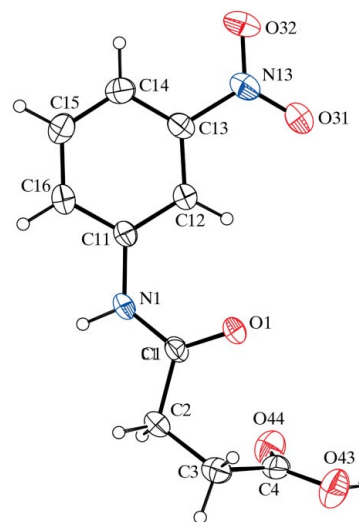


Figure 1

The molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

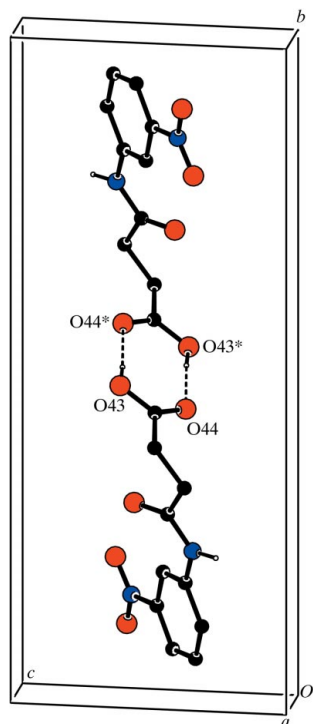


Figure 2
Part of the crystal structure of (I), showing the formation of an $R_2^2(8)$ dimer centred at $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$. Atoms marked with an asterisk (*) are at the symmetry position $(1-x, 1-y, 1-z)$.

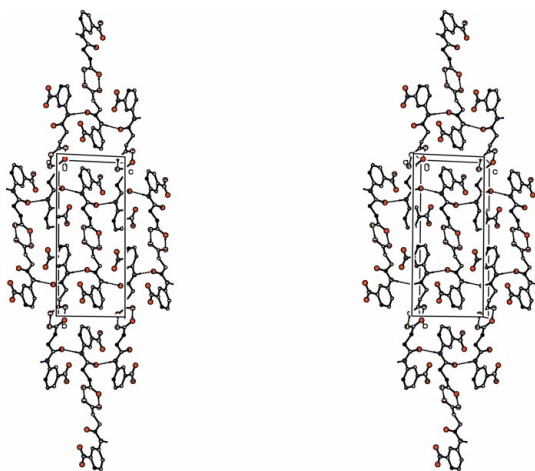


Figure 3
A stereoview of part of the crystal structure of (I), showing the formation of a (100) sheet built from $R_2^2(8)$ and $R_6^0(36)$ rings.

Experimental

A solution containing equimolar quantities of succinic anhydride and 3-nitroaniline (2 mmol of each) in 1,2-dichloroethane (20 ml) was heated under reflux for 1 h and then left overnight at room temperature. The solvent was removed under reduced pressure and the resulting solid product was recrystallized from ethanol (m.p. 455–457 K). IR (KBr): 3400–2000 (*br*), 1706, 1673, 1524, 1556, 1524, 1481, 1434, 1403, 1351, 1257, 1237, 1179, 1089, 1064, 993, 952, 891, 868, 847, 819, 806, 737, 684, 670, 606, 540, 421, 498 cm^{-1} .

Crystal data

$\text{C}_{10}\text{H}_{10}\text{N}_2\text{O}_5$
 $M_r = 238.20$
Monoclinic, $P2_1/c$
 $a = 6.6765$ (4) Å
 $b = 19.7961$ (13) Å
 $c = 9.0675$ (5) Å
 $\beta = 113.595$ (4)°
 $V = 1098.25$ (11) Å³
 $Z = 4$

$D_x = 1.441$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 2525 reflections
 $\theta = 2.1$ – 27.6 °
 $\mu = 0.12$ mm⁻¹
 $T = 293$ (2) K
Plate, colourless
 $0.38 \times 0.17 \times 0.04$ mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
 ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2000)
 $T_{\min} = 0.967$, $T_{\max} = 0.995$
9375 measured reflections

2525 independent reflections
1537 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.038$
 $\theta_{\max} = 27.6$ °
 $h = -8 \rightarrow 8$
 $k = -25 \rightarrow 25$
 $l = -11 \rightarrow 9$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.115$
 $S = 0.91$
2525 reflections
162 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0652P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.15$ e Å⁻³
 $\Delta\rho_{\min} = -0.22$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{N1}-\text{H1} \cdots \text{O1}^i$	0.89	1.96	2.850 (2)	173
$\text{O43}-\text{H43} \cdots \text{O44}^{ii}$	0.82	1.84	2.654 (2)	175

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

The space group $P2_1/c$ was uniquely assigned from the systematic absences. All H atoms were located from difference maps and then treated as riding atoms, with C–H distances of 0.93 (aromatic) or 0.97 Å (CH_2), an N–H distance of 0.89 Å, and an O–H distance of 0.82 Å, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N}, \text{O})$.

Data collection: SMART (Bruker, 2000); cell refinement: SAINT-Plus (Bruker, 2000); data reduction: SAINT-Plus; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: PLATON (Spek, 2003); software used to prepare material for publication: SHELXL97 and PRPKAPPA (Ferguson, 1999).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1893). Services for accessing these data are described at the back of the journal.

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