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A hydrogen-bonded chain of rings in (*E*)-3-(4-nitrophenylaminocarbonyl)prop-2-enoic acid

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Key indicators

Single-crystal X-ray study
T = 291 K
Mean σ(C–C) = 0.003 Å
R factor = 0.046
wR factor = 0.116
Data-to-parameter ratio = 17.4

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

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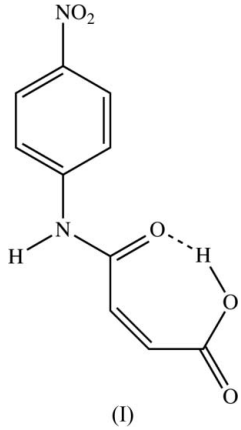
In the title compound, $C_{10}H_8N_2O_5$, the molecules are linked into chains of rings by the concerted action of one N—H···O and one C—H···O hydrogen bond.

Comment

We have recently reported the molecular and supramolecular structures of two 2-(*X*-nitrophenylaminocarbonyl)benzoic acids (*X* = 2 and 4), formed from phthalic anhydride and the appropriate nitroaniline (Glidewell *et al.*, 2004). We now report the related compound (*E*)-3-(4-nitrophenylaminocarbonyl)propen-2-oic acid, (I) (Fig. 1), formed from maleic anhydride and 4-nitroaniline. Although the molecule of (I) is simple, it contains a number of potential sites for involvement in intermolecular interactions; in particular, hard and soft hydrogen bonds and aromatic π-π stacking interactions are possible.



(I)

There are two intramolecular hydrogen bonds in compound (I). A rather short and almost linear O—H···O hydrogen bond (Fig. 1) controls the conformation of the maleate fragment, forming an *S*(7) ring, while a weaker C—H···O hydrogen bond forming an *S*(6) ring appears to control the mutual orientation of the maleate and nitroaryl fragments. Accordingly, the molecular skeleton is almost planar, as shown by the relevant torsion angles (Table 1). The bond distances within the acyclic portion of the molecule (Table 1) clearly show the location of single and double bonds; the C—O distances in the carboxylic acid group are consistent with the location of the carboxyl H atom deduced from difference maps.

The molecules of (I) are linked into chains of rings by an N—H···O hydrogen bond, augmented by a rather long C—H···O hydrogen bond. Amide atom N1 and aryl atom C16 in the molecule at (*x*, *y*, *z*) both act as hydrogen-bond donors to

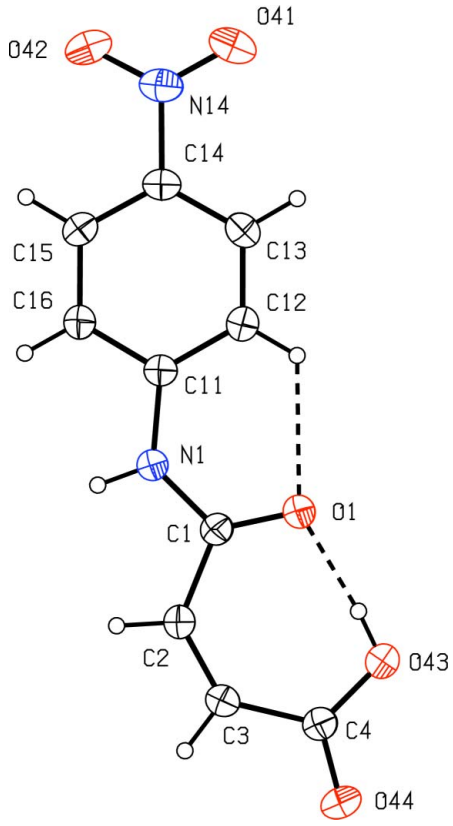


Figure 1
The molecule of compound (I), showing displacement ellipsoids drawn at the 30% probability level. The dashed line indicates a hydrogen bond.

carboxyl atom O44 in the molecule at $(1-x, \frac{1}{2}+y, \frac{3}{2}-z)$, so forming a $C(7)C(9)[R_1^2(6)]$ (Bernstein *et al.*, 1995) chain of rings running parallel to the [010] direction and generated by the 2_1 screw axis along $(\frac{1}{2}, y, \frac{3}{4})$ (Fig. 2). Two chains of this type pass through each unit cell but there are no significant direction-specific interactions between adjacent chains; in particular, C—H···π(arene) hydrogen bonds and aromatic π–π stacking interactions are absent.

