Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-Nitrophenylacetic acid: hydrogen-bonded sheets of $R_2^2(8)$ and $R_4^4(18)$ rings

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.002 Å
R factor = 0.036
wR factor = 0.108
Data-to-parameter ratio = 15.2

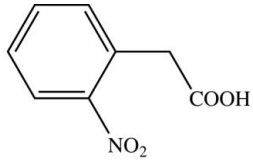
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 11 April 2006
Accepted 11 April 2006

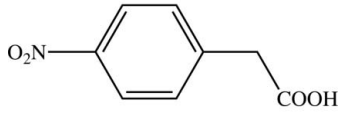
Molecules of the title compound, $C_8H_7NO_4$, are linked into centrosymmetric $R_2^2(8)$ dimers by paired O—H···O hydrogen bonds, and these dimers are linked by two C—H···O hydrogen bonds into sheets of $R_2^2(8)$ and $R_4^4(18)$ rings.

Comment

As part of our investigations of compounds containing nitro and carboxylic acid groups (Glidewell *et al.*, 2003*a*,*b*, 2004, 2006; Wardell *et al.*, 2005), we now report the molecular and supramolecular structure of 2-nitrophenylacetic acid, (I) (Fig. 1).



(I)



(II)

The plane of atoms C1/C11/C12 is almost orthogonal to the plane of the aryl ring (Fig. 1, Table 1), while the C—NO_2 plane makes a dihedral angle of 30.1 (2)° with the ring.

The molecules of (I) are linked into sheets by a combination of N—H···O and C—H···O hydrogen bonds (Table 2). Paired O—H···O hydrogen bonds link the molecules into centrosymmetric $R_2^2(8)$ (Bernstein *et al.*, 1995) dimers (Fig. 2). Two C—H···O hydrogen bonds link the dimers, so forming a (100) sheet built from $R_2^2(8)$ and $R_4^4(18)$ rings. The resulting net is of type (4,4) (Batten & Robson, 1998). There are no direction-specific interactions between adjacent sheets. In particular, C—H···π(arene) hydrogen bonds and aromatic π–π stacking interactions are both absent.

The structure of the isomeric 4-nitrophenylacetic acid, (II), was reported some years ago [Cambridge Structural Database (Version of November 2005; Allen, 2002) refcode SEMTAF; Grabowski *et al.*, 1990]. The authors reported the formation of a centrosymmetric hydrogen-bonded dimer, but further aggregation of the dimers was not reported. In the event, the dimers are linked into sheets by a single aromatic π–π stacking interaction (Fig. 4).

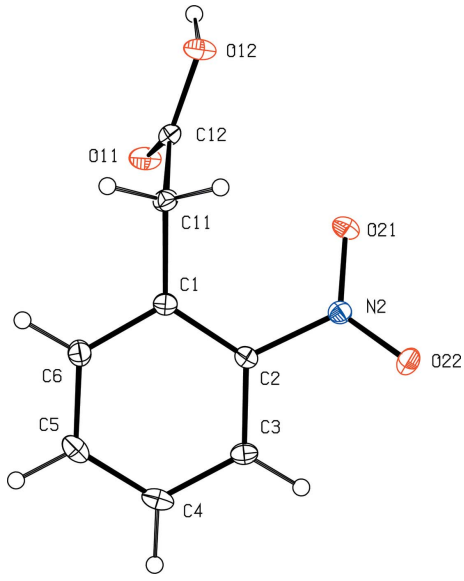


Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

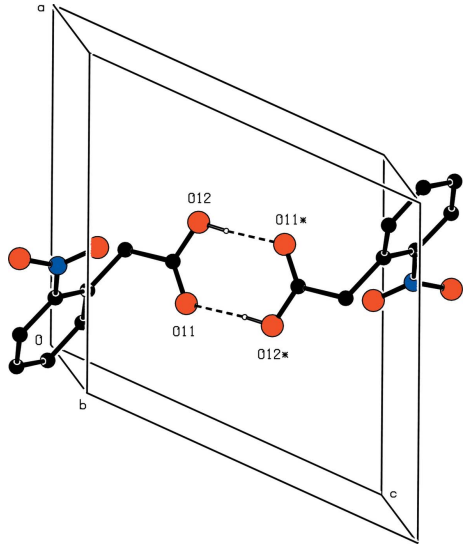


Figure 2
Part of the crystal structure of (I), showing the formation of a centrosymmetric $R_2^2(8)$ dimer. For the sake of clarity, H atoms bonded to C atoms have been omitted. Atoms marked with an asterisk (*) are at the symmetry position $(1-x, 1-y, 1-z)$.

