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

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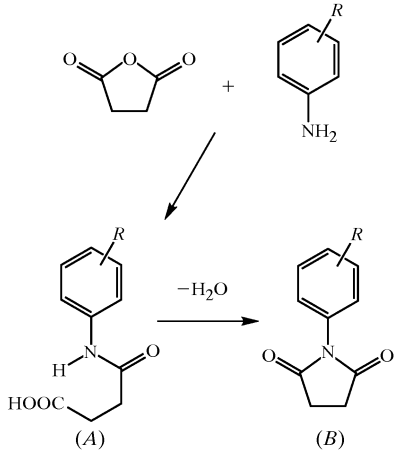
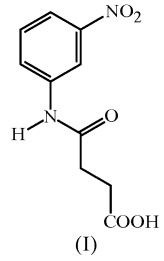
Molecules of the title compound, $C_{10}H_{10}N_2O_5$, are linked by a combination of O—H···O and N—H···O hydrogen bonds into (100) sheets containing alternating $R_2^2(8)$ and $R_6^6(36)$ rings.

Comment

The reaction of C-substituted anilines, such as nitroanilines, with succinic anhydride yields initially 3-(arylaminocarbonyl)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···O=C hydrogen bond, forming the usual amidic *C*(4) chain, and an O—H···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).

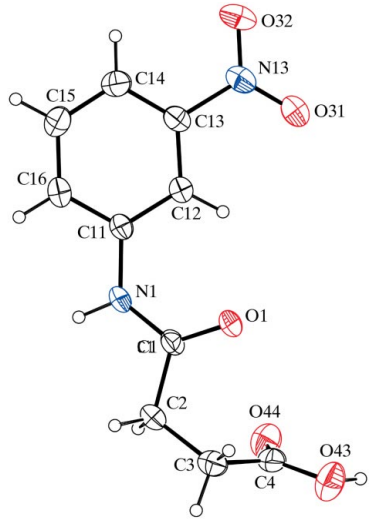


Figure 1
The molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

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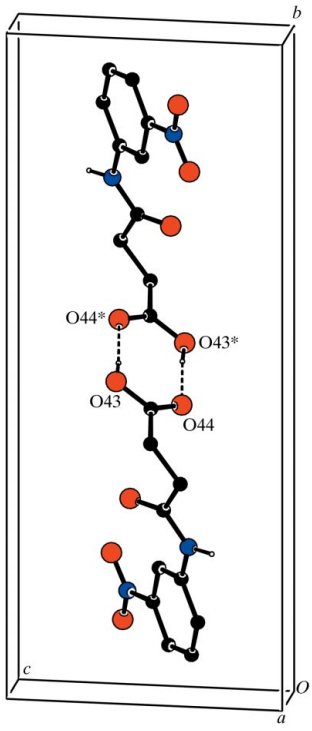

Figure 2
Part of the crystal structure of (I), showing the formation of an $R_2^2(8)$ dimer centred at $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$. Atoms marked with an asterisk (*) are at the symmetry position $(1 - x, 1 - y, 1 - z)$.

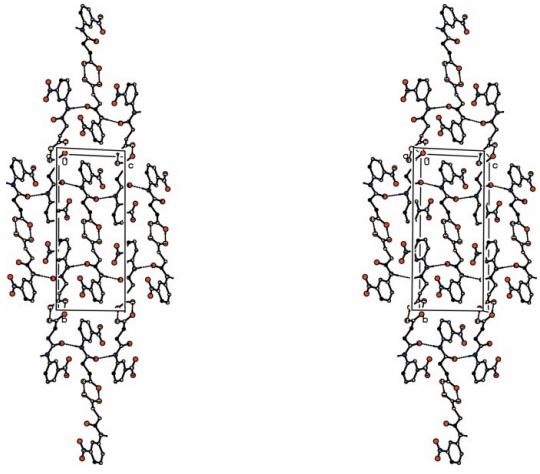

Figure 3
A stereoview of part of the crystal structure of (I), showing the formation of a (100) sheet built from $R_2^2(8)$ and $R_6^6(36)$ rings.

