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3-Methyl-1,4-dioxo-1,4-dihydro-naphthalen-2-yl 4-aminobenzoate

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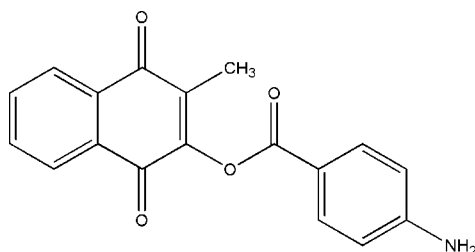
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Key indicators: single-crystal X-ray study; $T = 170$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.042; wR factor = 0.142; data-to-parameter ratio = 11.4.

The crystal structure of the title compound, $\text{C}_{18}\text{H}_{13}\text{NO}_4$, the oxidized form of the drug aminaftone used in venous disease therapy, is characterized by the presence of ribbons of hydrogen-bonded molecules parallel to the [111] crystallographic direction and by stacking interactions between rings [centroid-centroid distance between quinone rings = 3.684 (3) Å and between aminobenzoate rings = 4.157 (3) Å] along the ribbons.

Related literature

For related literature, see: De Anna *et al.* (1989); Martinez *et al.* (2005).



Experimental

Crystal data

$\text{C}_{18}\text{H}_{13}\text{NO}_4$
 $M_r = 307.29$
Triclinic, $P\bar{1}$
 $a = 7.6217$ (6) Å
 $b = 9.6142$ (7) Å

$c = 10.6456$ (7) Å
 $\alpha = 101.618$ (6)°
 $\beta = 110.770$ (7)°
 $\gamma = 89.019$ (6)°
 $V = 713.18$ (10) Å³

$Z = 2$
Cu $K\alpha$ radiation
 $\mu = 0.85$ mm⁻¹

$T = 170$ (2) K
 $0.60 \times 0.20 \times 0.05$ mm

Data collection

Oxford Diffraction Xcalibur PX
Ultra CCD diffractometer
Absorption correction: multi-scan
(*ABSPACK*; Oxford Diffraction, 2006)

$T_{\min} = 0.501$, $T_{\max} = 1.000$
(expected range = 0.480–0.959)
6339 measured reflections
2453 independent reflections
1845 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.022$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.041$
 $wR(F^2) = 0.142$
 $S = 1.12$
2453 reflections
215 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.17$ e Å⁻³
 $\Delta\rho_{\min} = -0.23$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N-H2N\cdots O3^i$	0.95 (3)	2.13 (3)	2.960 (2)	145.6 (19)
$N-H1N\cdots O4^{ii}$	0.89 (2)	2.25 (2)	3.045 (2)	147.3 (19)

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

Data collection: *CrysAlisPro CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlisPro CCD*; data reduction: *CrysAlisPro RED* (Oxford Diffraction, 2006); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RK2078).

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3-Methyl-1,4-dioxo-1,4-dihydronaphthalen-2-yl 4-aminobenzoate

Massimo Bambagiotti-Alberti, Gianluca Bartolucci, Bruno Bruni, Silvia Coran and Massimo Di Vaira

S1. Comment

Previous studies concerning the quality control of aminonaftone, 4-aminobenzoic acid 1,4-dihydroxy-3-methyl-naphthalen-2-yl ester, which is the active pharmaceutical ingredient of some commercial drugs used in the therapy of chronic venous and lymphatic stasis (De Anna *et al.*, 1989; Martinez *et al.*, 2005), have shown that this drug substance rapidly undergoes oxidation in solution at room temperature. The oxidized compound, featuring a quinone ring instead of a hydroquinone moiety, is also a potential impurity of the bulk drug. A structure determination of the oxidized form, **I**, has been undertaken at 170 K.

