

2-Methyl-3-(4-nitrophenyl)acrylic acid

Niaz Muhammad,^a M. Nawaz Tahir,^{b*} Zia-ur-Rehman^a and Saqib Ali^a

^aDepartment of Chemistry, Quaid-i-Azam University, Islamabad 45320, Pakistan, and ^bUniversity of Sargodha, Department of Physics, Sargodha, Pakistan
Correspondence e-mail: dmntahir_uos@yahoo.com

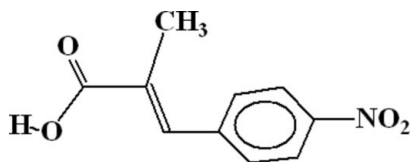
Received 10 July 2008; accepted 3 August 2008

Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.043; wR factor = 0.134; data-to-parameter ratio = 18.0.

The title compound, $\text{C}_{10}\text{H}_9\text{NO}_4$, forms $R_2^2(8)$ dimers due to intermolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonding in the crystal structure. Two dimers are further linked to each other through two intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds, forming an $R_3^3(7)$ ring motif. The nitro groups form an intramolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bond mimicking a five-membered ring. As a result of these hydrogen bonds, polymeric sheets are formed. The aromatic ring makes a dihedral angle of 42.84 (8°) with the carboxylate group and an angle of 8.01 (14°) with the nitro group. There is a π -interaction ($\text{N}-\text{O}\cdots\pi$) between the nitro group and the aromatic ring, with a distance of 3.7572 (14) Å between the N atom and the centroid of the aromatic ring.

Related literature

For related literature, see: Bernstein *et al.* (1995); Fujii *et al.* (2002); Ma & Hayes (2004); Muhammad *et al.* (2007, 2008a,b); Muhammad, Ali, Tahir & Zia-ur-Rehman (2008); Muhammad, Tahir, Ali, Zia-ur-Rehman & Kashmiri (2008); Muhammad, Tahir, Zia-ur-Rehman & Ali (2008); Muhammad, Tahir, Zia-ur-Rehman, Ali & Shaheen, (2008); Niaz *et al.* (2008).



Experimental

Crystal data

| | |
|--------------------------------------|-------------------------------------|
| $\text{C}_{10}\text{H}_9\text{NO}_4$ | $\gamma = 87.686$ (2°) |
| $M_r = 207.18$ | $V = 479.21$ (4) Å ³ |
| Triclinic, $P\bar{1}$ | $Z = 2$ |
| $a = 7.3878$ (3) Å | Mo $K\alpha$ radiation |
| $b = 8.1050$ (5) Å | $\mu = 0.11$ mm ⁻¹ |
| $c = 8.3402$ (4) Å | $T = 296$ (2) K |
| $\alpha = 75.793$ (2)° | $0.25 \times 0.20 \times 0.18$ mm |
| $\beta = 81.835$ (3)° | |

Data collection

| | |
|---|--|
| Bruker Kappa APEXII CCD diffractometer | 9039 measured reflections |
| Absorption correction: multi-scan (<i>SADABS</i> ; Bruker, 2005) | 2518 independent reflections |
| $T_{\min} = 0.970$, $T_{\max} = 0.981$ | 1926 reflections with $I > 2\sigma(I)$ |
| | $R_{\text{int}} = 0.023$ |

Refinement

| | |
|---------------------------------|--|
| $R[F^2 > 2\sigma(F^2)] = 0.042$ | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2) = 0.134$ | $\Delta\rho_{\text{max}} = 0.24$ e Å ⁻³ |
| $S = 1.02$ | $\Delta\rho_{\text{min}} = -0.21$ e Å ⁻³ |
| 2518 reflections | |
| 140 parameters | |

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|--|--------------|--------------------|-------------|----------------------|
| $\text{O1}-\text{H1}\cdots\text{O2}^i$ | 0.93 (2) | 1.71 (2) | 2.6333 (15) | 177 (2) |
| $\text{C3}-\text{H3}\cdots\text{O1}$ | 0.93 | 2.31 | 2.7080 (17) | 105 |
| $\text{C8}-\text{H8}\cdots\text{O1}^{ii}$ | 0.93 | 2.55 | 3.3471 (17) | 144 |
| $\text{C9}-\text{H9}\cdots\text{O2}^{iii}$ | 0.93 | 2.60 | 3.4912 (17) | 161 |

Symmetry codes: (i) $-x, -y, -z + 1$; (ii) $x, y + 1, z$; (iii) $-x, -y + 1, -z + 1$.

