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3-[(4-Nitrophenyl)aminocarbonyl]-propanoic acid

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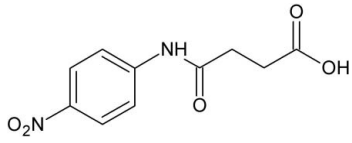
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.001 Å; R factor = 0.035; wR factor = 0.097; data-to-parameter ratio = 15.8.

The title compound, $C_{10}H_{10}N_2O_5$, crystallizes in the centrosymmetric space group $P2_1/c$. The carboxyl terminus end is almost perpendicular to the rest of the molecule [the dihedral angle between mean planes through the terminal COOH group, the adjacent C atom and rest of the molecule, except for the methylene H atoms, is 82.15 (18)°]. This molecule fails to show second-order nonlinear optical property due to the presence of inversion symmetry in the solid state. The crystal structure is stabilized by strong intermolecular O—H···O and N—H···O hydrogen bonding.

Related literature

For related literature, see: Glidewell *et al.* (2005); Munn & Ironside (1993); Ravindra *et al.* (2006); Brunton & Jones (2000).



Experimental

Crystal data

$C_{10}H_{10}N_2O_5$
M_r = 238.20
Monoclinic, $P2_1/c$
a = 5.0179 (4) Å
b = 17.3056 (15) Å
c = 11.5804 (9) Å
β = 94.487 (4)°
V = 1002.53 (14) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 100 (2) K
0.33 × 0.27 × 0.22 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.959, T_{max} = 0.972
30488 measured reflections
3066 independent reflections
2686 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.097
S = 1.06
3066 reflections
194 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

Table 1
Selected torsion angles (°).

C2—C3—C4—O3	−18.34 (12)	O4—N2—C8—C7	−5.76 (14)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2^{i}	0.863 (19)	1.843 (19)	2.7002 (10)	172.5 (17)
N1—H1A···O4^{ii}	0.852 (15)	2.536 (14)	3.3378 (11)	157.1 (12)
N1—H1A···O5^{ii}	0.852 (15)	2.616 (15)	3.3711 (12)	148.4 (12)

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2005); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KJ2061).

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supplementary materials

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3-[(4-Nitrophenyl)aminocarbonyl]propanoic acid

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Comment

Crystallization in a noncentrosymmetric space group is required for organic nonlinear optical (NLO) materials to exhibit efficient second harmonic generation (SHG) (Munn *et al.*, 1993). Our interest in organic nonlinear optical materials, particularly in acid anhydride derivatives (Ravindra *et al.*, 2006) of nitroaniline, has led us to synthesize the title compound. This compound crystallizes in a centrosymmetric structure and hence exhibits no second harmonic generation. However, the molecule shows third order nonlinear response (nonlinear absorption), which can be observed in molecules with both centrosymmetic and noncentrosymmetric structures.

A projection view of the molecule with 50% probability displacement ellipsoids is shown in Fig 1. The C2—C3 bond length is in good agreement with the three isomeric *N*-(*p*-chlorophenyl)succinimides (Glidewell *et al.*, 2005) which display C—C bond lengths of 1.5276 (19), 1.518 (3) and 1.524 (5) Å. The nitro and amine groups attached to C8 and C5 are almost coplanar with the C5—C10 benzene ring, with O5—N2—C8—C9 and C4—N1—C5—C10 torsion angles of 5.65 (14)° and 6.43 (15)°,respectively.

The hydrogen bonding parameters are listed in Table 2. A s expected, the carboxyl groups of two molecules form strong intermolecular hydrogen bonding resulting in the head to tail dimers. The N—H hydrogen atom forms bifurcated intermolecular hydrogen bonds to the NO_2 group of a second molecule. Thus the crystal structure is stabilized by the formation of two-dimensional networks involving bifurcated and dimeric hydrogen bonds involving N1—H1A···O4, N1—H1A···O5 and O1—H1···O1. The extended hydrogen bonding network results in a step like arrangement between the layers in the crystal lattice with the carboxyl dimers as the steps.

Experimental

The title compound (I) was prepared by the method reported by Brunton & Jones (2000). The compound was purified by recrystallization from ethanol. The crystals used for single-crystal X-ray studies were grown by slow evaporation of an acetone solution of the purified compound.

Refinement

H-atom positions were located from difference Fourier maps and all associated parameters were refined freely. Refined C—H distances were in the range 0.949 (14)–0.995 (14) Å, where as N—H and O—H distances are 0.852 (15) Å and 0.863 (19) Å respectively.