There are two molecules of **I**, related by the inversion centre, in the triclinic unit cell. The molecular geometry and labelling are shown in Fig 1. Bond distances are consistent with the presence of the quinonic form. Each molecule behaves as a hydrogen-bond donor, toward two other molecules, through its aminic H atoms: N—H2N \cdots O3ⁱ (N \cdots O3ⁱ = 2.960 (2) Å, N—H2N \cdots O3ⁱ = 146 (2)°; symmetry code (i): 2 - x, 2 - y, 1 - z) and N—H1N \cdots O4ⁱⁱ (N \cdots O4ⁱⁱ = 3.045 (2) Å, N—H1N \cdots O4ⁱⁱ = 147 (2)°; symmetry code (ii): 1 + x, 1 + y, 1 + z). Conversely, through the above hydrogen bonds each molecule behaves as an acceptor from two separate molecules, by means of its quinone O atoms. As a result, ribbons of hydrogen-bonded molecules, parallel to the crystallographic [1 1 1] direction, are formed (Fig. 2). Moreover, stacking interactions occur along the ribbons, with 3.684 (3) Å distance between the centroids of symmetry-related quinone rings and 4.157 (3) Å distance between the centroids of the aminobenzoic rings, the shortest C \cdots C contact distances between atoms of facing rings being C8 \cdots C16ⁱⁱⁱ = 3.532 (3) Å ((iii): 1 - x, 1 - y, -z) and C2 \cdots C3ⁱ = 3.286 (2) Å, respectively, for the above two types of interactions. The largest deviation (0.098 (1) Å) from the plane of the aminobenzoic group is presented by the carbonylic O2 atom whereas, among the atoms lying on the naphthoquinone plane, the hinge atom O1 exhibits the largest deviation (0.141 (2) Å) from that plane; the angle between these two planes measures 84.46 (3)°.

S2. Experimental

Samples of aminonaftone were kindly provided by SIMS (SIMS srl, Reggello Firenze, Italy). Crystals of **I**, suitable for X-ray diffraction analysis, were obtained by slow evaporation from methanol solutions of aminonaftone.

S3. Refinement

Crystals did not diffract strongly and it was deemed that collecting data at θ higher than 72° would not yield improvement. Hydrogen atoms were in geometrically generated positions, riding, except for the amino H atoms, which were refined freely. The constraint $U(H) = 1.2U_{eq}(C,N)$, or $1.5U_{eq}(C)$ for methyl group H atoms, was applied. Range of bond distances involving refined hydrogen atoms: N—H 0.89–0.95 Å.

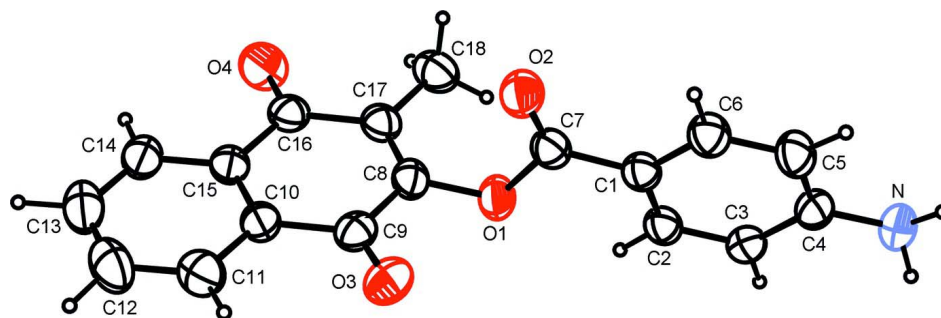


Figure 1

A view of the molecule of **I** with the atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are presented as a small spheres of arbitrary radius.