Data collection: *APEX2* (Bruker, 2007); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2007); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON*.

The authors acknowledge the Higher Education Commission, Islamabad, Pakistan, for funding the purchase of the diffractometer at GCU, Lahore and for financial support to Niaz Muhammad for PhD studies under the Indigenous Scholarship Scheme.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CS2087).

References

- Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
- Bruker (2005). *SADABS*. Bruker AXS Inc. Madison, Wisconsin, USA.
- Bruker (2007). *APEX2* and *SAINT*. Bruker AXS Inc. Madison, Wisconsin, USA.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Fujii, T., Shimaya, C., Yano, A., Terado, K., Sugino, H. & Fukuda, H. (2002). *Biotechnol. Lett.* **24**, 151–154.
- Ma, G. & Hayes, S. E. J. (2004). *Labelled Compd. Radiopharm.* **47**, 895–901.
- Muhammad, N., Ali, S., Tahir, M. N. & Zia-ur-Rehman (2008). *Acta Cryst.* **E64**, o1373.
- Muhammad, N., Tahir, M. N., Ali, S. & Zia-ur-Rehman (2008a). *Acta Cryst.* **E64**, m946–m947.
- Muhammad, N., Tahir, M. N., Ali, S. & Zia-ur-Rehman (2008b). *Acta Cryst.* **E64**, m978.
- Muhammad, N., Tahir, M. N., Ali, S., Zia-ur-Rehman & Kashmiri, M. A. (2008). *Acta Cryst.* **E64**, o1456.
- Muhammad, N., Tahir, M. N., Zia-ur-Rehman & Ali, S. (2008). *Acta Cryst.* **E64**, o1458.
- Muhammad, N., Tahir, M. N., Zia-ur-Rehman, Ali, S. & Shaheen, F. (2008). *Acta Cryst.* **E64**, o1542.

Muhammad, N., Zia-ur-Rehman, Ali, S. & Meetsma, A. (2007). *Acta Cryst.* **E63**, o2174–o2175.
Niaz, M., Tahir, M. N., Zia-ur-Rehman, Ali, S. & Khan, I. U. (2008). *Acta Cryst.* **E64**, o733.

Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

supplementary materials

Acta Cryst. (2008). E64, o1717-o1718 [doi:10.1107/S1600536808024999]

2-Methyl-3-(4-nitrophenyl)acrylic acid

N. Muhammad, M. N. Tahir, Zia-ur-Rehman and S. Ali

Comment

Cinnamic acid derivatives are widely used chemicals in a variety of fields (Ma *et al.*, 2004). They have been applied as antibacterial agents for suppression of bacterial growth (Fujii *et al.*, 2002). In wine, cinnamic acid and its derivatives join benzoic acid derivatives and flavonoids in creating pigments and tannin agents that give each vintage its characteristic bouquet and color. The title compound has been prepared in continuation of synthesizing various derivatives of cinnamic acids (Niaz *et al.*, 2008; Muhammad, Ali, Tahir & Zia-ur-Rehman, 2008; Muhammad, Tahir, Ali, Zia-ur-Rehman & Kashmiri, 2008) and their tin complexes (Muhammad *et al.*, 2008*a,b*).