Experimental

A solution containing equimolar quantities of maleic anhydride and 4-nitroaniline (2 mmol of each) in diethyl ether (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. 472–474 K). IR (cm^{-1}): 3200–2000 (*br*), 1707, 1635, 1596, 1566, 1497, 1457, 1407, 1335, 1307, 1271, 1230, 1110, 971, 898, 863, 797, 750, 687, 610, 597, 501, 432.

Crystal data

$C_{10}H_8N_2O_5$
$M_r = 236.18$
Monoclinic, $P2_1/c$
$a = 9.6052\ (7)$ Å
$b = 12.8416\ (10)$ Å
$c = 9.0921\ (7)$ Å
$\beta = 114.388\ (2)^\circ$
$V = 1021.41\ (13)$ Å^3
$Z = 4$
$D_x = 1.536$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2702 reflections
$\theta = 2.8$–29.0°
$\mu = 0.13$ mm^{-1}
$T = 291\ (2)$ K
Block, yellow
0.27 × 0.14 × 0.10 mm

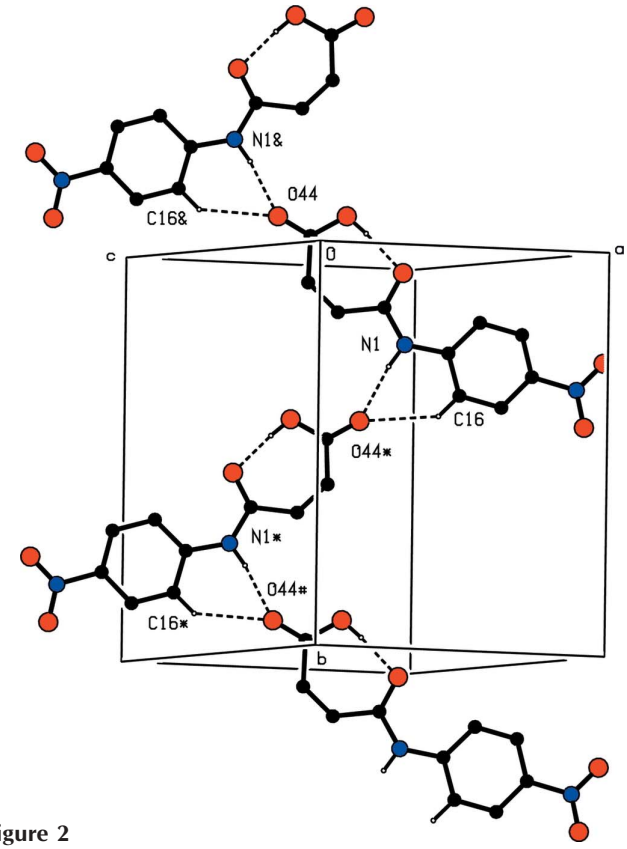


Figure 2
Part of the crystal structure of (I), showing the formation of a chain of rings along [010]. Atoms marked with an asterisk (*) or an ampersand (&) are at the symmetry positions $(1-x, \frac{1}{2}+y, \frac{3}{2}-z)$ and $(1-x, -\frac{1}{2}+y, \frac{3}{2}-z)$, respectively. Dashed lines indicate hydrogen bonds. H atoms have been omitted.

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min} = 0.956$, $T_{max} = 0.988$
8437 measured reflections
2702 independent reflections
1379 reflections with $I > 2\sigma(I)$
$R_{int} = 0.042$
$\theta_{max} = 29.0^\circ$
$h = -13 \rightarrow 13$
$k = -17 \rightarrow 17$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.116$
$S = 0.87$
2702 reflections
155 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0597P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.16$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—O1	1.2413 (19)	C3—C4	1.482 (3)
C1—C2	1.478 (2)	C4—O43	1.307 (2)
C2—C3	1.328 (2)	C4—O44	1.206 (2)
C13—C14—N14—O41	−6.8 (3)	N1—C1—C2—C3	175.98 (19)
C12—C11—N1—C1	−0.5 (3)	C2—C3—C4—O43	3.3 (3)
C11—N1—C1—C2	177.37 (17)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O43—H43···O1	0.82	1.72	2.537 (2)	174
C12—H12···O1	0.93	2.31	2.899 (2)	121
N1—H1···O44^{i}	0.86	1.96	2.814 (2)	172
C16—H16···O44^{i}	0.93	2.50	3.260 (2)	139

Symmetry code: (i) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$.

