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2-Methyl-2-(4-nitrophenoxy)propanoic acid

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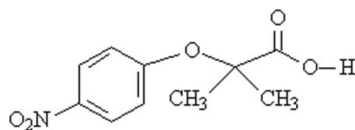
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Key indicators: single-crystal X-ray study; $T = 294$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.065; wR factor = 0.163; data-to-parameter ratio = 12.6.

The title compound, $\text{C}_{10}\text{H}_{11}\text{NO}_5$, is of interest with respect to its antidyslipidemic activity. It was prepared by reaction of 4-nitrophenol with ethyl 2-bromo-2-methylpropionate followed by ethyl ester hydrolysis. In the crystal, molecules are linked into centrosymmetric dimers by intermolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds and the dimers are connected into chains by weak $\text{C}-\text{H}\cdots\text{O}$ interactions. The packing is further stabilized by offset $\pi-\pi$ interactions between adjacent benzene rings with a centroid-centroid distance of 3.8643 (17) Å.

Related literature

For related literature on fibrate structures and hypolipidemic activity, see: Navarrete-Vázquez *et al.* (2008); Henry *et al.* (2003); Rath *et al.* (2005); Djinoić *et al.* (1989); Thorp (1962); Thorp & Waring (1962); Miller & Spence (1998); Forcheron *et al.* (2002). For details of the graph-set analysis of hydrogen-bonding patterns, see: Bernstein *et al.* (1995)



Experimental

Crystal data

| | |
|---|---------------------------------|
| $\text{C}_{10}\text{H}_{11}\text{NO}_5$ | $c = 14.518$ (2) Å |
| $M_r = 225.20$ | $\beta = 93.794$ (2)° |
| Monoclinic, $C2/c$ | $V = 2170.2$ (5) Å ³ |
| $a = 21.296$ (3) Å | $Z = 8$ |
| $b = 7.0348$ (9) Å | Mo $K\alpha$ radiation |

$\mu = 0.11$ mm⁻¹
 $T = 294$ (2) K

0.32 × 0.25 × 0.20 mm

Data collection

| | |
|---|--|
| Bruker SMART APEX CCD area-detector diffractometer | 10072 measured reflections |
| Absorption correction: multi-scan (SADABS; Sheldrick, 1996) | 1920 independent reflections |
| $T_{\min} = 0.763$, $T_{\max} = 0.978$ | 1745 reflections with $I > 2\sigma(I)$ |
| | $R_{\text{int}} = 0.035$ |

Refinement

| | |
|---------------------------------|--|
| $R[F^2 > 2\sigma(F^2)] = 0.065$ | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2) = 0.163$ | $\Delta\rho_{\text{max}} = 0.23$ e Å ⁻³ |
| $S = 1.12$ | $\Delta\rho_{\text{min}} = -0.22$ e Å ⁻³ |
| 1920 reflections | |
| 152 parameters | |

Table 1
Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|---|--------------|--------------------|-------------|----------------------|
| $\text{O5}-\text{H5A}\cdots\text{O4}^i$ | 0.92 (4) | 1.75 (4) | 2.659 (3) | 173 (3) |
| $\text{C6}-\text{H6}\cdots\text{O1}^{ii}$ | 0.93 | 2.34 | 3.165 (4) | 147 |

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

Data collection: SMART (Bruker, 2000); cell refinement: SAINT-Plus-NT (Bruker, 2001); data reduction: SAINT-Plus-NT; program(s) used to solve structure: SHELXTL-NT (Sheldrick, 2008); program(s) used to refine structure: SHELXTL-NT; molecular graphics: SHELXTL-NT (Sheldrick, 2008); software used to prepare material for publication: PLATON (Spek, 2003) and publCIF (Westrip, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2550).