Experimental

A commercial sample of (I) (Acros) was crystallized from ethanol (m.p. 412–413 K).

Crystal data

$C_8H_7NO_4$
M_r = 181.15
Monoclinic, $P2_1/c$
a = 9.3182 (3) Å
b = 9.4466 (2) Å
c = 9.9733 (3) Å
β = 114.7990 (17)°
V = 796.95 (4) Å^3
Z = 4
D_x = 1.510 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 120 (2) K
Lath, colourless
0.52 × 0.26 × 0.10 mm

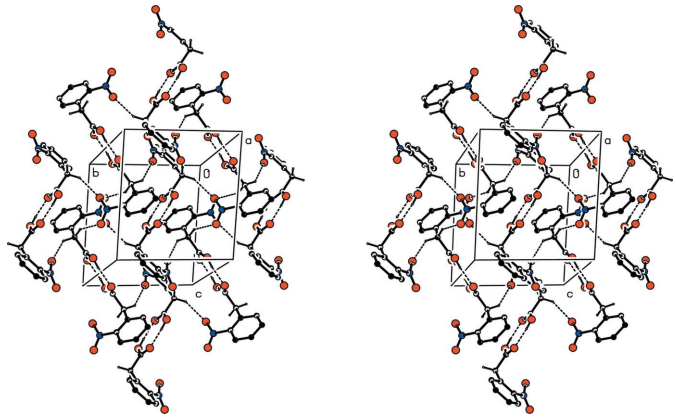


Figure 3
A stereoview of part of the crystal structure of (I), showing the formation of a (100) sheet of $R_2^2(8)$ and $R_4^4(18)$ rings. For the sake of clarity, H atoms bonded to aromatic C atoms have been omitted.

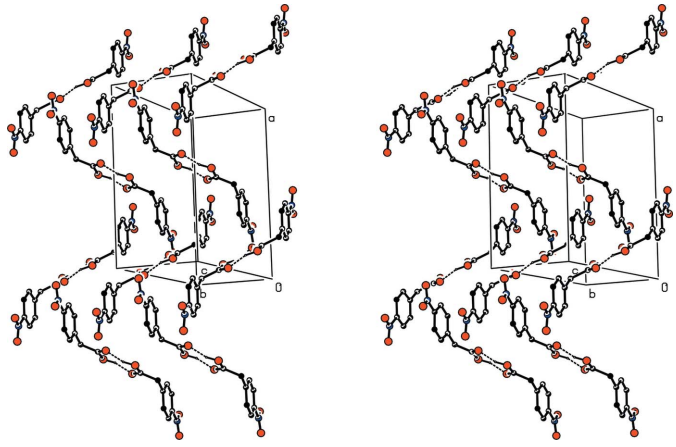


Figure 4
A stereoview of part of the crystal structure of (II), showing the formation of a sheet of π-stacked hydrogen-bonded dimers. The original atomic coordinates (Grabowski *et al.*, 1990) have been used. For the sake of clarity, H atoms bonded to C atoms have been omitted.

Data collection

Bruker Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.949, T_{max} = 0.988
8852 measured reflections
1829 independent reflections
1682 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 27.7°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.108
S = 1.16
1829 reflections
120 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0444P)^2 + 0.341P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.103 (10)

Table 1
Selected torsion angles (°).

C2—C1—C11—C12	83.34 (16)	C1—C2—N2—O21	−29.64 (17)
C1—C11—C12—O12	−159.51 (11)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O12—H12···O11^{i}	0.84	1.83	2.6622 (14)	173
C11—H11*A*···O21^{ii}	0.99	2.35	3.1758 (16)	140
C11—H11*B*···O22^{iii}	0.99	2.54	3.4398 (19)	151

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

All H atoms were located in a difference map and then treated as riding, with C—H distances of 0.95 Å (aromatic) or 0.99 Å (CH_2), and O—H distances of 0.84 Å, and with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(O)$.

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *OSCAIL* (McArdle, 2003) and *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *OSCAIL* and *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PRPKAPPA* (Ferguson, 1999).