The crystal structures of 3-(4-isopropylphenyl)-2-methylacrylic acid (Muhammad, Tahir, Ali, Zia-ur-Rehman & Kashmiri, 2008), of 3-(4-chlorophenyl)-2-methylacrylic acid (Muhammad, Tahir, Zia-ur-Rehman, Ali & Shaheen, 2008) and of 3-(4-bromophenyl)-2-methylacrylic acid (Muhammad *et al.*, 2007) have been reported. The title compound differs from these compounds due to the nitro group at *para* position. In the crystal structure of the title compound, the exocyclic C_{sp2}—C_{sp2} bonds are of 1.4770 (18) and 1.4880 (18) Å, the C=C is of 1.3376 (18) Å. The C—O bond length 1.2996 (16) Å is normal, much like the C=O bond length of 1.2300 (15) Å. The resonant N—O bond lengths are equal (1.2185 (16) and 1.2204 (17) Å). There is an intermolecular H-bond of C—H \cdots O type (Table 1, Fig 1). Centrosymmetric R₂²(8) dimers (Bernstein *et al.* 1995) are formed due to the intermolecular O1—H1 \cdots O2ⁱ [symmetry code: *i* = -*x*, -*y*, -*z* + 1] hydrogen bonding. Two adjacent dimers are linked to each other through two intermolecular H-bonds of C—H \cdots O type forming an R₃³(7) motif (Bernstein *et al.* 1995). The group of two dimers are linked to each other by intermolecular H-bonding (Table 1, Fig 2). There exist an N1—O4 \cdots Cgⁱⁱ [symmetry code: *ii* = -*x* + 1, -*y* + 2, -*z*] interaction with a distance of 3.7572 (14) Å between the N-atom and the centroid of the (C4—C9) aromatic ring. The aromatic ring makes a dihedral angle of 42.84 (8)° with the carboxylate (O1/C1/O2) moiety and 8.01 (14)° with the (N1/O3/O4) nitro group. Due to the intermolecular H-bonding polymeric sheets are formed.

Experimental

The title compound was prepared according to a reported procedure (Muhammad *et al.*, 2007). A mixture of 4-nitrobenzaldehyde (1.51 g, 10 mmol), methylmalonic acid (2.36 g, 20 mmol) and piperidine (1.98 ml, 20 mmol) in a pyridine (12.5 ml) solution was heated on a steam-bath for 24 h. The reaction mixture was cooled and added to a mixture of 25 ml of concentrated HCl and 50 g of ice. The precipitate formed in the acidified mixture was filtered off and washed with ice-cold water. The product was recrystallized from ethanol. The yield was 79%.

Refinement

The coordinates of H-atom attached with O1 were refined. The H-atoms attached with C-atoms were positioned geometrically, C—H = 0.93, and 0.96 Å for aromatic and methyl H, and constrained to ride on their parent atoms. The H-atoms were treated as isotropic with $U_{\text{iso}}(\text{H}) = xU_{\text{eq}}(\text{C}, \text{O})$, where $x = 1.5$ for methyl H, and $x = 1.2$ for all other H atoms.

Figures

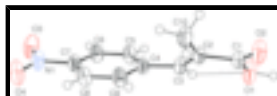


Fig. 1. ORTEP drawing of the title compound, $\text{C}_{11}\text{H}_{12}\text{O}_2$ with the atom numbering scheme. The thermal ellipsoids are drawn at the 50% probability level. H-atoms are shown by small circles of arbitrary radii. The intramolecular H-bonds are shown by dotted lines.

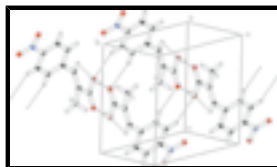


Fig. 2. The packing figure (PLATON: Spek, 2003) which shows the dimeric nature of the compound and the interlinkages of the dimers.

2-Methyl-3-(4-nitrophenyl)acrylic acid

Crystal data

| | |
|--------------------------------------|---|
| $\text{C}_{10}\text{H}_9\text{NO}_4$ | $Z = 2$ |
| $M_r = 207.18$ | $F_{000} = 216$ |
| Triclinic, $P\bar{1}$ | $D_x = 1.436 \text{ Mg m}^{-3}$ |
| Hall symbol: -P 1 | Mo $K\alpha$ radiation |
| $a = 7.3878 (3) \text{ \AA}$ | $\lambda = 0.71073 \text{ \AA}$ |
| $b = 8.1050 (5) \text{ \AA}$ | Cell parameters from 2518 reflections |
| $c = 8.3402 (4) \text{ \AA}$ | $\theta = 2.5\text{--}29.1^\circ$ |
| $\alpha = 75.793 (2)^\circ$ | $\mu = 0.11 \text{ mm}^{-1}$ |
| $\beta = 81.835 (3)^\circ$ | $T = 296 (2) \text{ K}$ |
| $\gamma = 87.686 (2)^\circ$ | Prismatic, colourless |
| $V = 479.21 (4) \text{ \AA}^3$ | $0.25 \times 0.20 \times 0.18 \text{ mm}$ |