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supporting information

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2-Methyl-2-(4-nitrophenoxy)propanoic acid

Gabriel Navarrete-Vázquez, Hector Torres-Gómez, Sergio Hidalgo-Figueroa and Hugo Tlahuext

S1. Comment

The fibrates belong to a class of lipid-modifying agents that decrease plasma triglycerides (Thorp, 1962; Miller & Spence, 1998; Forcheron *et al.*, 2002). These compounds are used as therapeutic agents in the treatment of dyslipidemia, heart disease and diabetic complications in humans. The fibric acid pharmacophore has been of interest to medicinal chemists ever since the initial discovery that ethyl chlorophenoxyisobutyrate possessed hypolipidemic properties (Thorp & Waring, 1962).

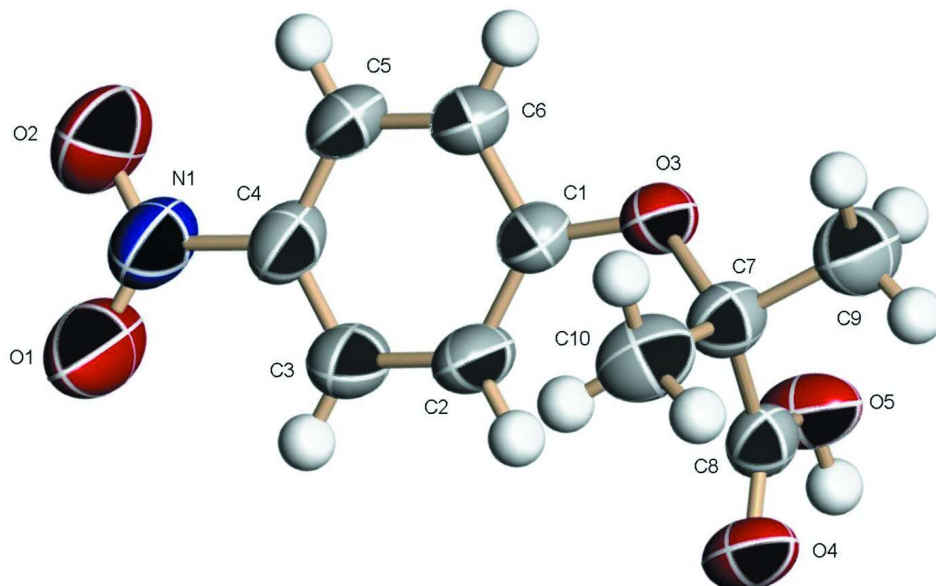
In order to assist our knowledge about the electronic and steric requirements to shown antihyperlipidemic activity, we have determined the crystal structure of the title compound, (I), Fig 1, which is a bioisoster of clofibrac acid, with a nitro group instead of chlorine atom. The crystal structure is stabilized by strong O5—H5A···O4 hydrogen-bonding interactions, forming $R_2^2(8)$ motifs (Bernstein *et al.*, 1995) (Fig. 1; Table 2). In the crystal packing there are also weak C6—H6···O1 hydrogen bonding interactions that interconnect molecules into chains running along the *b* axis. The crystal structure is also stabilized by offset π – π interactions between two adjacent molecules, with a distance between centroids of the C1—C6 benzene rings [Cg1, Cg1" (Symmetry code: $-x, y, -z + 1/2$)] of 3.8643 (17) Å. (Fig. 2; Table 1).

