

A hydrogen-bonded chain of rings in (*E*)-3-(4-nitrophenylaminocarbonyl)prop-2-enoic acidJames L. Wardell,^a Janet M. S. Skakle,^b John N. Low^b and Christopher Glidewell^{c*}^aInstituto de Química, Departamento de Química Inorgânica, Universidade Federal do Rio de Janeiro, 21945-970 Rio de Janeiro, RJ, Brazil, ^bDepartment of Chemistry, University of Aberdeen, Meston Walk, Old Aberdeen AB24 3UE, Scotland, and ^cSchool of Chemistry, University of St Andrews, Fife KY16 9ST, Scotland

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Key indicators

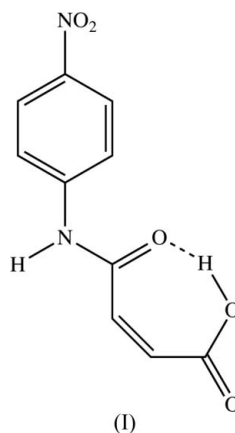
Single-crystal X-ray study
T = 291 K
Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$
R factor = 0.046
wR factor = 0.116
Data-to-parameter ratio = 17.4For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.In the title compound, $\text{C}_{10}\text{H}_8\text{N}_2\text{O}_5$, the molecules are linked into chains of rings by the concerted action of one $\text{N}-\text{H}\cdots\text{O}$ and one $\text{C}-\text{H}\cdots\text{O}$ hydrogen bond.

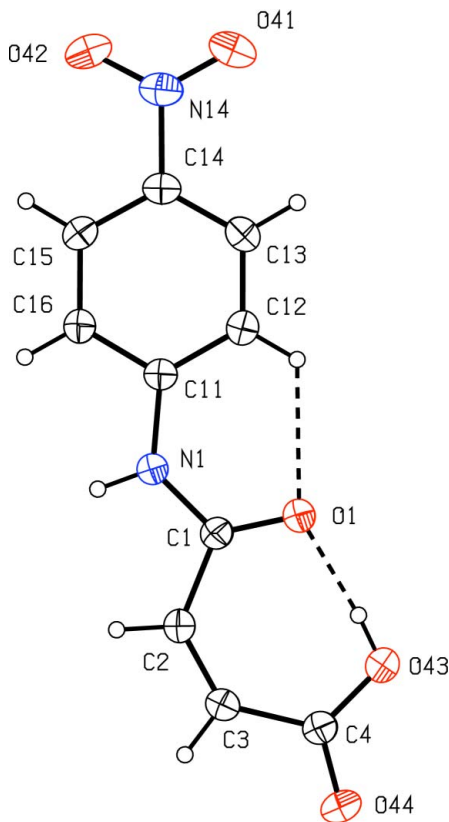
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Comment

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.There are two intramolecular hydrogen bonds in compound (I). A rather short and almost linear $\text{O}-\text{H}\cdots\text{O}$ hydrogen bond (Fig. 1) controls the conformation of the maleate fragment, forming an *S*(7) ring, while a weaker $\text{C}-\text{H}\cdots\text{O}$ 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 $\text{C}-\text{O}$ 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 $\text{N}-\text{H}\cdots\text{O}$ hydrogen bond, augmented by a rather long $\text{C}-\text{H}\cdots\text{O}$ 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.

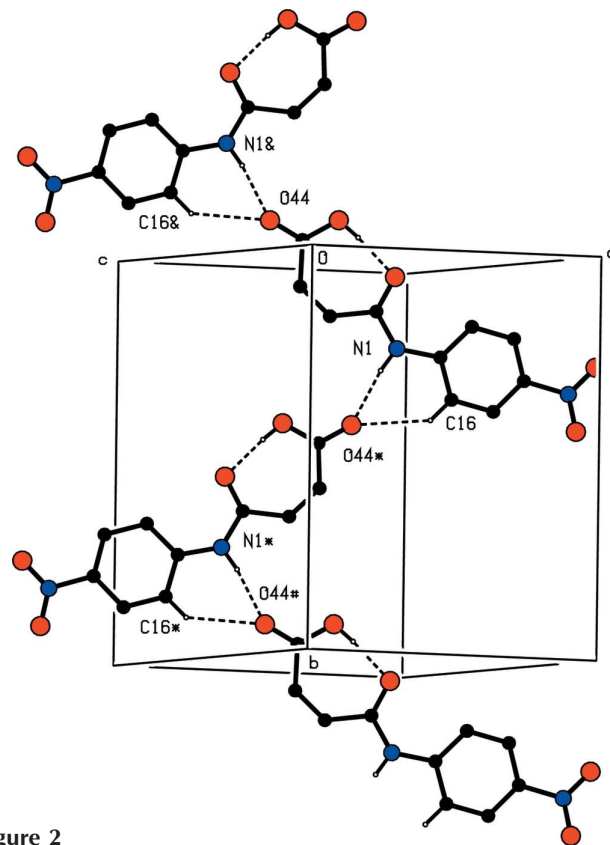
carboxyl atom O44 in the molecule at $(1 - x, \frac{1}{2} + y, \frac{3}{2} - z)$, so forming a $C(7)C(9)[R_1^2(6)]$ (Bernstein *et al.*, 1995) chain of rings running parallel to the [010] direction and generated by the 2_1 screw axis along $(\frac{1}{2}, y, \frac{3}{4})$ (Fig. 2). Two chains of this 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

| | |
|---|---|
| $\text{C}_{10}\text{H}_8\text{N}_2\text{O}_5$ | $D_x = 1.536 \text{ Mg m}^{-3}$ |
| $M_r = 236.18$ | Mo $K\alpha$ radiation |
| Monoclinic, $P2_1/c$ | Cell parameters from 2702 reflections |
| $a = 9.6052 (7) \text{ \AA}$ | $\theta = 2.8\text{--}29.0^\circ$ |
| $b = 12.8416 (10) \text{ \AA}$ | $\mu = 0.13 \text{ mm}^{-1}$ |
| $c = 9.0921 (7) \text{ \AA}$ | $T = 291 (2) \text{ K}$ |
| $\beta = 114.388 (2)^\circ$ | Block, yellow |
| $V = 1021.41 (13) \text{ \AA}^3$ | $0.27 \times 0.14 \times 0.10 \text{ mm}$ |
| $Z = 4$ | |


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

Refinement

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

Table 1

Selected geometric parameters (\AA , $^\circ$).

| | | | |
|-----------------|-------------|--------------|-------------|
| 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, °).

| <i>D</i> —H... <i>A</i> | <i>D</i> —H | H... <i>A</i> | <i>D</i> ... <i>A</i> | <i>D</i> —H... <i>A</i> |
|----------------------------|-------------|---------------|-----------------------|-------------------------|
| 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 $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C,N})$ or $1.5U_{\text{eq}}(\text{O})$.

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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