Data collection

| | |
|--|--|
| Bruker Kappa APEXII CCD diffractometer | 2518 independent reflections |
| Radiation source: fine-focus sealed tube | 1926 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{\text{int}} = 0.023$ |
| Detector resolution: 7.4 pixels mm^{-1} | $\theta_{\text{max}} = 29.1^\circ$ |
| $T = 296(2) \text{ K}$ | $\theta_{\text{min}} = 2.5^\circ$ |
| ω scans | $h = -10 \rightarrow 9$ |
| Absorption correction: multi-scan (SADABS; Bruker, 2005) | $k = -11 \rightarrow 9$ |
| $T_{\text{min}} = 0.970$, $T_{\text{max}} = 0.981$ | $l = -11 \rightarrow 11$ |

9039 measured reflections

Refinement

| | |
|--|--|
| Refinement on F^2 | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.042$ | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2) = 0.134$ | $w = 1/[\sigma^2(F_o^2) + (0.0713P)^2 + 0.0915P]$ |
| $S = 1.02$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| 2518 reflections | $(\Delta/\sigma)_{\max} < 0.001$ |
| 140 parameters | $\Delta\rho_{\max} = 0.24 \text{ e } \text{\AA}^{-3}$ |
| Primary atom site location: structure-invariant direct methods | $\Delta\rho_{\min} = -0.21 \text{ e } \text{\AA}^{-3}$ |
| | Extinction coefficient: ? |

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

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|--------------|--------------|---------------|----------------------------------|
| O1 | 0.04661 (16) | 0.13519 (13) | 0.29456 (12) | 0.0547 (4) |
| O2 | 0.09796 (15) | 0.16087 (12) | 0.54300 (12) | 0.0526 (3) |
| O3 | 0.52309 (18) | 1.11600 (15) | -0.36244 (14) | 0.0661 (4) |
| O4 | 0.35761 (19) | 1.23296 (13) | -0.18699 (15) | 0.0648 (4) |
| N1 | 0.41247 (18) | 1.10856 (14) | -0.23745 (14) | 0.0466 (4) |
| C1 | 0.10453 (16) | 0.21896 (15) | 0.39150 (15) | 0.0357 (3) |
| C2 | 0.17797 (17) | 0.39231 (15) | 0.31101 (16) | 0.0362 (3) |
| C3 | 0.17195 (18) | 0.45348 (15) | 0.14759 (16) | 0.0378 (3) |
| C4 | 0.22936 (17) | 0.62471 (15) | 0.04743 (15) | 0.0361 (3) |
| C5 | 0.3308 (2) | 0.64219 (16) | -0.11015 (16) | 0.0424 (4) |
| C6 | 0.39099 (19) | 0.80039 (17) | -0.20464 (16) | 0.0425 (4) |
| C7 | 0.34487 (18) | 0.94026 (15) | -0.14089 (15) | 0.0375 (4) |
| C8 | 0.2375 (2) | 0.92912 (16) | 0.01030 (17) | 0.0432 (4) |
| C9 | 0.1804 (2) | 0.76957 (16) | 0.10462 (16) | 0.0430 (4) |
| C10 | 0.2580 (2) | 0.47901 (17) | 0.42356 (17) | 0.0480 (4) |
| H1 | -0.005 (3) | 0.032 (3) | 0.355 (2) | 0.0656* |
| H3 | 0.12727 | 0.38114 | 0.09157 | 0.0454* |
| H5 | 0.35821 | 0.54652 | -0.15208 | 0.0508* |
| H6 | 0.46089 | 0.81202 | -0.30868 | 0.0510* |
| H8 | 0.20402 | 1.02624 | 0.04821 | 0.0518* |
| H9 | 0.10844 | 0.75935 | 0.20750 | 0.0516* |
| H10A | 0.33667 | 0.40104 | 0.48836 | 0.0719* |
| H10B | 0.16126 | 0.51574 | 0.49695 | 0.0719* |
| H10C | 0.32723 | 0.57588 | 0.35757 | 0.0719* |

