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ISSN 2414-3146

tert-Butyl 3-(3-methyl-1-oxidopyridin-1-ium-2-yl)-benzoate

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Received 12 December 2015
Accepted 29 December 2015

Edited by G. Smith, Queensland University of Technology, Australia

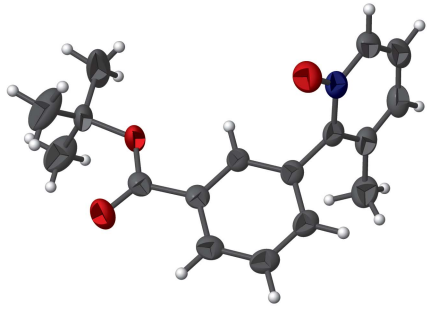
Keywords: crystal structure; hydrogen bonding; *N*-oxide; π–π ring interactions; lumacaftor.

CCDC reference: 1444673

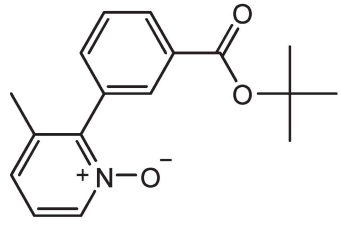
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{17}H_{19}NO_3$, which was obtained by oxidation of the corresponding pyridine derivative, the dihedral angle between the benzene and the pyridine rings is 68.2 (1)°. In the crystal, C—H···O hydrogen bonds to carboxyl and *N*-oxide O-atom acceptors gives a cyclic dimer substructure with an $R_2^2(18)$ motif which is extended into a undulating sheet structure lying parallel to (100) through weak C—H···O_{oxide} hydrogen bonds. Also present are π–π ring interactions [ring centroid separation = 3.561 (2) Å].

3D view



Chemical scheme



Structure description

The title compound, $C_{17}H_{19}NO_3$, is a key intermediate in the synthesis of the experimental drug lumacaftor for the therapy of cystic fibrosis (Norman, 2014; McColley, 2014).

The benzene and pyridine rings give a twisted conformation to the molecule (Fig. 1), with an interplanar dihedral angle of 68.2 (1)°. The carboxyl group is essentially coplanar with the benzene ring [torsion angle C12—C11—C14—O2 = −174.7 (2)°]. The methyl C atoms of the *tert*-butyl group display somewhat elongated ellipsoids which is not unusual for this group. In the crystal there is an absence of classic hydrogen bonding, but dual C—H···O hydrogen-bonding interactions to carboxyl and oxide O-atom acceptors (C12—H12···O2^{i} and C4—H4···O1^{i}, respectively; Table 1) give a cyclic dimer substructure (Fig. 2), with an $R_2^2(18)$ motif (Bernstein *et al.*, 1995). The cyclic aggregates are arranged in rows along *c* (Fig. 3), which are linked through weak C8—H8···O1^{ii} hydrogen bonds, forming zigzag layered structures which lie parallel to (100) (Fig. 4). Similar hydrogen-bonding contacts have been observed in other pyridine oxides (McKay *et al.*, 2006; Babu

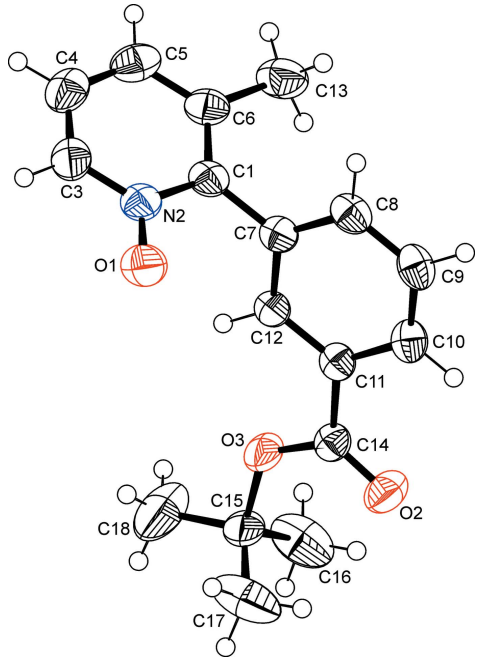


Figure 1
The molecular structure of the title compound, with atom labels and 50% probability displacement ellipsoids for non-H atoms.

et al., 2007; Bowers *et al.*, 2005). Present also in the crystal are π–π ring interactions between inversion-related pyridine rings [ring centroid separation = 3.561 (2) Å].