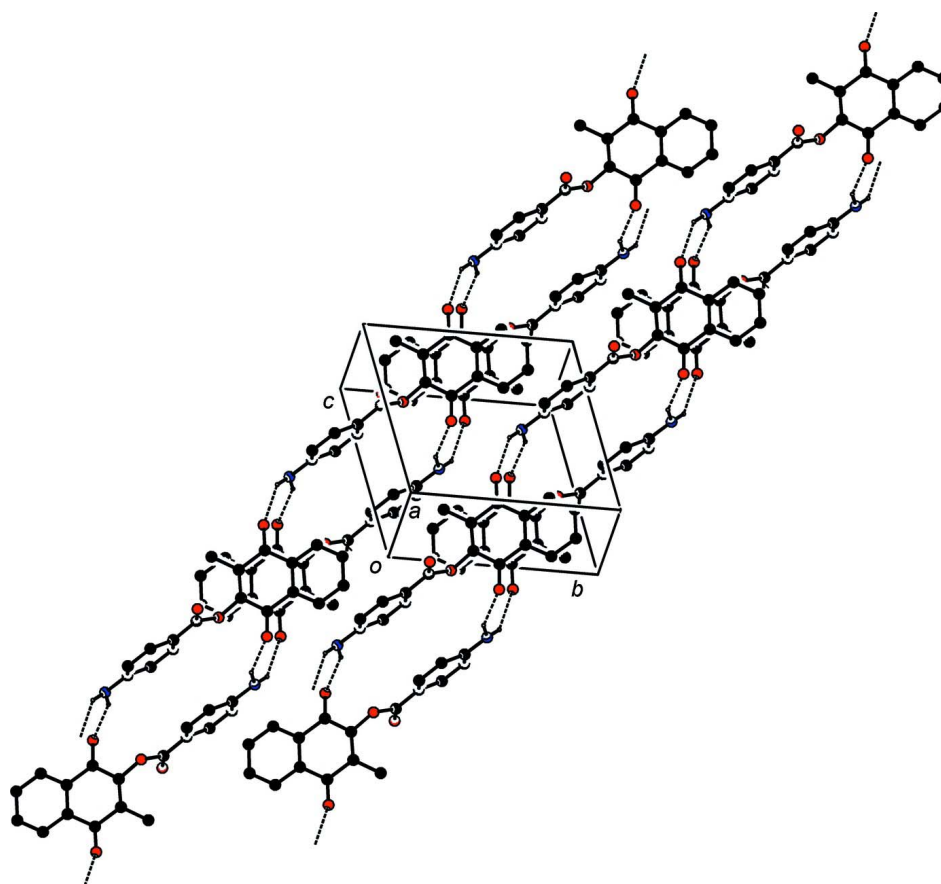


Figure 2

A view of the crystal packing in the structure of **I**, showing the presence of ribbons formed by H-bonded molecules. H-bonds are denoted by dashed lines.

3-Methyl-1,4-dioxo-1,4-dihydronaphthalen-2-yl 4-aminobenzoate

Crystal data

$C_{18}H_{13}NO_4$
 $M_r = 307.29$
 Triclinic, $P\bar{1}$

Hall symbol: $-P\ 1$
 $a = 7.6217(6)\ \text{\AA}$
 $b = 9.6142(7)\ \text{\AA}$

$c = 10.6456 (7) \text{ \AA}$
 $\alpha = 101.618 (6)^\circ$
 $\beta = 110.770 (7)^\circ$
 $\gamma = 89.019 (6)^\circ$
 $V = 713.18 (10) \text{ \AA}^3$
 $Z = 2$
 $F(000) = 320$
 $D_x = 1.431 \text{ Mg m}^{-3}$

Cu $K\alpha$ radiation, $\lambda = 1.54180 \text{ \AA}$
 Cell parameters from 3411 reflections
 $\theta = 9.6\text{--}53.4^\circ$
 $\mu = 0.85 \text{ mm}^{-1}$
 $T = 170 \text{ K}$
 Flat prism, red
 $0.60 \times 0.20 \times 0.05 \text{ mm}$

Data collection

Oxford Diffraction Xcalibur PX Ultra CCD diffractometer
 Radiation source: Fine-focus sealed tube
 Oxford Diffraction Enhance ULTRA assembly monochromator
 Detector resolution: $8.1241 \text{ pixels mm}^{-1}$
 ω scans
 Absorption correction: multi-scan (ABSPACK; Oxford Diffraction, 2006)

$T_{\min} = 0.501, T_{\max} = 1.000$
 6339 measured reflections
 2453 independent reflections
 1845 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.022$
 $\theta_{\max} = 72.4^\circ, \theta_{\min} = 4.5^\circ$
 $h = -9 \rightarrow 9$
 $k = -11 \rightarrow 11$
 $l = -12 \rightarrow 12$

Refinement

Refinement on F^2
 Least-squares matrix: Full
 $R[F^2 > 2\sigma(F^2)] = 0.041$
 $wR(F^2) = 0.142$
 $S = 1.12$
 2453 reflections
 215 parameters
 0 restraints
 Primary atom site location: Direct

Secondary atom site location: Difmap
 Hydrogen site location: Difmap
 H atoms treated by a mixture of independent and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0941P)^2 + 0.0064P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.17 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.23 \text{ e \AA}^{-3}$