S2. Experimental

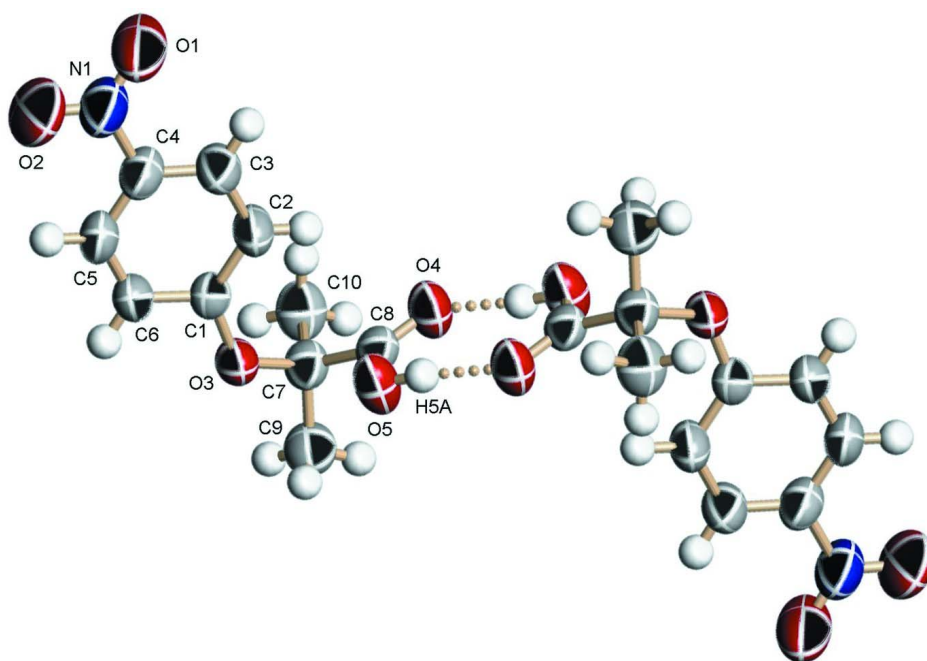
A mixture of 4-nitrophenol (1.0 g, 4.44 mmol), potassium carbonate (1.22 g, 8.88 mmol) in acetonitrile, was added dropwise to 1.04 ml of ethyl 2-bromo-2-methylpropionate (1.29 g, 6.66 mmol). The mixture was stirred and heated under reflux for 8 h then poured onto cold water. The resulting oil was treated with a mixture of THF/MeOH/H₂O (3:2:1), and LiOH (5 equiv) was added. The mixture stirred at room temperature for 3 h, 10% HCl solution added, and most of the organic solvents removed *in vacuo*. The partly solid residue was extracted with CH₂Cl₂ (3 x 10 ml), dried with Na₂SO₄, filtered, and concentrated in *vacuo* to give a yellow solid (m.p. 396 K). Single crystals of (I) were obtained from chloroform.