Synthesis and crystallization

The title compound was synthesized by stirring 2-(3-(*tert*-butoxycarbonyl)phenyl)-3-methylpyridine (Siesel, 2009) and *m*-chloroperoxybenzoic acid in dichloromethane. The mixture

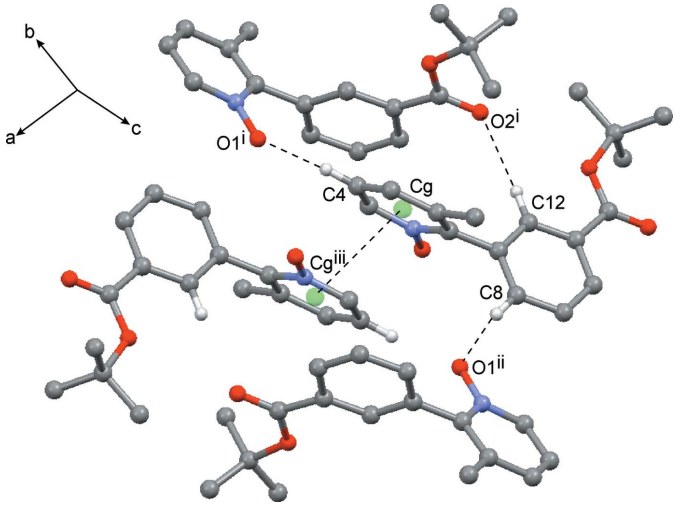


Figure 2
Short C—H···O and π–π contacts in the crystal structure of the title compound. H atoms engaged in hydrogen bonding are drawn as spheres and all other H atoms are omitted. Symmetry code: (iii) $-x+1, -y, -z+1$. For other codes, see Table 1.

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···O1^{i}	0.95	2.44	3.204 (3)	137
C12—H12···O2^{i}	0.95	2.39	3.327 (2)	169
C8—H8···O1^{ii}	0.95	2.50	3.438 (3)	169

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

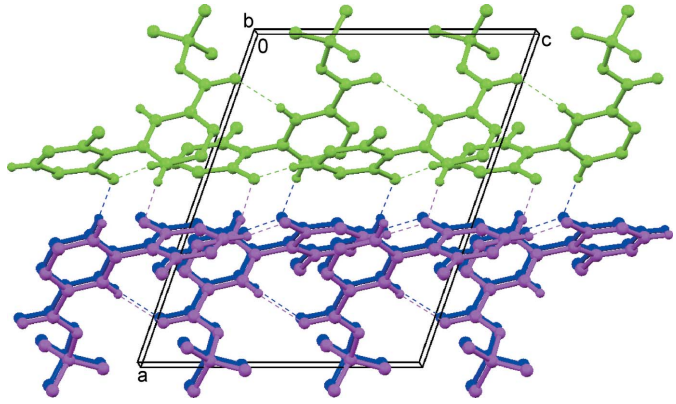


Figure 3
Rows of cyclic hydrogen-bond aggregates along *c*.