The X-ray data were collected at the EPSRC X-Ray Crystallographic Service, University of Southampton, UK; the authors thank the staff of the Service for all their help and advice. JLW thanks CNPq and FAPERJ for financial support.

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supporting information

Acta Cryst. (2006). E**62**, o1915–o1917 [https://doi.org/10.1107/S1600536806013274]

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2-Nitrophenylacetic acid

Crystal data

$C_8H_7NO_4$
$M_r = 181.15$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 9.3182$ (3) Å
$b = 9.4466$ (2) Å
$c = 9.9733$ (3) Å
$\beta = 114.7990$ (17)°
$V = 796.95$ (4) Å^3
$Z = 4$
$F(000) = 376$
$D_x = 1.510$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1848 reflections
$\theta = 2.9$–27.5°
$\mu = 0.12$ mm^{-1}
$T = 120$ K
Lath, colourless
0.52 × 0.26 × 0.10 mm

Data collection

Bruker Nonius KappaCCD area-detector diffractometer
Radiation source: Bruker Nonius FR591 rotating anode
Graphite monochromator
Detector resolution: 9.091 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
$T_{min} = 0.949$, $T_{max} = 0.988$
8852 measured reflections
1829 independent reflections
1682 reflections with $I > 2\sigma(I)$
$R_{int} = 0.031$
$\theta_{max} = 27.7°$, $\theta_{min} = 3.2°$
$h = -12\rightarrow11$
$k = -11\rightarrow12$
$l = -11\rightarrow12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.108$
$S = 1.16$
1829 reflections
120 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
$w = 1/[\sigma^2(F_o^2) + (0.0444P)^2 + 0.341P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.34$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}
Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.103 (10)

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.23687 (14)	0.30836 (13)	0.07521 (14)	0.0171 (3)

C11	0.40523 (15)	0.29551 (14)	0.18979 (14)	0.0196 (3)
C12	0.44276 (14)	0.38895 (14)	0.32300 (14)	0.0191 (3)
O11	0.34184 (11)	0.43438 (11)	0.35993 (11)	0.0260 (3)
O12	0.59458 (11)	0.41254 (11)	0.39762 (11)	0.0254 (3)
C2	0.18252 (14)	0.41302 (13)	−0.03303 (14)	0.0161 (3)
N2	0.28971 (12)	0.52395 (11)	−0.03878 (12)	0.0181 (3)
O21	0.39974 (11)	0.56059 (10)	0.07718 (11)	0.0238 (3)
O22	0.26447 (12)	0.57548 (11)	−0.15940 (11)	0.0287 (3)
C3	0.02718 (15)	0.42023 (14)	−0.13979 (14)	0.0191 (3)
C4	−0.07970 (15)	0.31978 (15)	−0.13815 (15)	0.0233 (3)
C5	−0.03015 (16)	0.21349 (15)	−0.03267 (16)	0.0252 (3)
C6	0.12610 (16)	0.20739 (14)	0.07154 (15)	0.0221 (3)
H11A	0.4257	0.1958	0.2228	0.024*
H11B	0.4775	0.3199	0.1435	0.024*
H12	0.6100	0.4558	0.4761	0.038*
H3	−0.0046	0.4929	−0.2123	0.023*
H4	−0.1866	0.3235	−0.2090	0.028*
H5	−0.1035	0.1442	−0.0315	0.030*
H6	0.1581	0.1327	0.1418	0.027*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0180 (6)	0.0173 (6)	0.0159 (6)	0.0001 (4)	0.0071 (5)	−0.0028 (5)
C11	0.0185 (6)	0.0190 (6)	0.0184 (6)	0.0023 (4)	0.0048 (5)	0.0015 (5)
C12	0.0175 (6)	0.0211 (6)	0.0163 (6)	0.0006 (5)	0.0047 (5)	0.0043 (5)
O11	0.0179 (5)	0.0390 (6)	0.0195 (5)	−0.0005 (4)	0.0064 (4)	−0.0050 (4)
O12	0.0159 (5)	0.0363 (6)	0.0202 (5)	−0.0015 (4)	0.0038 (4)	−0.0065 (4)
C2	0.0156 (6)	0.0167 (6)	0.0168 (6)	−0.0007 (4)	0.0076 (5)	−0.0030 (5)
N2	0.0160 (5)	0.0173 (5)	0.0202 (5)	0.0021 (4)	0.0069 (4)	0.0010 (4)
O21	0.0209 (5)	0.0234 (5)	0.0228 (5)	−0.0064 (4)	0.0049 (4)	−0.0045 (4)
O22	0.0255 (5)	0.0333 (6)	0.0250 (5)	0.0000 (4)	0.0082 (4)	0.0130 (4)
C3	0.0177 (6)	0.0211 (6)	0.0166 (6)	0.0024 (4)	0.0054 (5)	−0.0031 (5)
C4	0.0153 (6)	0.0282 (7)	0.0234 (7)	−0.0015 (5)	0.0052 (5)	−0.0098 (5)
C5	0.0225 (7)	0.0258 (7)	0.0295 (7)	−0.0083 (5)	0.0132 (6)	−0.0075 (6)
C6	0.0252 (7)	0.0204 (6)	0.0219 (7)	−0.0031 (5)	0.0109 (5)	−0.0002 (5)