S3. Refinement

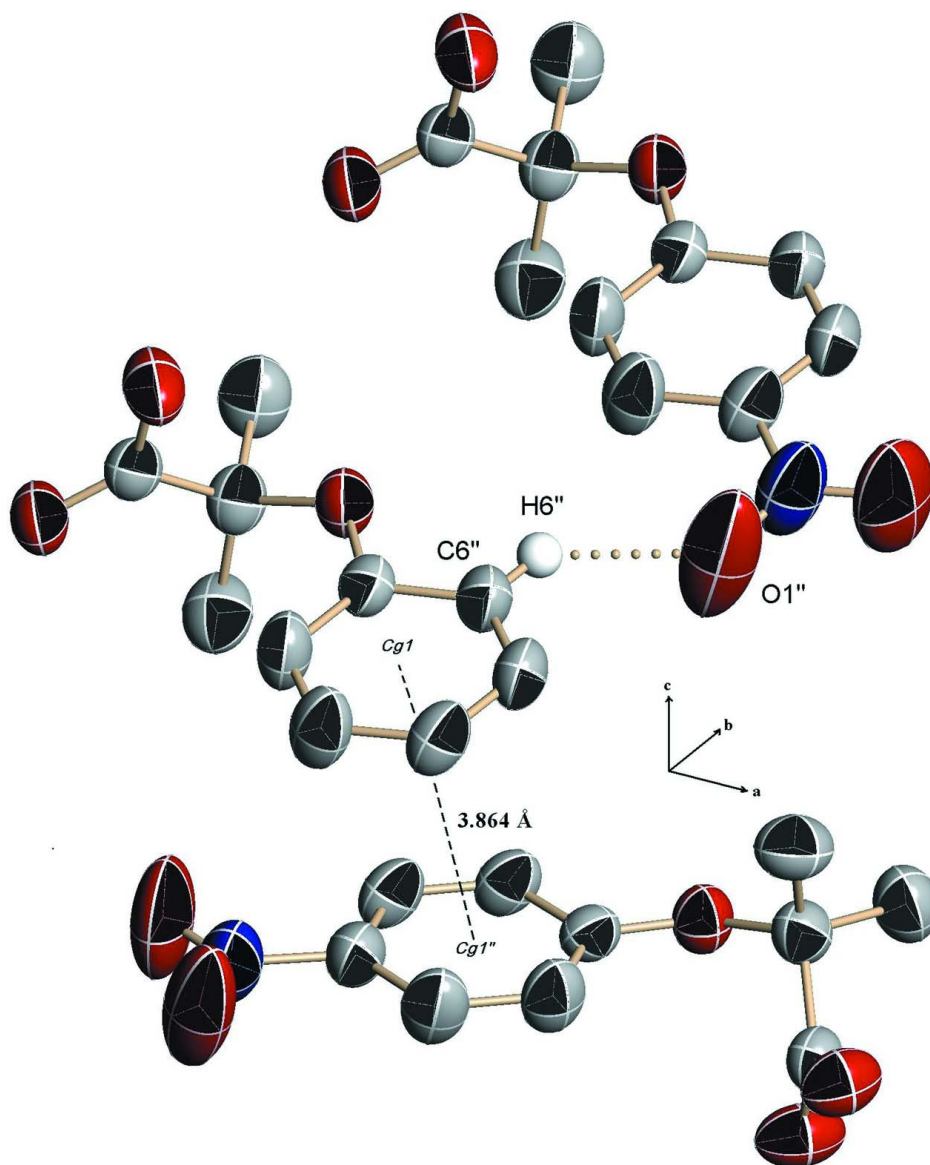
Aromatic and methyl H atoms were positioned geometrically, were constrained to the riding-model approximation [$C-H_{\text{aryl}} = 0.93$ Å, $U_{\text{iso}}(H_{\text{aryl}}) = 1.2 U_{\text{eq}}(C_{\text{aryl}})$; $C-H_{\text{methyl}} = 0.96$ Å, $U_{\text{iso}}(H_{\text{methyl}}) = 1.5 U_{\text{eq}}(C_{\text{methyl}})$]. Atom H5A, which is involved in hydrogen-bonding interaction, was located in a difference Fourier map and refined freely with isotropic displacement parameters.

**Figure 1**

The molecular structure of I showing 50% probability displacement ellipsoids and the atomic numbering. H atoms are shown as small spheres of arbitrary radius.

**Figure 2**

Centrosymmetric dimers generated by O4—H4...O3 intermolecular hydrogen bonds (dotted lines) forming an $R_2^2(8)$ motif.

**Figure 3**

View of the offset π - π interaction and C—H \cdots O hydrogen bonds between adjacent molecules. The dashed line indicates the vector between the centroids (Cg1, Cg1''). The hydrogen bond is represented by a dotted line and H atoms not involved in hydrogen bonding have been omitted for clarity.

2-Methyl-2-(4-nitrophenoxy)propanoic acid

Crystal data

$C_{10}H_{11}NO_5$

$M_r = 225.20$

Monoclinic, $C2/c$

Hall symbol: $-C 2yc$

$a = 21.296 (3) \text{ \AA}$

$b = 7.0348 (9) \text{ \AA}$

$c = 14.518 (2) \text{ \AA}$

$\beta = 93.794 (2)^\circ$

$V = 2170.2 (5) \text{ \AA}^3$

$Z = 8$

$F(000) = 944$

$D_x = 1.378 \text{ Mg m}^{-3}$

Melting point: 396 K

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 5496 reflections

$\theta = 2.5\text{--}2.9^\circ$

$\mu = 0.11 \text{ mm}^{-1}$
 $T = 294 \text{ K}$

Prism, colourless
 $0.32 \times 0.25 \times 0.20 \text{ mm}$

Data collection

Bruker SMART APEX CCD area-detector
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 φ and ω scans
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.763$, $T_{\max} = 0.978$