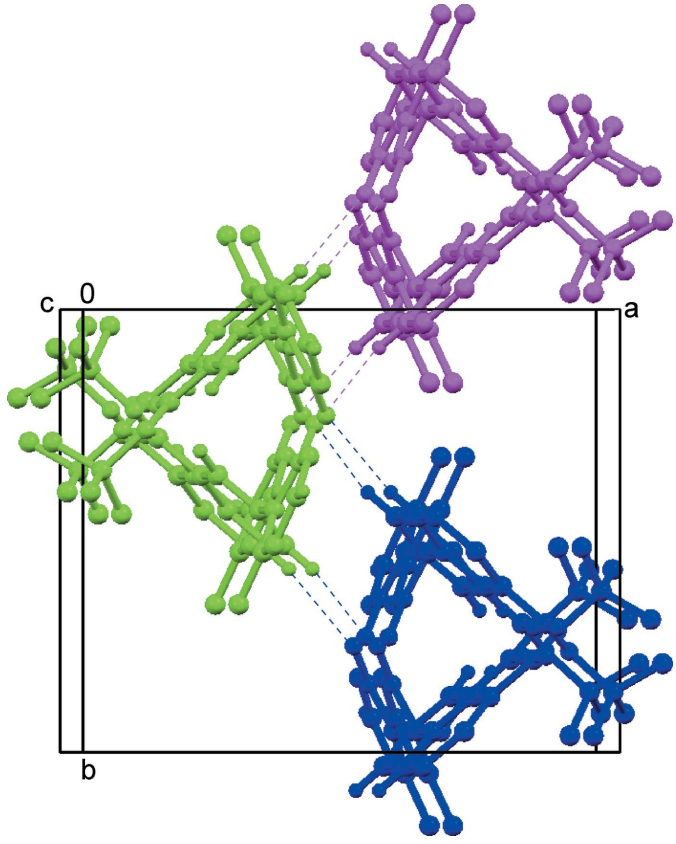


Figure 4
The undulating sheet structure extending along the general *b*-axis direction.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{17}H_{19}NO_3$
M_r	285.33
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	173
a, b, c (Å)	13.6690 (7), 10.8170 (6), 10.9271 (7)
β (°)	109.365 (7)
V (Å^3)	1524.25 (16)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.69
Crystal size (mm)	0.16 × 0.11 × 0.1
Data collection	
Diffractometer	Agilent Xcalibur, Ruby, Gemini ultra
Absorption correction	Multi-scan (*CrysAlis PRO*; Agilent, 2014)
T_{min}, T_{max}	0.884, 1
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	15403, 2730, 1984
R_{int}	0.066
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.600
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.053, 0.158, 1.04
No. of reflections	2730
No. of parameters	194
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.19, −0.20

Computer programs: *CrysAlis PRO* (Agilent, 2014), *SIR2002* (Burla *et al.*, 2003), *SHELXL2014* (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012), *Mercury* (Macrae *et al.*, 2006).

was treated with solid sodium sulfite, potassium carbonate and magnesium sulfate and the solvent was removed (Bremner *et al.*, 1997). The resulting viscous oil crystallized after two weeks at room temperature giving the title compound (m.p. 352–354 K).

^{1}H NMR (DMSO-d_6, 300 MHz): δ 1.54 (*s*, 9H), 2.03 (*s*, 3H), 7.34–7.38 (*m*, 2H), 7.57–7.65 (*m*, 2H), 7.85 (*s*, 1H), 7.97 (*d*, J = 7.1 Hz, 1H), 8.23 (*d*, J = 5.3 Hz, 1H) p.p.m. ^{13}C NMR (DMSO-d_6, 75 MHz): δ 19.3 (CH_3), 27.7 (3 CH_3), 80.9 (C), 124.9 (CH), 127.1 (CH), 128.8 (CH), 129.1 (CH), 130.1 (CH), 131.6 (C), 132.8 (C), 134.0 (CH), 135.5 (C), 137.1 (CH), 147.5 (C), 164.6 (C═O) p.p.m. IR (neat): ν 3066, 2975, 2931, 1411, 1366, 1312, 1254, 1230, 1161, 1118, 1082, 1050, 959, 850, 787, 757, 738, 698, 569 cm^{-1}.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

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full crystallographic data

IUCrData (2016). **1**, x152490 [doi:10.1107/S2414314615024906]

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Crystal data

$C_{17}H_{19}NO_3$
M_r = 285.33
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 13.6690 (7) Å
b = 10.8170 (6) Å
c = 10.9271 (7) Å
β = 109.365 (7)°
V = 1524.25 (16) Å^3
Z = 4
$F(000)$ = 608
D_x = 1.243 Mg m^{-3}
Melting point = 352–354 K
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 3088 reflections
θ = 3.4–66.0°
μ = 0.69 mm^{-1}
T = 173 K
Prismatic fragment, colourless
0.16 × 0.11 × 0.1 mm

Data collection

Agilent Xcalibur, Ruby, Gemini ultra diffractometer
Radiation source: sealed X-ray tube
Mirror monochromator
Detector resolution: 10.3575 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2014)
T_{min} = 0.884, T_{max} = 1
15403 measured reflections
2730 independent reflections
1984 reflections with $I > 2\sigma(I)$
R_{int} = 0.066
θ_{max} = 67.7°, θ_{min} = 3.4°
h = −16→13
k = −12→12
l = −13→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.158
S = 1.04
2730 reflections
194 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
$w = 1/[\sigma^2(F_o^2) + (0.0816P)^2 + 0.290P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Special details