Figures

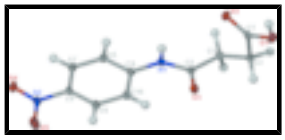

Fig. 1. The molecules of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level.

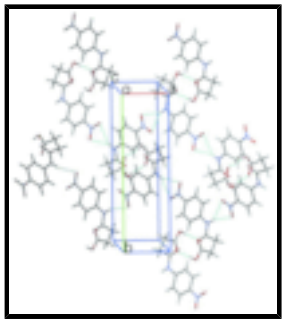

Fig. 2. Molecular packing diagram as viewed down *a* axis, showing the hydrogen bonding (dashed lines).

3-[(4-Nitrophenyl)aminocarbonyl]propanoic acid

Crystal data

$C_{10}H_{10}N_2O_5$	F_{000} = 496
M_r = 238.20	D_x = 1.578 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 6073 reflections
a = 5.0179 (4) Å	θ = 2.4–30.6°
b = 17.3056 (15) Å	μ = 0.13 mm^{-1}
c = 11.5804 (9) Å	T = 100 (2) K
β = 94.487 (4)°	Irregular, colourless
V = 1002.53 (14) Å^3	0.33 × 0.27 × 0.22 mm
Z = 4	

Data collection

Bruker APEXII CCD area-detector diffractometer	3066 independent reflections
Radiation source: fine-focus sealed tube	2686 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.031
T = 100(2) K	θ_{max} = 30.5°
φ and ω scans	θ_{min} = 2.1°
Absorption correction: multi-scan (SADABS; Bruker, 2005)	h = −6→7
T_{min} = 0.959, T_{max} = 0.972	k = −24→24
30488 measured reflections	l = −16→16

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.035$	All H-atom parameters refined
$wR(F^2) = 0.097$	$w = 1/[\sigma^2(F_o^2) + (0.0495P)^2 + 0.3297P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} = 0.001$
3066 reflections	$\Delta\rho_{max} = 0.39$ e Å^{-3}
194 parameters	$\Delta\rho_{min} = -0.26$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Experimental. All H atoms were located and refined freely.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2$sigma(F^2) is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.31721 (15)	0.59418 (4)	0.95848 (7)	0.01813 (16)
O2	0.25598 (14)	0.46856 (4)	0.91943 (6)	0.01693 (16)
O3	0.20764 (14)	0.50534 (4)	0.63580 (6)	0.01685 (16)
O4	0.47768 (17)	0.15785 (4)	0.28173 (7)	0.02248 (18)
O5	0.68389 (18)	0.26212 (5)	0.23997 (8)	0.0289 (2)
N1	−0.02051 (16)	0.39108 (5)	0.62342 (7)	0.01322 (16)
N2	0.52247 (17)	0.22718 (5)	0.29519 (7)	0.01579 (17)
C1	0.19088 (18)	0.53594 (5)	0.90384 (8)	0.01307 (18)
C2	−0.04231 (18)	0.55979 (6)	0.82212 (8)	0.01367 (18)
C3	−0.15950 (19)	0.49103 (6)	0.75392 (8)	0.01358 (18)
C4	0.02909 (18)	0.46387 (5)	0.66623 (8)	0.01233 (17)
C5	0.12121 (18)	0.35175 (5)	0.54208 (8)	0.01224 (17)
C6	0.05349 (19)	0.27432 (5)	0.51835 (8)	0.01431 (18)
C7	0.18377 (19)	0.23258 (6)	0.43764 (8)	0.01480 (18)
C8	0.38209 (19)	0.26926 (5)	0.38056 (8)	0.01336 (18)
C9	0.45402 (19)	0.34517 (6)	0.40309 (8)	0.01467 (18)
C10	0.32460 (19)	0.38663 (6)	0.48410 (8)	0.01455 (18)