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.6680 (2)	1.03157 (16)	0.34314 (16)	0.0361 (4)
C2	0.8411 (2)	1.01124 (16)	0.32984 (16)	0.0373 (4)
H2	0.8613	0.9234	0.2787	0.045*
C3	0.9857 (3)	1.11583 (17)	0.38923 (16)	0.0392 (4)
H3	1.1038	1.0996	0.3791	0.047*
C4	0.9568 (3)	1.24645 (17)	0.46471 (16)	0.0398 (4)
N	1.0987 (3)	1.35138 (17)	0.52199 (17)	0.0508 (4)
H1N	1.083 (3)	1.425 (3)	0.582 (2)	0.061*
H2N	1.225 (4)	1.326 (2)	0.542 (2)	0.061*

C5	0.7833 (3)	1.26644 (17)	0.47801 (17)	0.0449 (5)
H5	0.7624	1.3542	0.5289	0.054*
C6	0.6405 (3)	1.16127 (18)	0.41876 (17)	0.0431 (4)
H6	0.5226	1.1770	0.4294	0.052*
C7	0.5122 (3)	0.92331 (17)	0.27898 (16)	0.0384 (4)
O1	0.57285 (17)	0.79775 (12)	0.22176 (13)	0.0464 (4)
O2	0.35157 (19)	0.93363 (13)	0.27039 (14)	0.0498 (4)
C8	0.4401 (2)	0.68767 (16)	0.14533 (17)	0.0377 (4)
C9	0.4125 (2)	0.58405 (18)	0.22396 (17)	0.0386 (4)
O3	0.48719 (18)	0.60587 (14)	0.34797 (12)	0.0497 (4)
C10	0.2922 (2)	0.45428 (17)	0.14241 (18)	0.0388 (4)
C11	0.2663 (3)	0.3494 (2)	0.2090 (2)	0.0498 (5)
H11	0.3227	0.3623	0.3060	0.060*
C12	0.1572 (3)	0.2263 (2)	0.1317 (2)	0.0602 (6)
H12	0.1414	0.1536	0.1761	0.072*
C13	0.0713 (3)	0.2085 (2)	-0.0091 (2)	0.0552 (5)
H13	-0.0036	0.1239	-0.0607	0.066*
C14	0.0936 (3)	0.31287 (18)	-0.0752 (2)	0.0466 (5)
H14	0.0330	0.3008	-0.1719	0.056*
C15	0.2056 (2)	0.43605 (16)	0.00057 (17)	0.0376 (4)
O4	0.1557 (2)	0.53290 (14)	-0.19534 (13)	0.0593 (4)
C16	0.2311 (2)	0.54714 (17)	-0.07165 (17)	0.0405 (4)
C17	0.3535 (2)	0.67684 (17)	0.01047 (18)	0.0393 (4)
C18	0.3757 (3)	0.7879 (2)	-0.0642 (2)	0.0516 (5)
H181	0.4980	0.8401	-0.0144	0.077*
H182	0.2753	0.8541	-0.0699	0.077*
H183	0.3683	0.7421	-0.1570	0.077*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0393 (10)	0.0345 (8)	0.0337 (8)	0.0027 (8)	0.0114 (7)	0.0083 (6)
C2	0.0443 (10)	0.0325 (7)	0.0329 (8)	0.0035 (8)	0.0118 (7)	0.0059 (6)
C3	0.0385 (10)	0.0398 (8)	0.0357 (8)	0.0033 (8)	0.0088 (7)	0.0085 (7)
C4	0.0435 (11)	0.0382 (8)	0.0318 (8)	-0.0001 (8)	0.0065 (7)	0.0066 (6)
N	0.0484 (10)	0.0413 (8)	0.0487 (9)	-0.0045 (8)	0.0063 (7)	-0.0016 (7)
C5	0.0529 (12)	0.0360 (8)	0.0417 (10)	0.0019 (9)	0.0175 (8)	-0.0017 (7)
C6	0.0449 (11)	0.0407 (9)	0.0436 (9)	0.0035 (8)	0.0176 (8)	0.0053 (7)
C7	0.0443 (11)	0.0347 (8)	0.0362 (8)	0.0032 (8)	0.0136 (7)	0.0091 (6)
O1	0.0370 (7)	0.0339 (6)	0.0615 (8)	-0.0006 (5)	0.0156 (6)	-0.0013 (5)
O2	0.0410 (8)	0.0444 (7)	0.0622 (8)	0.0016 (6)	0.0202 (6)	0.0040 (6)
C8	0.0326 (9)	0.0313 (7)	0.0480 (10)	0.0026 (7)	0.0164 (7)	0.0021 (7)
C9	0.0322 (9)	0.0423 (8)	0.0402 (9)	0.0065 (8)	0.0137 (7)	0.0050 (7)
O3	0.0438 (8)	0.0612 (8)	0.0397 (7)	0.0022 (6)	0.0124 (5)	0.0057 (6)
C10	0.0331 (9)	0.0366 (8)	0.0476 (10)	0.0031 (8)	0.0153 (7)	0.0092 (7)
C11	0.0484 (12)	0.0500 (10)	0.0583 (11)	0.0063 (9)	0.0248 (9)	0.0173 (9)
C12	0.0629 (14)	0.0438 (10)	0.0877 (16)	-0.0002 (10)	0.0405 (12)	0.0192 (10)
C13	0.0464 (12)	0.0389 (9)	0.0817 (15)	-0.0021 (9)	0.0316 (10)	-0.0003 (9)