Experimental. Absorption correction: *CrysAlis PRO* (Agilent, 2014). Agilent Technologies, Version 1.171.37.31 (release 14–01-2014 CrysAlis171. NET) (compiled Jan 14 2014,18:38:05) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O3	0.10663 (13)	0.27239 (17)	0.77336 (16)	0.0605 (5)
O2	0.13706 (12)	0.21671 (17)	0.97944 (16)	0.0562 (5)
O1	0.43812 (13)	0.23977 (15)	0.67190 (18)	0.0573 (5)
N2	0.40653 (13)	0.15378 (17)	0.58465 (19)	0.0442 (5)
C11	0.23596 (14)	0.12519 (18)	0.86088 (19)	0.0367 (5)
C12	0.26102 (14)	0.12596 (18)	0.7478 (2)	0.0362 (5)
H12	0.2256	0.1801	0.6788	0.043*
C9	0.36423 (15)	−0.0318 (2)	0.9494 (2)	0.0464 (5)
H9	0.3995	−0.0859	1.0184	0.056*
C7	0.33842 (14)	0.04709 (19)	0.7351 (2)	0.0383 (5)
C10	0.28839 (15)	0.0467 (2)	0.9625 (2)	0.0423 (5)
H10	0.2721	0.0471	1.0405	0.051*
C1	0.36110 (14)	0.04809 (19)	0.6119 (2)	0.0407 (5)
C8	0.38909 (15)	−0.0320 (2)	0.8361 (2)	0.0444 (5)
H8	0.441	−0.0865	0.8277	0.053*
C14	0.15541 (15)	0.20928 (19)	0.8791 (2)	0.0387 (5)
C6	0.33577 (16)	−0.0479 (2)	0.5220 (2)	0.0474 (5)
C5	0.35176 (17)	−0.0325 (3)	0.4037 (2)	0.0563 (6)
H5	0.3352	−0.0976	0.3419	0.068*
C3	0.41890 (16)	0.1679 (2)	0.4671 (2)	0.0507 (6)
H3	0.4472	0.2428	0.448	0.061*
C15	0.02417 (19)	0.3638 (2)	0.7682 (2)	0.0550 (6)
C4	0.39149 (17)	0.0768 (3)	0.3761 (2)	0.0556 (6)
H4	0.3997	0.0887	0.2939	0.067*
C13	0.2918 (2)	−0.1661 (2)	0.5543 (3)	0.0640 (7)
H13A	0.2693	−0.2192	0.4773	0.096*
H13B	0.2323	−0.1472	0.5822	0.096*
H13C	0.3451	−0.2089	0.6243	0.096*
C16	−0.0668 (2)	0.2999 (3)	0.7882 (4)	0.0834 (10)
H16A	−0.1239	0.3589	0.7744	0.125*
H16B	−0.0467	0.2675	0.8768	0.125*
H16C	−0.0894	0.2316	0.7263	0.125*
C17	0.0654 (2)	0.4624 (3)	0.8682 (4)	0.0920 (12)
H17A	0.129	0.4971	0.8596	0.138*
H17B	0.0806	0.4267	0.955	0.138*
H17C	0.0135	0.528	0.8556	0.138*