Atomic displacement parameters (\AA^2)

| | | | | | |
|----------|----------|----------|----------|----------|----------|
| U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|----------|----------|----------|----------|----------|----------|

supplementary materials

| | | | | | | |
|-----|------------|------------|------------|-------------|-------------|-------------|
| O1 | 0.0855 (8) | 0.0371 (5) | 0.0397 (5) | -0.0259 (5) | -0.0071 (5) | -0.0026 (4) |
| O2 | 0.0801 (7) | 0.0381 (5) | 0.0357 (5) | -0.0229 (5) | -0.0058 (5) | 0.0008 (4) |
| O3 | 0.0862 (8) | 0.0524 (7) | 0.0487 (6) | -0.0249 (6) | 0.0068 (6) | 0.0033 (5) |
| O4 | 0.1010 (9) | 0.0290 (5) | 0.0614 (7) | -0.0085 (5) | -0.0120 (6) | -0.0036 (5) |
| N1 | 0.0638 (7) | 0.0340 (6) | 0.0385 (6) | -0.0119 (5) | -0.0133 (5) | 0.0030 (5) |
| C1 | 0.0399 (6) | 0.0288 (6) | 0.0353 (6) | -0.0057 (5) | -0.0021 (5) | -0.0028 (4) |
| C2 | 0.0379 (6) | 0.0278 (5) | 0.0392 (6) | -0.0052 (5) | -0.0024 (5) | -0.0019 (5) |
| C3 | 0.0443 (6) | 0.0284 (6) | 0.0380 (6) | -0.0068 (5) | -0.0037 (5) | -0.0029 (5) |
| C4 | 0.0416 (6) | 0.0294 (6) | 0.0342 (6) | -0.0041 (5) | -0.0064 (5) | -0.0004 (4) |
| C5 | 0.0569 (8) | 0.0300 (6) | 0.0375 (6) | -0.0005 (5) | -0.0009 (5) | -0.0062 (5) |
| C6 | 0.0521 (7) | 0.0360 (6) | 0.0338 (6) | -0.0020 (5) | 0.0023 (5) | -0.0023 (5) |
| C7 | 0.0468 (7) | 0.0281 (6) | 0.0343 (6) | -0.0062 (5) | -0.0093 (5) | 0.0017 (5) |
| C8 | 0.0607 (8) | 0.0292 (6) | 0.0380 (6) | 0.0016 (5) | -0.0052 (6) | -0.0062 (5) |
| C9 | 0.0554 (8) | 0.0348 (6) | 0.0334 (6) | -0.0009 (5) | 0.0030 (5) | -0.0029 (5) |
| C10 | 0.0626 (8) | 0.0353 (7) | 0.0437 (7) | -0.0160 (6) | -0.0124 (6) | 0.0001 (5) |

Geometric parameters (Å, °)

| | | | |
|-------------------------|-------------|-------------------------|-------------|
| O1—C1 | 1.2996 (16) | C5—C6 | 1.3832 (19) |
| O2—C1 | 1.2300 (15) | C6—C7 | 1.3774 (19) |
| O3—N1 | 1.2204 (17) | C7—C8 | 1.3759 (19) |
| O4—N1 | 1.2185 (16) | C8—C9 | 1.3855 (19) |
| O1—H1 | 0.93 (2) | C3—H3 | 0.9300 |
| N1—C7 | 1.4698 (17) | C5—H5 | 0.9300 |
| C1—C2 | 1.4880 (18) | C6—H6 | 0.9300 |
| C2—C3 | 1.3376 (18) | C8—H8 | 0.9300 |
| C2—C10 | 1.4965 (19) | C9—H9 | 0.9300 |
| C3—C4 | 1.4770 (18) | C10—H10A | 0.9600 |
| C4—C9 | 1.3887 (18) | C10—H10B | 0.9600 |
| C4—C5 | 1.3951 (18) | C10—H10C | 0.9600 |
| O1...C8 ⁱ | 3.3471 (17) | C4...H10C | 2.7200 |
| O1...C6 ⁱⁱ | 3.4128 (19) | C4...H3 ⁱⁱ | 3.0300 |
| O1...O2 ⁱⁱⁱ | 2.6333 (15) | C9...H10C | 2.6400 |
| O2...O1 ⁱⁱⁱ | 2.6333 (15) | C10...H9 | 2.8300 |
| O2...C1 ⁱⁱⁱ | 3.3657 (16) | C10...H10B ^v | 3.0700 |
| O2...N1 ^{iv} | 3.1112 (17) | H1...O1 ⁱⁱⁱ | 2.882 (17) |
| O1...H1 ⁱⁱⁱ | 2.882 (17) | H1...O2 ⁱⁱⁱ | 1.71 (2) |
| O1...H3 | 2.3100 | H1...C1 ⁱⁱⁱ | 2.59 (2) |
| O1...H8 ⁱ | 2.5500 | H1...H1 ⁱⁱⁱ | 2.36 (2) |
| O2...H10B | 2.8600 | H3...O1 | 2.3100 |
| O2...H10A | 2.5900 | H3...H5 | 2.5900 |
| O2...H1 ⁱⁱⁱ | 1.71 (2) | H3...C4 ⁱⁱ | 3.0300 |
| O2...H9 ^v | 2.6000 | H5...O4 ⁱ | 2.6300 |
| O3...H6 | 2.4400 | H5...H3 | 2.5900 |
| O3...H10A ^{vi} | 2.7600 | H6...O3 | 2.4400 |
| O3...H6 ^{vii} | 2.6500 | H6...O3 ^{vii} | 2.6500 |