10072 measured reflections
 1920 independent reflections
 1745 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.035$
 $\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 1.9^\circ$
 $h = -25 \rightarrow 25$
 $k = -8 \rightarrow 8$
 $l = -17 \rightarrow 17$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.065$
 $wR(F^2) = 0.163$
 $S = 1.12$
 1920 reflections
 152 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 atoms treated by a mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0689P)^2 + 2.0562P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.23 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.22 \text{ e } \text{\AA}^{-3}$
 Extinction correction: SHELXTL-NT (Sheldrick,
 2008), $F_c^* = kFc[1 + 0.001x \text{Fc}^2\lambda^3/\sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.020 (2)

Special details

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|----|--------------|------------|--------------|----------------------------------|
| C1 | 0.43849 (11) | 0.9257 (3) | 0.38604 (15) | 0.0513 (6) |
| C2 | 0.42396 (12) | 0.7351 (4) | 0.3768 (2) | 0.0696 (8) |
| H2 | 0.3822 | 0.6954 | 0.3747 | 0.084* |
| C3 | 0.47162 (13) | 0.6040 (4) | 0.3706 (2) | 0.0783 (9) |
| H3 | 0.4623 | 0.4754 | 0.3641 | 0.094* |
| C4 | 0.53299 (12) | 0.6654 (4) | 0.3741 (2) | 0.0667 (7) |
| C5 | 0.54791 (12) | 0.8533 (4) | 0.38383 (19) | 0.0661 (7) |
| H5 | 0.5897 | 0.8926 | 0.3866 | 0.079* |
| C6 | 0.50077 (11) | 0.9819 (4) | 0.38940 (18) | 0.0613 (7) |
| H6 | 0.5106 | 1.1103 | 0.3956 | 0.074* |
| C7 | 0.33120 (11) | 1.0562 (4) | 0.36820 (17) | 0.0584 (7) |
| C8 | 0.29840 (10) | 0.9120 (3) | 0.42697 (16) | 0.0532 (6) |

| | | | | |
|------|--------------|------------|--------------|-------------|
| C9 | 0.30573 (14) | 1.2523 (4) | 0.3905 (2) | 0.0801 (9) |
| H9A | 0.3183 | 1.2844 | 0.4533 | 0.120* |
| H9B | 0.2606 | 1.2512 | 0.3823 | 0.120* |
| H9C | 0.3222 | 1.3448 | 0.3499 | 0.120* |
| C10 | 0.32173 (14) | 1.0132 (5) | 0.26611 (19) | 0.0772 (8) |
| H10A | 0.3449 | 1.1033 | 0.2320 | 0.116* |
| H10B | 0.2778 | 1.0221 | 0.2471 | 0.116* |
| H10C | 0.3365 | 0.8871 | 0.2544 | 0.116* |
| H5A | 0.2918 (16) | 0.838 (5) | 0.547 (3) | 0.101 (11)* |
| N1 | 0.58387 (13) | 0.5287 (4) | 0.3648 (2) | 0.0966 (9) |
| O1 | 0.57049 (14) | 0.3662 (4) | 0.3469 (3) | 0.1713 (19) |
| O2 | 0.63689 (12) | 0.5842 (4) | 0.3687 (3) | 0.1449 (13) |
| O3 | 0.39722 (7) | 1.0706 (2) | 0.39675 (12) | 0.0594 (5) |
| O4 | 0.25911 (8) | 0.8033 (3) | 0.39279 (12) | 0.0711 (6) |
| O5 | 0.31446 (10) | 0.9212 (3) | 0.51375 (13) | 0.0770 (7) |