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

$C_{10}H_{10}N_2O_5$
M_r = 238.20
Monoclinic, $P2_1/c$
a = 6.6765 (4) Å
b = 19.7961 (13) Å
c = 9.0675 (5) Å
β = 113.595 (4)°
V = 1098.25 (11) Å^3
Z = 4
D_x = 1.441 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2525 reflections
θ = 2.1–27.6°
μ = 0.12 mm^{-1}
T = 293 (2) K
Plate, colourless
0.38 × 0.17 × 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_{int} = 0.038
θ_{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···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.89	1.96	2.850 (2)	173
O43—H43···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_{iso}(H) = 1.2U_{eq}(C,N,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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supporting information

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$\theta_{max} = 27.6°$, $\theta_{min} = 2.1°$
$h = -8\rightarrow8$
$k = -25\rightarrow25$
$l = -11\rightarrow9$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.115$
$S = 0.91$
2525 reflections
162 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
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}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.6360 (2)	0.29810 (6)	0.56794 (13)	0.0575 (3)
O31	0.0969 (3)	0.19919 (8)	0.6589 (2)	0.0904 (5)
O32	−0.0117 (2)	0.09628 (8)	0.6223 (2)	0.0893 (5)
O43	0.7815 (2)	0.47720 (7)	0.61217 (16)	0.0787 (4)
O44	0.5264 (2)	0.43856 (6)	0.38524 (15)	0.0678 (4)
N1	0.5794 (2)	0.22718 (7)	0.35833 (16)	0.0483 (4)
N13	0.0921 (2)	0.14318 (8)	0.60141 (19)	0.0603 (4)
C1	0.6608 (3)	0.28337 (8)	0.44555 (19)	0.0435 (4)
C2	0.7889 (3)	0.32755 (8)	0.3793 (2)	0.0522 (4)
C3	0.8823 (3)	0.38893 (9)	0.4831 (2)	0.0591 (5)
C4	0.7125 (3)	0.43650 (8)	0.4891 (2)	0.0532 (4)
C11	0.4565 (3)	0.17529 (8)	0.39002 (18)	0.0427 (4)
C12	0.3293 (2)	0.18641 (8)	0.47620 (19)	0.0441 (4)
C13	0.2191 (2)	0.13195 (8)	0.50369 (19)	0.0464 (4)
C14	0.2248 (3)	0.06807 (9)	0.4459 (2)	0.0569 (5)
C15	0.3478 (3)	0.05859 (9)	0.3576 (2)	0.0622 (5)
C16	0.4634 (3)	0.11115 (9)	0.3298 (2)	0.0556 (4)
H1	0.6095	0.2191	0.2727	0.058*
H2A	0.6943	0.3421	0.2716	0.059 (5)*
H2B	0.9073	0.3014	0.3716	0.068 (5)*
H3A	0.9720	0.3741	0.5917	0.067 (5)*
H3B	0.9763	0.4128	0.4423	0.074 (6)*
H12	0.3183	0.2292	0.5146	0.050 (4)*
H14	0.1479	0.0326	0.4660	0.073 (6)*
H15	0.3534	0.0160	0.3159	0.076 (6)*
H16	0.5468	0.1037	0.2702	0.069 (6)*
H43	0.6813	0.5022	0.6082	0.094*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0883 (9)	0.0563 (7)	0.0480 (7)	−0.0122 (6)	0.0483 (7)	−0.0082 (5)
O31	0.1100 (12)	0.0823 (10)	0.1222 (13)	−0.0204 (8)	0.0920 (11)	−0.0257 (9)
O32	0.0977 (10)	0.0837 (10)	0.1189 (13)	−0.0270 (8)	0.0775 (10)	−0.0017 (9)
O43	0.0750 (9)	0.0744 (9)	0.0763 (9)	−0.0001 (7)	0.0191 (7)	−0.0268 (8)
O44	0.0728 (9)	0.0643 (8)	0.0584 (8)	0.0009 (6)	0.0181 (7)	−0.0069 (6)
N1	0.0629 (8)	0.0554 (8)	0.0410 (7)	−0.0074 (6)	0.0357 (7)	−0.0065 (6)
N13	0.0582 (9)	0.0686 (10)	0.0668 (10)	−0.0079 (8)	0.0383 (8)	−0.0006 (8)
C1	0.0540 (9)	0.0471 (9)	0.0378 (8)	0.0018 (7)	0.0272 (7)	0.0029 (7)
C2	0.0637 (10)	0.0556 (10)	0.0512 (10)	−0.0067 (8)	0.0377 (9)	−0.0014 (8)
C3	0.0618 (10)	0.0627 (11)	0.0615 (12)	−0.0149 (9)	0.0338 (9)	−0.0038 (9)