C18	−0.0024 (4)	0.4125 (6)	0.6342 (4)	0.165 (3)
H18A	0.0609	0.4401	0.6191	0.248*
H18B	−0.0502	0.4825	0.6229	0.248*
H18C	−0.0356	0.3473	0.5721	0.248*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O3	0.0708 (11)	0.0713 (11)	0.0529 (10)	0.0386 (9)	0.0385 (8)	0.0204 (8)
O2	0.0531 (9)	0.0745 (12)	0.0468 (9)	0.0113 (8)	0.0244 (7)	0.0025 (8)
O1	0.0585 (10)	0.0479 (9)	0.0741 (11)	−0.0125 (7)	0.0335 (8)	−0.0086 (9)
N2	0.0360 (9)	0.0454 (10)	0.0567 (11)	0.0049 (7)	0.0228 (8)	0.0012 (9)
C11	0.0303 (9)	0.0366 (10)	0.0434 (11)	−0.0046 (8)	0.0127 (8)	0.0008 (9)
C12	0.0314 (9)	0.0351 (10)	0.0436 (11)	−0.0008 (8)	0.0145 (8)	0.0011 (9)
C9	0.0358 (10)	0.0452 (12)	0.0534 (13)	0.0003 (9)	0.0084 (9)	0.0118 (10)
C7	0.0314 (9)	0.0368 (10)	0.0482 (12)	−0.0021 (8)	0.0151 (8)	−0.0014 (9)
C10	0.0342 (10)	0.0456 (12)	0.0462 (12)	−0.0058 (9)	0.0122 (9)	0.0053 (10)
C1	0.0290 (9)	0.0414 (11)	0.0535 (13)	0.0075 (8)	0.0163 (9)	0.0025 (10)
C8	0.0325 (10)	0.0405 (12)	0.0584 (14)	0.0031 (8)	0.0126 (9)	0.0040 (10)
C14	0.0349 (10)	0.0416 (11)	0.0414 (12)	−0.0049 (8)	0.0150 (9)	−0.0008 (9)
C6	0.0381 (10)	0.0475 (12)	0.0578 (14)	0.0077 (9)	0.0177 (10)	−0.0038 (11)
C5	0.0464 (12)	0.0679 (16)	0.0557 (14)	0.0099 (11)	0.0185 (11)	−0.0111 (13)
C3	0.0404 (11)	0.0578 (14)	0.0605 (15)	0.0124 (10)	0.0255 (11)	0.0132 (12)
C15	0.0586 (14)	0.0583 (14)	0.0602 (14)	0.0281 (11)	0.0360 (12)	0.0115 (12)
C4	0.0412 (11)	0.0764 (18)	0.0542 (14)	0.0150 (11)	0.0224 (10)	0.0082 (13)
C13	0.0694 (16)	0.0486 (14)	0.0786 (18)	−0.0065 (12)	0.0306 (14)	−0.0162 (13)
C16	0.0440 (13)	0.0639 (18)	0.132 (3)	0.0037 (12)	0.0153 (16)	−0.0009 (18)
C17	0.0592 (16)	0.0502 (16)	0.160 (3)	0.0044 (13)	0.0279 (19)	−0.0169 (19)
C18	0.219 (5)	0.216 (6)	0.103 (3)	0.184 (5)	0.111 (3)	0.098 (3)

Geometric parameters (Å, º)

O3—C14	1.317 (3)	C5—C4	1.376 (4)
O3—C15	1.487 (3)	C5—H5	0.95
O2—C14	1.206 (3)	C3—C4	1.361 (4)
O1—N2	1.299 (2)	C3—H3	0.95
N2—C3	1.359 (3)	C15—C18	1.483 (4)
N2—C1	1.380 (3)	C15—C17	1.497 (4)
C11—C12	1.387 (3)	C15—C16	1.501 (4)
C11—C10	1.393 (3)	C4—H4	0.95
C11—C14	1.491 (3)	C13—H13A	0.98
C12—C7	1.401 (3)	C13—H13B	0.98
C12—H12	0.95	C13—H13C	0.98
C9—C10	1.383 (3)	C16—H16A	0.98
C9—C8	1.388 (3)	C16—H16B	0.98
C9—H9	0.95	C16—H16C	0.98
C7—C8	1.386 (3)	C17—H17A	0.98
C7—C1	1.479 (3)	C17—H17B	0.98

C10—H10	0.95	C17—H17C	0.98
C1—C6	1.392 (3)	C18—H18A	0.98
C8—H8	0.95	C18—H18B	0.98
C6—C5	1.390 (3)	C18—H18C	0.98
C6—C13	1.504 (4)		