| | | | |
|---------------------------|--------------|---------------------------|--------------|
| O3...H10C ^{viii} | 2.7800 | H6...C2 ^x | 3.0800 |
| O4...H5 ^{ix} | 2.6300 | H8...O1 ^{ix} | 2.5500 |
| O4...H8 | 2.4200 | H8...O4 | 2.4200 |
| O4...H10A ^{vi} | 2.7400 | H9...C2 | 2.9300 |
| O4...H10C ^{viii} | 2.8500 | H9...C10 | 2.8300 |
| N1...O2 ^{vi} | 3.1112 (17) | H9...H10C | 2.4200 |
| N1...C8 ^{viii} | 3.378 (2) | H9...O2 ^v | 2.6000 |
| C1...O2 ⁱⁱⁱ | 3.3657 (16) | H10A...O2 | 2.5900 |
| C2...C6 ^x | 3.5837 (19) | H10A...O3 ^{iv} | 2.7600 |
| C6...O1 ⁱⁱ | 3.4128 (19) | H10A...O4 ^{iv} | 2.7400 |
| C6...C2 ^x | 3.5837 (19) | H10B...O2 | 2.8600 |
| C8...N1 ^{viii} | 3.378 (2) | H10B...C1 ^v | 3.0800 |
| C8...O1 ^{ix} | 3.3471 (17) | H10B...C2 ^v | 2.9500 |
| C9...C10 | 3.1937 (19) | H10B...C10 ^v | 3.0700 |
| C10...C9 | 3.1937 (19) | H10B...H10B ^v | 2.4000 |
| C1...H10B ^v | 3.0800 | H10C...C4 | 2.7200 |
| C1...H1 ⁱⁱⁱ | 2.59 (2) | H10C...C9 | 2.6400 |
| C2...H9 | 2.9300 | H10C...H9 | 2.4200 |
| C2...H10B ^v | 2.9500 | H10C...O3 ^{viii} | 2.7800 |
| C2...H6 ^x | 3.0800 | H10C...O4 ^{viii} | 2.8500 |
| C1—O1—H1 | 111.6 (12) | C7—C8—C9 | 118.33 (12) |
| O3—N1—O4 | 123.45 (13) | C4—C9—C8 | 120.81 (12) |
| O3—N1—C7 | 118.21 (12) | C2—C3—H3 | 117.00 |
| O4—N1—C7 | 118.33 (12) | C4—C3—H3 | 117.00 |
| O1—C1—O2 | 122.55 (12) | C4—C5—H5 | 120.00 |
| O1—C1—C2 | 116.77 (11) | C6—C5—H5 | 120.00 |
| O2—C1—C2 | 120.68 (11) | C5—C6—H6 | 121.00 |
| C1—C2—C10 | 115.28 (11) | C7—C6—H6 | 121.00 |
| C3—C2—C10 | 126.40 (12) | C7—C8—H8 | 121.00 |
| C1—C2—C3 | 118.29 (11) | C9—C8—H8 | 121.00 |
| C2—C3—C4 | 126.35 (12) | C4—C9—H9 | 120.00 |
| C3—C4—C9 | 121.47 (11) | C8—C9—H9 | 120.00 |
| C5—C4—C9 | 119.09 (12) | C2—C10—H10A | 109.00 |
| C3—C4—C5 | 119.41 (11) | C2—C10—H10B | 109.00 |
| C4—C5—C6 | 120.66 (12) | C2—C10—H10C | 109.00 |
| C5—C6—C7 | 118.38 (12) | H10A—C10—H10B | 109.00 |
| N1—C7—C6 | 119.04 (11) | H10A—C10—H10C | 110.00 |
| N1—C7—C8 | 118.35 (11) | H10B—C10—H10C | 109.00 |
| C6—C7—C8 | 122.61 (12) | | |
| O3—N1—C7—C6 | -7.7 (2) | C2—C3—C4—C9 | -44.9 (2) |
| O3—N1—C7—C8 | 172.34 (13) | C3—C4—C5—C6 | -178.09 (13) |
| O4—N1—C7—C6 | 173.58 (14) | C9—C4—C5—C6 | 3.7 (2) |
| O4—N1—C7—C8 | -6.4 (2) | C3—C4—C9—C8 | 179.06 (13) |
| O1—C1—C2—C3 | 3.07 (18) | C5—C4—C9—C8 | -2.7 (2) |
| O1—C1—C2—C10 | -174.99 (12) | C4—C5—C6—C7 | -1.4 (2) |