All H atoms were located in difference maps and then treated as riding atoms, with distances C—H = 0.93 Å, N—H 0.86 Å and O—H = 0.82 Å, and with U_{iso}(H) = 1.2U_{eq}(C,N) or 1.5U_{eq}(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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References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Bruker (2000). *SMART* (Version 5.624), *SAINT-Plus* (Version 6.02A) and *SADABS* (Version 2.03), Bruker AXS Inc., Madison, Wisconsin, USA.
Ferguson, G. (1999). *PRPKAPPA*. University of Guelph, Canada.
Glidewell, C., Low, J. N., Skakle, J. M. S. & Wardell, J. L. (2004). *Acta Cryst.* C**60**, o120–o124.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

supporting information

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0.27 × 0.14 × 0.10 mm

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Graphite monochromator
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Absorption correction: multi-scan (SADABS; Bruker, 2000)
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$\theta_{max} = 29.0°$, $\theta_{min} = 2.8°$
$h = -13 \rightarrow 13$
$k = -17 \rightarrow 17$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.116$
$S = 0.87$
2702 reflections
155 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
$w = 1/[\sigma^2(F_o^2) + (0.0597P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.16$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.72801 (13)	0.03077 (9)	0.65778 (16)	0.0570 (4)

O41	1.28952 (17)	0.24682 (13)	0.4548 (2)	0.0831 (5)
O42	1.22748 (17)	0.40730 (12)	0.45950 (19)	0.0764 (5)
O43	0.54851 (15)	−0.10562 (9)	0.68397 (18)	0.0606 (4)
O44	0.36370 (16)	−0.10330 (10)	0.7637 (2)	0.0779 (5)
N1	0.74726 (15)	0.20691 (11)	0.67282 (18)	0.0472 (4)
N14	1.21330 (18)	0.31419 (15)	0.4797 (2)	0.0592 (4)
C1	0.68626 (19)	0.11613 (13)	0.6895 (2)	0.0452 (4)
C2	0.5665 (2)	0.12856 (14)	0.7505 (2)	0.0511 (5)
C3	0.47741 (19)	0.05686 (14)	0.7728 (2)	0.0518 (5)
C4	0.4612 (2)	−0.05654 (14)	0.7397 (2)	0.0513 (5)
C11	0.86599 (18)	0.22698 (13)	0.6247 (2)	0.0432 (4)
C12	0.9471 (2)	0.15067 (14)	0.5844 (2)	0.0525 (5)
C13	1.0627 (2)	0.17921 (15)	0.5391 (2)	0.0535 (5)
C14	1.09543 (19)	0.28272 (14)	0.5340 (2)	0.0473 (4)
C15	1.01897 (19)	0.35913 (14)	0.5764 (2)	0.0506 (5)
C16	0.9034 (2)	0.33121 (13)	0.6206 (2)	0.0490 (5)
H1	0.7074	0.2613	0.6951	0.057*
H2	0.5513	0.1963	0.7771	0.061*
H3	0.4131	0.0827	0.8175	0.062*
H12	0.9237	0.0807	0.5879	0.063*
H13	1.1176	0.1287	0.5124	0.064*
H15	1.0449	0.4288	0.5752	0.061*
H16	0.8499	0.3824	0.6480	0.059*
H43	0.6074	−0.0641	0.6711	0.091*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0632 (8)	0.0394 (7)	0.0860 (10)	0.0039 (6)	0.0485 (8)	0.0025 (6)
O41	0.0746 (10)	0.0901 (12)	0.1107 (13)	0.0030 (8)	0.0647 (11)	−0.0045 (9)
O42	0.0798 (10)	0.0701 (11)	0.0976 (12)	−0.0170 (8)	0.0549 (10)	0.0073 (8)
O43	0.0678 (9)	0.0419 (7)	0.0883 (10)	−0.0045 (6)	0.0485 (8)	−0.0033 (7)
O44	0.0809 (10)	0.0540 (9)	0.1270 (14)	−0.0141 (7)	0.0711 (10)	0.0005 (8)
N1	0.0492 (9)	0.0366 (8)	0.0661 (10)	0.0007 (6)	0.0343 (8)	0.0008 (7)
N14	0.0509 (10)	0.0737 (12)	0.0605 (11)	−0.0059 (8)	0.0306 (9)	−0.0009 (9)
C1	0.0464 (10)	0.0390 (10)	0.0559 (11)	0.0013 (8)	0.0268 (9)	0.0046 (8)
C2	0.0551 (11)	0.0375 (9)	0.0710 (13)	0.0022 (8)	0.0364 (11)	0.0010 (9)
C3	0.0532 (11)	0.0458 (11)	0.0689 (13)	0.0022 (8)	0.0379 (11)	0.0011 (9)
C4	0.0511 (11)	0.0440 (10)	0.0664 (13)	0.0000 (8)	0.0318 (10)	0.0040 (9)
C11	0.0434 (10)	0.0420 (10)	0.0487 (11)	−0.0006 (7)	0.0238 (9)	0.0008 (8)
C12	0.0559 (11)	0.0395 (10)	0.0705 (13)	−0.0012 (8)	0.0344 (11)	−0.0020 (9)
C13	0.0524 (11)	0.0507 (11)	0.0662 (13)	0.0033 (8)	0.0332 (11)	−0.0052 (9)
C14	0.0431 (10)	0.0546 (11)	0.0488 (11)	−0.0034 (8)	0.0235 (9)	0.0014 (9)
C15	0.0543 (11)	0.0425 (10)	0.0611 (12)	−0.0047 (8)	0.0300 (11)	0.0032 (9)
C16	0.0551 (11)	0.0385 (10)	0.0616 (12)	0.0023 (8)	0.0324 (10)	0.0012 (8)