C14	0.0366 (10)	0.0391 (9)	0.0578 (11)	0.0037 (8)	0.0157 (8)	-0.0017 (8)
C15	0.0320 (9)	0.0330 (8)	0.0472 (10)	0.0068 (7)	0.0168 (7)	0.0032 (7)
O4	0.0774 (11)	0.0530 (7)	0.0385 (7)	0.0067 (7)	0.0124 (6)	0.0053 (6)
C16	0.0386 (10)	0.0385 (8)	0.0414 (10)	0.0089 (8)	0.0121 (7)	0.0064 (7)
C17	0.0368 (10)	0.0355 (8)	0.0477 (10)	0.0073 (8)	0.0173 (8)	0.0095 (7)
C18	0.0535 (12)	0.0465 (10)	0.0626 (12)	0.0078 (9)	0.0245 (9)	0.0222 (9)

Geometric parameters (Å, °)

C1—C2	1.383 (3)	C9—O3	1.213 (2)
C1—C6	1.398 (2)	C9—C10	1.480 (2)
C1—C7	1.463 (2)	C10—C15	1.392 (2)
C2—C3	1.383 (2)	C10—C11	1.397 (2)
C2—H2	0.9500	C11—C12	1.387 (3)
C3—C4	1.407 (2)	C11—H11	0.9500
C3—H3	0.9500	C12—C13	1.382 (3)
C4—N	1.371 (2)	C12—H12	0.9500
C4—C5	1.385 (3)	C13—C14	1.381 (3)
N—H1N	0.89 (2)	C13—H13	0.9500
N—H2N	0.95 (3)	C14—C15	1.394 (2)
C5—C6	1.378 (2)	C14—H14	0.9500
C5—H5	0.9500	C15—C16	1.488 (2)
C6—H6	0.9500	O4—C16	1.215 (2)
C7—O2	1.199 (2)	C16—C17	1.487 (2)
C7—O1	1.388 (2)	C17—C18	1.498 (2)
O1—C8	1.3827 (19)	C18—H181	0.9800
C8—C17	1.333 (2)	C18—H182	0.9800
C8—C9	1.485 (2)	C18—H183	0.9800
C2—C1—C6	118.63 (15)	C15—C10—C11	120.01 (16)
C2—C1—C7	122.37 (15)	C15—C10—C9	120.57 (14)
C6—C1—C7	118.99 (16)	C11—C10—C9	119.42 (16)
C1—C2—C3	121.52 (15)	C12—C11—C10	119.25 (19)
C1—C2—H2	119.2	C12—C11—H11	120.4
C3—C2—H2	119.2	C10—C11—H11	120.4
C2—C3—C4	119.58 (17)	C13—C12—C11	120.62 (17)
C2—C3—H3	120.2	C13—C12—H12	119.7
C4—C3—H3	120.2	C11—C12—H12	119.7
N—C4—C5	121.40 (16)	C14—C13—C12	120.40 (17)
N—C4—C3	119.83 (18)	C14—C13—H13	119.8
C5—C4—C3	118.76 (15)	C12—C13—H13	119.8
C4—N—H1N	116.7 (15)	C13—C14—C15	119.68 (18)
C4—N—H2N	119.0 (13)	C13—C14—H14	120.2
H1N—N—H2N	114 (2)	C15—C14—H14	120.2
C6—C5—C4	121.14 (16)	C10—C15—C14	120.02 (16)
C6—C5—H5	119.4	C10—C15—C16	120.62 (14)
C4—C5—H5	119.4	C14—C15—C16	119.37 (16)
C5—C6—C1	120.36 (17)	O4—C16—C17	120.01 (15)