supplementary materials

| | | | |
|--------------|--------------|-------------|--------------|
| O2—C1—C2—C3 | -176.04 (13) | C5—C6—C7—N1 | 178.15 (13) |
| O2—C1—C2—C10 | 5.90 (18) | C5—C6—C7—C8 | -1.9 (2) |
| C1—C2—C3—C4 | 176.84 (12) | N1—C7—C8—C9 | -177.24 (13) |
| C10—C2—C3—C4 | -5.3 (2) | C6—C7—C8—C9 | 2.8 (2) |
| C2—C3—C4—C5 | 136.88 (15) | C7—C8—C9—C4 | -0.4 (2) |

Symmetry codes: (i) $x, y-1, z$; (ii) $-x, -y+1, -z$; (iii) $-x, -y, -z+1$; (iv) $x, y-1, z+1$; (v) $-x, -y+1, -z+1$; (vi) $x, y+1, z-1$; (vii) $-x+1, -y+2, -z-1$; (viii) $-x+1, -y+2, -z$; (ix) $x, y+1, z$; (x) $-x+1, -y+1, -z$.

Hydrogen-bond geometry (\AA , $^\circ$)

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|----------------------------------|----------|-------------|-------------|---------------|
| O1—H1 \cdots O2 ⁱⁱⁱ | 0.93 (2) | 1.71 (2) | 2.6333 (15) | 177 (2) |
| C3—H3 \cdots O1 | 0.93 | 2.31 | 2.7080 (17) | 105 |
| C8—H8 \cdots O1 ^{ix} | 0.93 | 2.55 | 3.3471 (17) | 144 |
| C9—H9 \cdots O2 ^v | 0.93 | 2.60 | 3.4912 (17) | 161 |

Symmetry codes: (iii) $-x, -y, -z+1$; (ix) $x, y+1, z$; (v) $-x, -y+1, -z+1$.

Fig. 1

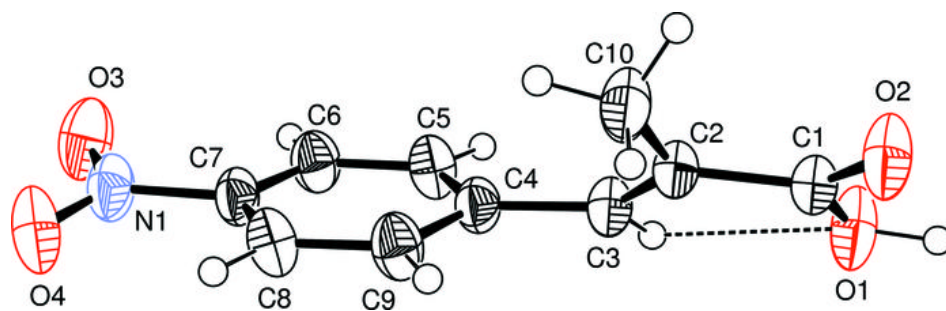


Fig. 2