Geometric parameters (Å, º)

C11—C16	1.391 (2)	N14—O42	1.226 (2)
C11—C12	1.392 (2)	N1—C1	1.341 (2)
C11—N1	1.4037 (19)	N1—H1	0.86
C12—C13	1.383 (2)	C1—O1	1.2413 (19)
C12—H12	0.93	C1—C2	1.478 (2)
C13—C14	1.371 (3)	C2—C3	1.328 (2)
C13—H13	0.93	C2—H2	0.93
C14—C15	1.373 (2)	C3—C4	1.482 (3)
C14—N14	1.467 (2)	C3—H3	0.93
C15—C16	1.375 (2)	C4—O43	1.307 (2)
C15—H15	0.93	C4—O44	1.206 (2)
C16—H16	0.93	O43—H43	0.82
N14—O41	1.214 (2)		
C16—C11—C12	119.45 (15)	O41—N14—C14	118.40 (17)
C16—C11—N1	115.95 (14)	O42—N14—C14	117.92 (16)
C12—C11—N1	124.60 (15)	C1—N1—C11	130.08 (14)
C13—C12—C11	119.81 (16)	C1—N1—H1	115.0
C13—C12—H12	120.1	C11—N1—H1	115.0
C11—C12—H12	120.1	O1—C1—N1	122.92 (15)
C14—C13—C12	119.36 (16)	O1—C1—C2	123.96 (15)
C14—C13—H13	120.3	N1—C1—C2	113.12 (15)
C12—C13—H13	120.3	C3—C2—C1	129.25 (17)
C13—C14—C15	121.78 (15)	C3—C2—H2	115.4
C13—C14—N14	119.88 (16)	C1—C2—H2	115.4
C15—C14—N14	118.33 (16)	C2—C3—C4	132.11 (17)
C14—C15—C16	119.06 (16)	C2—C3—H3	113.9
C14—C15—H15	120.5	C4—C3—H3	113.9
C16—C15—H15	120.5	O44—C4—O43	120.03 (17)
C15—C16—C11	120.52 (16)	O44—C4—C3	118.49 (17)
C15—C16—H16	119.7	O43—C4—C3	121.47 (15)
C11—C16—H16	119.7	C4—O43—H43	109.5
O41—N14—O42	123.67 (16)		
C16—C11—C12—C13	−0.7 (3)	C13—C14—N14—O42	171.96 (18)
N1—C11—C12—C13	−179.74 (17)	C15—C14—N14—O42	−7.1 (3)
C11—C12—C13—C14	−0.2 (3)	C16—C11—N1—C1	−179.60 (17)
C12—C13—C14—C15	1.6 (3)	C12—C11—N1—C1	−0.5 (3)
C12—C13—C14—N14	−177.45 (17)	C11—N1—C1—O1	−2.0 (3)
C13—C14—C15—C16	−1.9 (3)	C11—N1—C1—C2	177.37 (17)
N14—C14—C15—C16	177.11 (16)	O1—C1—C2—C3	−4.6 (3)
C14—C15—C16—C11	0.9 (3)	N1—C1—C2—C3	175.98 (19)
C12—C11—C16—C15	0.4 (3)	C1—C2—C3—C4	−2.9 (4)
N1—C11—C16—C15	179.46 (15)	C2—C3—C4—O44	−175.9 (2)
C13—C14—N14—O41	−6.8 (3)	C2—C3—C4—O43	3.3 (3)
C15—C14—N14—O41	174.12 (18)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O43—H43···O1	0.82	1.72	2.537 (2)	174
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