Geometric parameters (Å, º)

C1—C2	1.3931 (18)	C2—N2	1.4653 (16)
C1—C6	1.3947 (18)	N2—O22	1.2256 (15)
C1—C11	1.5092 (17)	N2—O21	1.2303 (14)
C11—C12	1.5089 (18)	C3—C4	1.3807 (19)
C11—H11A	0.99	C3—H3	0.95
C11—H11B	0.99	C4—C5	1.386 (2)
C12—O11	1.2226 (17)	C4—H4	0.95
C12—O12	1.3121 (15)	C5—C6	1.3907 (19)
O12—H12	0.84	C5—H5	0.95

C2—C3	1.3930 (17)	C6—H6	0.95
C2—C1—C6	116.14 (11)	O22—N2—O21	123.51 (11)
C2—C1—C11	124.64 (11)	O22—N2—C2	117.97 (10)
C6—C1—C11	119.19 (11)	O21—N2—C2	118.51 (10)
C12—C11—C1	113.77 (10)	C4—C3—C2	118.74 (12)
C12—C11—H11A	108.8	C4—C3—H3	120.6
C1—C11—H11A	108.8	C2—C3—H3	120.6
C12—C11—H11B	108.8	C3—C4—C5	119.64 (12)
C1—C11—H11B	108.8	C3—C4—H4	120.2
H11A—C11—H11B	107.7	C5—C4—H4	120.2
O11—C12—O12	123.51 (12)	C4—C5—C6	120.56 (12)
O11—C12—C11	123.17 (11)	C4—C5—H5	119.7
O12—C12—C11	113.29 (11)	C6—C5—H5	119.7
C12—O12—H12	109.5	C5—C6—C1	121.51 (13)
C3—C2—C1	123.40 (12)	C5—C6—H6	119.2
C3—C2—N2	116.23 (11)	C1—C6—H6	119.2
C1—C2—N2	120.37 (10)		
C2—C1—C11—C12	83.34 (16)	C3—C2—N2—O21	150.02 (12)
C6—C1—C11—C12	−98.81 (14)	C1—C2—N2—O21	−29.64 (17)
C1—C11—C12—O11	22.60 (18)	C1—C2—C3—C4	0.71 (19)
C1—C11—C12—O12	−159.51 (11)	N2—C2—C3—C4	−178.94 (11)
C6—C1—C2—C3	0.43 (19)	C2—C3—C4—C5	−0.97 (19)
C11—C1—C2—C3	178.34 (12)	C3—C4—C5—C6	0.1 (2)
C6—C1—C2—N2	−179.94 (11)	C4—C5—C6—C1	1.1 (2)
C11—C1—C2—N2	−2.03 (19)	C2—C1—C6—C5	−1.32 (19)
C3—C2—N2—O22	−29.86 (16)	C11—C1—C6—C5	−179.35 (12)
C1—C2—N2—O22	150.48 (12)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O12—H12···O11^{i}	0.84	1.83	2.6622 (14)	173
C11—H11*A*···O21^{ii}	0.99	2.35	3.1758 (16)	140
C11—H11*B*···O22^{iii}	0.99	2.54	3.4398 (19)	151

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) −*x*+1, *y*−1/2, −*z*+1/2; (iii) −*x*+1, −*y*+1, −*z*.