H1	0.446 (4)	0.5748 (11)	1.0030 (16)	0.045 (5)*
H1A	−0.147 (3)	0.3663 (8)	0.6518 (12)	0.022 (3)*
H2A	−0.175 (3)	0.5839 (8)	0.8700 (12)	0.021 (3)*
H2B	0.022 (3)	0.5992 (8)	0.7723 (12)	0.022 (3)*
H3A	−0.327 (3)	0.5060 (8)	0.7124 (12)	0.020 (3)*
H3B	−0.198 (3)	0.4475 (8)	0.8060 (12)	0.020 (3)*
H6	−0.084 (3)	0.2505 (8)	0.5578 (12)	0.022 (3)*
H7	0.135 (3)	0.1795 (9)	0.4192 (12)	0.023 (3)*
H9	0.593 (3)	0.3686 (8)	0.3641 (13)	0.025 (4)*
H10	0.375 (3)	0.4392 (8)	0.5002 (12)	0.020 (3)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0185 (3)	0.0143 (3)	0.0208 (3)	−0.0005 (3)	−0.0030 (3)	−0.0024 (3)
O2	0.0178 (3)	0.0139 (3)	0.0186 (3)	0.0014 (3)	−0.0016 (3)	−0.0013 (3)
O3	0.0156 (3)	0.0158 (3)	0.0200 (3)	−0.0042 (3)	0.0065 (3)	−0.0034 (3)
O4	0.0297 (4)	0.0142 (3)	0.0242 (4)	0.0033 (3)	0.0061 (3)	−0.0035 (3)
O5	0.0329 (5)	0.0281 (4)	0.0285 (4)	−0.0060 (3)	0.0208 (4)	−0.0062 (3)
N1	0.0126 (4)	0.0135 (4)	0.0142 (4)	−0.0030 (3)	0.0053 (3)	−0.0010 (3)
N2	0.0164 (4)	0.0169 (4)	0.0144 (4)	0.0024 (3)	0.0029 (3)	−0.0016 (3)
C1	0.0127 (4)	0.0152 (4)	0.0119 (4)	−0.0005 (3)	0.0042 (3)	−0.0013 (3)
C2	0.0137 (4)	0.0139 (4)	0.0135 (4)	0.0015 (3)	0.0021 (3)	−0.0015 (3)
C3	0.0118 (4)	0.0155 (4)	0.0137 (4)	−0.0003 (3)	0.0029 (3)	−0.0010 (3)
C4	0.0118 (4)	0.0135 (4)	0.0117 (4)	0.0004 (3)	0.0005 (3)	−0.0002 (3)
C5	0.0124 (4)	0.0129 (4)	0.0115 (4)	0.0005 (3)	0.0018 (3)	−0.0001 (3)
C6	0.0144 (4)	0.0132 (4)	0.0158 (4)	−0.0023 (3)	0.0044 (3)	0.0007 (3)
C7	0.0165 (4)	0.0123 (4)	0.0159 (4)	−0.0006 (3)	0.0026 (3)	0.0001 (3)
C8	0.0143 (4)	0.0139 (4)	0.0122 (4)	0.0024 (3)	0.0031 (3)	−0.0010 (3)
C9	0.0145 (4)	0.0151 (4)	0.0150 (4)	−0.0018 (3)	0.0047 (3)	−0.0003 (3)
C10	0.0157 (4)	0.0132 (4)	0.0153 (4)	−0.0028 (3)	0.0043 (3)	−0.0013 (3)

Geometric parameters (Å, °)

O1—C1	1.3248 (12)	C3—C4	1.5159 (13)
O1—H1	0.863 (19)	C3—H3A	0.970 (14)
O2—C1	1.2206 (12)	C3—H3B	0.995 (14)
O3—C4	1.2212 (11)	C5—C10	1.4013 (12)
O4—N2	1.2283 (11)	C5—C6	1.4043 (13)
O5—N2	1.2295 (12)	C6—C7	1.3851 (13)
N1—C4	1.3695 (12)	C6—H6	0.949 (14)
N1—C5	1.4004 (11)	C7—C8	1.3904 (13)
N1—H1A	0.852 (15)	C7—H7	0.971 (15)
N2—C8	1.4536 (12)	C8—C9	1.3818 (13)
C1—C2	1.5039 (13)	C9—C10	1.3826 (13)
C2—C3	1.5208 (13)	C9—H9	0.952 (14)
C2—H2A	0.991 (14)	C10—H10	0.959 (14)
C2—H2B	0.964 (14)		