C14—O3—C15	122.35 (17)	N2—C3—H3	119.4
O1—N2—C3	119.87 (19)	C4—C3—H3	119.4
O1—N2—C1	119.92 (18)	C18—C15—O3	102.03 (19)
C3—N2—C1	120.2 (2)	C18—C15—C17	112.2 (3)
C12—C11—C10	120.00 (18)	O3—C15—C17	110.4 (2)
C12—C11—C14	121.76 (18)	C18—C15—C16	111.3 (4)
C10—C11—C14	118.22 (19)	O3—C15—C16	110.0 (2)
C11—C12—C7	120.12 (19)	C17—C15—C16	110.6 (2)
C11—C12—H12	119.9	C3—C4—C5	119.7 (2)
C7—C12—H12	119.9	C3—C4—H4	120.2
C10—C9—C8	120.4 (2)	C5—C4—H4	120.2
C10—C9—H9	119.8	C6—C13—H13A	109.5
C8—C9—H9	119.8	C6—C13—H13B	109.5
C8—C7—C12	119.43 (19)	H13A—C13—H13B	109.5
C8—C7—C1	121.94 (18)	C6—C13—H13C	109.5
C12—C7—C1	118.60 (18)	H13A—C13—H13C	109.5
C9—C10—C11	119.8 (2)	H13B—C13—H13C	109.5
C9—C10—H10	120.1	C15—C16—H16A	109.5
C11—C10—H10	120.1	C15—C16—H16B	109.5
N2—C1—C6	119.4 (2)	H16A—C16—H16B	109.5
N2—C1—C7	116.60 (18)	C15—C16—H16C	109.5
C6—C1—C7	124.0 (2)	H16A—C16—H16C	109.5
C7—C8—C9	120.21 (19)	H16B—C16—H16C	109.5
C7—C8—H8	119.9	C15—C17—H17A	109.5
C9—C8—H8	119.9	C15—C17—H17B	109.5
O2—C14—O3	124.45 (19)	H17A—C17—H17B	109.5
O2—C14—C11	123.18 (19)	C15—C17—H17C	109.5
O3—C14—C11	112.37 (18)	H17A—C17—H17C	109.5
C5—C6—C1	119.2 (2)	H17B—C17—H17C	109.5
C5—C6—C13	121.2 (2)	C15—C18—H18A	109.5
C1—C6—C13	119.6 (2)	C15—C18—H18B	109.5
C4—C5—C6	120.1 (2)	H18A—C18—H18B	109.5
C4—C5—H5	119.9	C15—C18—H18C	109.5
C6—C5—H5	119.9	H18A—C18—H18C	109.5
N2—C3—C4	121.3 (2)	H18B—C18—H18C	109.5

C10—C11—C12—C7	0.6 (3)	C15—O3—C14—C11	−179.0 (2)
C14—C11—C12—C7	178.89 (18)	C12—C11—C14—O2	−174.72 (19)
C11—C12—C7—C8	0.4 (3)	C10—C11—C14—O2	3.6 (3)
C11—C12—C7—C1	178.40 (17)	C12—C11—C14—O3	6.0 (3)
C8—C9—C10—C11	0.6 (3)	C10—C11—C14—O3	−175.61 (18)
C12—C11—C10—C9	−1.1 (3)	N2—C1—C6—C5	−3.4 (3)

C14—C11—C10—C9	−179.44 (19)	C7—C1—C6—C5	173.95 (18)
O1—N2—C1—C6	−174.47 (18)	N2—C1—C6—C13	176.57 (19)
C3—N2—C1—C6	5.1 (3)	C7—C1—C6—C13	−6.1 (3)
O1—N2—C1—C7	8.0 (3)	C1—C6—C5—C4	−0.5 (3)
C3—N2—C1—C7	−172.40 (17)	C13—C6—C5—C4	179.6 (2)
C8—C7—C1—N2	−113.6 (2)	O1—N2—C3—C4	176.59 (19)
C12—C7—C1—N2	68.4 (2)	C1—N2—C3—C4	−3.0 (3)
C8—C7—C1—C6	68.9 (3)	C14—O3—C15—C18	177.7 (4)
C12—C7—C1—C6	−109.0 (2)	C14—O3—C15—C17	58.2 (3)
C12—C7—C8—C9	−0.9 (3)	C14—O3—C15—C16	−64.1 (3)
C1—C7—C8—C9	−178.79 (19)	N2—C3—C4—C5	−0.9 (3)
C10—C9—C8—C7	0.4 (3)	C6—C5—C4—C3	2.6 (3)
C15—O3—C14—O2	1.8 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···O1^{i}	0.95	2.44	3.204 (3)	137
C12—H12···O2^{i}	0.95	2.39	3.327 (2)	169
C8—H8···O1^{ii}	0.95	2.50	3.438 (3)	169

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