Atomic displacement parameters (Å²)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C1 | 0.0472 (12) | 0.0552 (14) | 0.0519 (13) | -0.0041 (10) | 0.0076 (9) | 0.0040 (10) |
| C2 | 0.0477 (13) | 0.0595 (16) | 0.103 (2) | -0.0087 (12) | 0.0140 (13) | 0.0028 (14) |
| C3 | 0.0658 (17) | 0.0506 (15) | 0.121 (2) | -0.0042 (13) | 0.0250 (16) | 0.0062 (15) |
| C4 | 0.0515 (14) | 0.0686 (17) | 0.0816 (18) | 0.0059 (13) | 0.0163 (12) | 0.0123 (14) |
| C5 | 0.0453 (13) | 0.0758 (18) | 0.0784 (17) | -0.0102 (13) | 0.0142 (12) | -0.0012 (14) |
| C6 | 0.0536 (14) | 0.0583 (14) | 0.0736 (16) | -0.0112 (12) | 0.0151 (11) | -0.0034 (12) |
| C7 | 0.0481 (13) | 0.0609 (15) | 0.0659 (15) | 0.0001 (11) | 0.0021 (10) | 0.0060 (12) |
| C8 | 0.0418 (11) | 0.0595 (14) | 0.0583 (14) | 0.0012 (10) | 0.0025 (10) | -0.0016 (11) |
| C9 | 0.0691 (17) | 0.0653 (17) | 0.106 (2) | 0.0078 (14) | 0.0085 (15) | 0.0093 (16) |
| C10 | 0.0730 (18) | 0.092 (2) | 0.0657 (17) | -0.0098 (16) | 0.0012 (13) | 0.0144 (15) |
| N1 | 0.0659 (17) | 0.0787 (19) | 0.148 (3) | 0.0083 (14) | 0.0311 (16) | 0.0179 (18) |
| O1 | 0.105 (2) | 0.0664 (17) | 0.353 (6) | 0.0126 (15) | 0.091 (3) | 0.017 (2) |
| O2 | 0.0574 (15) | 0.127 (2) | 0.252 (4) | 0.0171 (15) | 0.0225 (18) | -0.013 (2) |
| O3 | 0.0483 (9) | 0.0532 (10) | 0.0770 (12) | -0.0036 (7) | 0.0049 (8) | -0.0015 (8) |
| O4 | 0.0619 (11) | 0.0864 (14) | 0.0648 (11) | -0.0253 (10) | 0.0017 (8) | -0.0029 (9) |
| O5 | 0.0797 (13) | 0.0928 (15) | 0.0581 (11) | -0.0337 (11) | 0.0008 (9) | 0.0035 (10) |

Geometric parameters (Å, °)

| | | | |
|-------|-----------|----------|-----------|
| C1—O3 | 1.361 (3) | C7—C8 | 1.524 (3) |
| C1—C2 | 1.380 (4) | C7—C9 | 1.525 (4) |
| C1—C6 | 1.382 (3) | C8—O4 | 1.215 (3) |
| C2—C3 | 1.379 (4) | C8—O5 | 1.285 (3) |
| C2—H2 | 0.9300 | C9—H9A | 0.9600 |
| C3—C4 | 1.374 (4) | C9—H9B | 0.9600 |
| C3—H3 | 0.9300 | C9—H9C | 0.9600 |
| C4—C5 | 1.365 (4) | C10—H10A | 0.9600 |
| C4—N1 | 1.462 (4) | C10—H10B | 0.9600 |
| C5—C6 | 1.358 (4) | C10—H10C | 0.9600 |