C4	0.0683 (11)	0.0470 (9)	0.0509 (10)	−0.0139 (8)	0.0310 (9)	−0.0015 (8)
C11	0.0500 (8)	0.0477 (8)	0.0347 (8)	−0.0018 (7)	0.0216 (7)	−0.0009 (7)
C12	0.0488 (9)	0.0454 (9)	0.0432 (9)	−0.0007 (7)	0.0238 (7)	−0.0026 (7)
C13	0.0444 (8)	0.0553 (9)	0.0431 (9)	−0.0025 (7)	0.0212 (7)	0.0024 (7)
C14	0.0643 (11)	0.0503 (10)	0.0590 (11)	−0.0109 (8)	0.0278 (9)	−0.0012 (8)
C15	0.0809 (12)	0.0473 (10)	0.0633 (12)	−0.0040 (9)	0.0342 (10)	−0.0104 (9)
C16	0.0680 (11)	0.0572 (10)	0.0525 (10)	−0.0005 (8)	0.0357 (9)	−0.0093 (8)

Geometric parameters (Å, º)

N1—C1	1.347 (2)	C14—H14	0.93
N1—C11	1.4145 (19)	C15—C16	1.377 (2)
N1—H1	0.89	C15—H15	0.9304
C1—O1	1.2214 (17)	C16—H16	0.93
C1—C2	1.506 (2)	C2—C3	1.511 (2)
C11—C12	1.382 (2)	C2—H2A	0.97
C11—C16	1.390 (2)	C2—H2B	0.97
C12—C13	1.383 (2)	C3—C4	1.491 (3)
C12—H12	0.93	C3—H3A	0.97
C13—C14	1.375 (2)	C3—H3B	0.97
C13—N13	1.468 (2)	C4—O44	1.223 (2)
N13—O32	1.2180 (18)	C4—O43	1.302 (2)
N13—O31	1.2201 (19)	O43—H43	0.82
C14—C15	1.370 (2)		
C1—N1—C11	127.67 (13)	C14—C15—H15	119.5
C1—N1—H1	118.8	C16—C15—H15	119.6
C11—N1—H1	113.5	C15—C16—C11	120.61 (16)
O1—C1—N1	124.13 (14)	C15—C16—H16	119.7
O1—C1—C2	121.94 (15)	C11—C16—H16	119.7
N1—C1—C2	113.93 (13)	C1—C2—C3	112.19 (13)
C12—C11—C16	119.47 (14)	C1—C2—H2A	109.2
C12—C11—N1	122.47 (14)	C3—C2—H2A	109.2
C16—C11—N1	118.05 (14)	C1—C2—H2B	109.1
C11—C12—C13	117.97 (14)	C3—C2—H2B	109.2
C11—C12—H12	121.0	H2A—C2—H2B	107.9
C13—C12—H12	121.0	C4—C3—C2	113.60 (15)
C14—C13—C12	123.39 (15)	C4—C3—H3A	108.9
C14—C13—N13	118.69 (14)	C2—C3—H3A	108.8
C12—C13—N13	117.91 (15)	C4—C3—H3B	108.9
O32—N13—O31	123.06 (16)	C2—C3—H3B	108.9
O32—N13—C13	118.72 (16)	H3A—C3—H3B	107.7
O31—N13—C13	118.22 (14)	O44—C4—O43	122.76 (17)
C15—C14—C13	117.59 (15)	O44—C4—C3	123.07 (16)
C15—C14—H14	121.2	O43—C4—C3	114.13 (16)
C13—C14—H14	121.2	C4—O43—H43	109.4
C14—C15—C16	120.93 (16)		

C11—N1—C1—O1	−1.0 (3)	C12—C13—C14—C15	0.5 (3)
C11—N1—C1—C2	178.73 (15)	N13—C13—C14—C15	−178.77 (15)
C1—N1—C11—C12	26.7 (2)	C13—C14—C15—C16	0.8 (3)
C1—N1—C11—C16	−154.11 (17)	C14—C15—C16—C11	−0.5 (3)
C16—C11—C12—C13	2.2 (2)	C12—C11—C16—C15	−1.0 (3)
N1—C11—C12—C13	−178.59 (14)	N1—C11—C16—C15	179.69 (16)
C11—C12—C13—C14	−1.9 (2)	O1—C1—C2—C3	1.3 (2)
C11—C12—C13—N13	177.29 (14)	N1—C1—C2—C3	−178.43 (15)
C14—C13—N13—O32	−3.1 (2)	C1—C2—C3—C4	−64.9 (2)
C12—C13—N13—O32	177.59 (16)	C2—C3—C4—O44	−22.3 (2)
C14—C13—N13—O31	176.42 (18)	C2—C3—C4—O43	159.71 (15)
C12—C13—N13—O31	−2.9 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.89	1.96	2.850 (2)	173
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Symmetry codes: (i) *x*, −*y*+1/2, *z*−1/2; (ii) −*x*+1, −*y*+1, −*z*+1.