C5—C6—H6	119.8	O4—C16—C15	121.32 (15)
C1—C6—H6	119.8	C17—C16—C15	118.67 (14)
O2—C7—O1	121.27 (15)	C8—C17—C16	118.88 (14)
O2—C7—C1	128.52 (16)	C8—C17—C18	123.35 (16)
O1—C7—C1	110.20 (15)	C16—C17—C18	117.75 (15)
C8—O1—C7	118.14 (13)	C17—C18—H181	109.5
C17—C8—O1	120.33 (14)	C17—C18—H182	109.5
C17—C8—C9	124.77 (14)	H181—C18—H182	109.5
O1—C8—C9	114.83 (14)	C17—C18—H183	109.5
O3—C9—C10	122.82 (15)	H181—C18—H183	109.5
O3—C9—C8	120.89 (15)	H182—C18—H183	109.5
C10—C9—C8	116.28 (14)		
C6—C1—C2—C3	-0.1 (2)	C8—C9—C10—C11	177.37 (17)
C7—C1—C2—C3	178.82 (14)	C15—C10—C11—C12	1.4 (3)
C1—C2—C3—C4	-0.2 (2)	C9—C10—C11—C12	-178.40 (17)
C2—C3—C4—N	-178.97 (15)	C10—C11—C12—C13	-1.5 (3)
C2—C3—C4—C5	0.3 (2)	C11—C12—C13—C14	0.3 (3)
N—C4—C5—C6	179.17 (15)	C12—C13—C14—C15	0.8 (3)
C3—C4—C5—C6	-0.1 (3)	C11—C10—C15—C14	-0.3 (3)
C4—C5—C6—C1	-0.2 (3)	C9—C10—C15—C14	179.56 (16)
C2—C1—C6—C5	0.3 (2)	C11—C10—C15—C16	179.70 (16)
C7—C1—C6—C5	-178.65 (15)	C9—C10—C15—C16	-0.5 (3)
C2—C1—C7—O2	-171.14 (16)	C13—C14—C15—C10	-0.9 (3)
C6—C1—C7—O2	7.8 (3)	C13—C14—C15—C16	179.15 (17)
C2—C1—C7—O1	7.5 (2)	C10—C15—C16—O4	-179.69 (17)
C6—C1—C7—O1	-173.56 (14)	C14—C15—C16—O4	0.3 (3)
O2—C7—O1—C8	4.8 (2)	C10—C15—C16—C17	1.0 (3)
C1—C7—O1—C8	-174.01 (13)	C14—C15—C16—C17	-179.01 (16)
C7—O1—C8—C17	89.9 (2)	O1—C8—C17—C16	171.84 (14)
C7—O1—C8—C9	-92.86 (17)	C9—C8—C17—C16	-5.1 (3)
C17—C8—C9—O3	-175.24 (17)	O1—C8—C17—C18	-7.0 (3)
O1—C8—C9—O3	7.7 (2)	C9—C8—C17—C18	176.11 (16)
C17—C8—C9—C10	5.5 (3)	O4—C16—C17—C8	-177.60 (17)
O1—C8—C9—C10	-171.56 (14)	C15—C16—C17—C8	1.7 (2)
O3—C9—C10—C15	178.30 (17)	O4—C16—C17—C18	1.3 (3)
C8—C9—C10—C15	-2.5 (2)	C15—C16—C17—C18	-179.42 (16)
O3—C9—C10—C11	-1.9 (3)		

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N—H2N \cdots O3 ⁱ	0.95 (3)	2.13 (3)	2.960 (2)	145.6 (19)
N—H1N \cdots O4 ⁱⁱ	0.89 (2)	2.25 (2)	3.045 (2)	147.3 (19)

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