| | | | |
|--------------|-------------|---------------|-------------|
| C5—H5 | 0.9300 | N1—O2 | 1.193 (4) |
| C6—H6 | 0.9300 | N1—O1 | 1.202 (4) |
| C7—O3 | 1.443 (3) | O5—H5A | 0.92 (4) |
| C7—C10 | 1.513 (4) | | |
| O3—C1—C2 | 126.6 (2) | C10—C7—C9 | 111.2 (2) |
| O3—C1—C6 | 114.0 (2) | C8—C7—C9 | 107.5 (2) |
| C2—C1—C6 | 119.3 (2) | O4—C8—O5 | 124.3 (2) |
| C3—C2—C1 | 119.6 (2) | O4—C8—C7 | 121.3 (2) |
| C3—C2—H2 | 120.2 | O5—C8—C7 | 114.4 (2) |
| C1—C2—H2 | 120.2 | C7—C9—H9A | 109.5 |
| C4—C3—C2 | 119.3 (3) | C7—C9—H9B | 109.5 |
| C4—C3—H3 | 120.3 | H9A—C9—H9B | 109.5 |
| C2—C3—H3 | 120.3 | C7—C9—H9C | 109.5 |
| C5—C4—C3 | 121.5 (2) | H9A—C9—H9C | 109.5 |
| C5—C4—N1 | 118.6 (2) | H9B—C9—H9C | 109.5 |
| C3—C4—N1 | 119.9 (3) | C7—C10—H10A | 109.5 |
| C6—C5—C4 | 118.9 (2) | C7—C10—H10B | 109.5 |
| C6—C5—H5 | 120.5 | H10A—C10—H10B | 109.5 |
| C4—C5—H5 | 120.5 | C7—C10—H10C | 109.5 |
| C5—C6—C1 | 121.2 (2) | H10A—C10—H10C | 109.5 |
| C5—C6—H6 | 119.4 | H10B—C10—H10C | 109.5 |
| C1—C6—H6 | 119.4 | O2—N1—O1 | 122.1 (3) |
| O3—C7—C10 | 111.1 (2) | O2—N1—C4 | 119.0 (3) |
| O3—C7—C8 | 111.08 (19) | O1—N1—C4 | 118.6 (3) |
| C10—C7—C8 | 112.3 (2) | C1—O3—C7 | 122.60 (18) |
| O3—C7—C9 | 103.2 (2) | C8—O5—H5A | 111 (2) |
| O3—C1—C2—C3 | -177.1 (3) | O3—C7—C8—O5 | 42.7 (3) |
| C6—C1—C2—C3 | -0.3 (4) | C10—C7—C8—O5 | 167.7 (2) |
| C1—C2—C3—C4 | 0.2 (5) | C9—C7—C8—O5 | -69.6 (3) |
| C2—C3—C4—C5 | 0.2 (5) | C5—C4—N1—O2 | 2.2 (5) |
| C2—C3—C4—N1 | -178.2 (3) | C3—C4—N1—O2 | -179.4 (4) |
| C3—C4—C5—C6 | -0.6 (4) | C5—C4—N1—O1 | -172.6 (4) |
| N1—C4—C5—C6 | 177.8 (3) | C3—C4—N1—O1 | 5.9 (5) |
| C4—C5—C6—C1 | 0.5 (4) | C2—C1—O3—C7 | -22.5 (4) |
| O3—C1—C6—C5 | 177.1 (2) | C6—C1—O3—C7 | 160.6 (2) |
| C2—C1—C6—C5 | -0.1 (4) | C10—C7—O3—C1 | -58.6 (3) |
| O3—C7—C8—O4 | -139.5 (2) | C8—C7—O3—C1 | 67.2 (3) |
| C10—C7—C8—O4 | -14.5 (3) | C9—C7—O3—C1 | -177.9 (2) |
| C9—C7—C8—O4 | 108.2 (3) | | |

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

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|---------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| O5—H5A \cdots O4 ⁱ | 0.92 (4) | 1.75 (4) | 2.659 (3) | 173 (3) |

| | | | | |
|--------------------------|------|------|-----------|-----|
| C6—H6···O1 ⁱⁱ | 0.93 | 2.34 | 3.165 (4) | 147 |
|--------------------------|------|------|-----------|-----|

Symmetry codes: (i) $-x+1/2, -y+3/2, -z+1$; (ii) $x, y+1, z$.