C1—O1—H1	107.4 (12)	O3—C4—N1	123.55 (9)
C4—N1—C5	127.09 (8)	O3—C4—C3	121.45 (8)
C4—N1—H1A	116.3 (9)	N1—C4—C3	114.99 (8)
C5—N1—H1A	116.6 (10)	N1—C5—C10	122.70 (8)
O4—N2—O5	122.40 (9)	N1—C5—C6	117.87 (8)
O4—N2—C8	118.82 (8)	C10—C5—C6	119.43 (8)
O5—N2—C8	118.78 (8)	C7—C6—C5	120.66 (8)
O2—C1—O1	122.93 (9)	C7—C6—H6	119.7 (9)
O2—C1—C2	122.77 (9)	C5—C6—H6	119.6 (9)
O1—C1—C2	114.30 (8)	C6—C7—C8	118.41 (9)
C1—C2—C3	111.24 (8)	C6—C7—H7	121.3 (8)
C1—C2—H2A	106.7 (8)	C8—C7—H7	120.3 (8)
C3—C2—H2A	111.8 (8)	C9—C8—C7	122.03 (8)
C1—C2—H2B	106.9 (9)	C9—C8—N2	118.13 (8)
C3—C2—H2B	112.1 (8)	C7—C8—N2	119.84 (8)
H2A—C2—H2B	107.9 (12)	C8—C9—C10	119.45 (8)
C4—C3—C2	110.74 (8)	C8—C9—H9	120.4 (9)
C4—C3—H3A	108.2 (8)	C10—C9—H9	120.2 (9)
C2—C3—H3A	109.5 (8)	C9—C10—C5	120.00 (9)
C4—C3—H3B	109.5 (8)	C9—C10—H10	119.6 (8)
C2—C3—H3B	111.4 (8)	C5—C10—H10	120.4 (8)
H3A—C3—H3B	107.4 (11)		
O2—C1—C2—C3	−6.53 (12)	C6—C7—C8—C9	0.80 (15)
O1—C1—C2—C3	174.30 (8)	C6—C7—C8—N2	−179.90 (8)
C1—C2—C3—C4	−70.73 (10)	O4—N2—C8—C9	173.57 (9)
C5—N1—C4—O3	1.31 (15)	O5—N2—C8—C9	−5.65 (14)
C5—N1—C4—C3	−179.94 (8)	O4—N2—C8—C7	−5.76 (14)
C2—C3—C4—O3	−18.34 (12)	O5—N2—C8—C7	175.02 (9)
C2—C3—C4—N1	162.88 (8)	C7—C8—C9—C10	−0.51 (15)
C4—N1—C5—C10	−6.43 (15)	N2—C8—C9—C10	−179.82 (9)
C4—N1—C5—C6	173.81 (9)	C8—C9—C10—C5	−0.38 (15)
N1—C5—C6—C7	179.13 (9)	N1—C5—C10—C9	−178.81 (9)
C10—C5—C6—C7	−0.64 (14)	C6—C5—C10—C9	0.94 (14)
C5—C6—C7—C8	−0.22 (14)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2^{i}	0.863 (19)	1.843 (19)	2.7002 (10)	172.5 (17)
N1—H1A···O4^{ii}	0.852 (15)	2.536 (14)	3.3378 (11)	157.1 (12)
N1—H1A···O5^{ii}	0.852 (15)	2.616 (15)	3.3711 (12)	148.4 (12)

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

Fig. 1

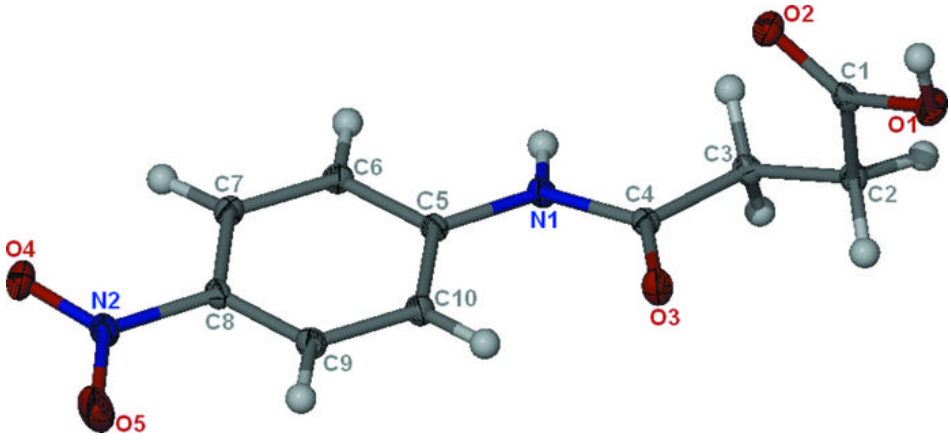

Fig. 2