Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4-(3-Carboxy-1-ethyl-6-fluoro-4-oxo-1,4-dihydroquinolin-7-yl)piperazin-1-ium 4-carboxybenzoate–benzene-1,4-dicarboxylic acid (2/1)

Shi-Wei Yan,* Hai-Yan Chen, Guang-Ju Zhang, Qin Liao and Yan-Chen Liang

College of Chemistry and Chemical Engineering, Southwest University, Chongqing 400715, People's Republic of China
Correspondence e-mail: yanshiwei158@163.com

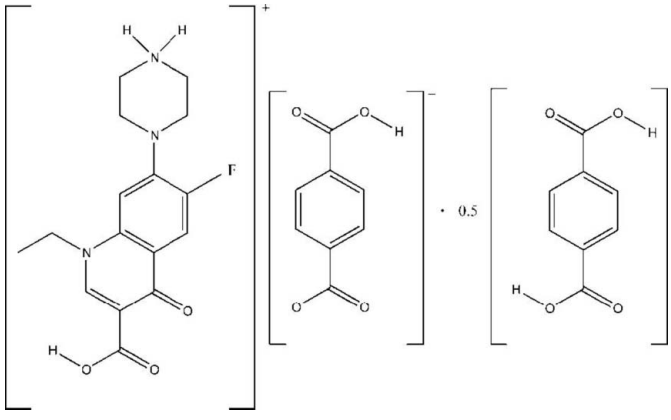
Received 2 March 2011; accepted 13 March 2011

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.002 Å; R factor = 0.042; wR factor = 0.137; data-to-parameter ratio = 13.1.

In the title compound, $C_{16}H_{19}FN_3O_3^+\cdot C_8H_5O_4^-\cdot 0.5C_8H_6O_4$, the benzene-1,4-dicarboxylic acid molecule is located on a centre of symmetry. In the crystal, the molecules and ions are connected by intermolecular C—H···O and O—H···O hydrogen bonds and π–π stacking interactions [with a centroid–centroid distance of 3.402 (2) Å], generating a three-dimensional supramolecular structure.

Related literature

For general background to the use of quinolones in the treatment of infections, see: Barbas *et al.* (2006); Basavoju *et al.*(2006); Xiao *et al.* (2005).



Experimental

Crystal data

$C_{16}H_{19}FN_3O_3^+\cdot C_8H_5O_4^-\cdot 0.5C_8H_6O_4$
M_r = 568.53
Triclinic, $P\overline{1}$
a = 9.8901 (15) Å
b = 10.2420 (16) Å
c = 13.665 (2) Å
α = 89.304 (2)°
β = 74.672 (2)°
γ = 71.677 (2)°
V = 1263.5 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 296 K
0.46 × 0.45 × 0.36 mm

Data collection

Bruker APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.948, T_{max} = 0.959
10704 measured reflections
5143 independent reflections
4031 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.137
S = 1.00
5143 reflections
394 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C15—H15B···O1^{i}	0.97	2.38	3.274 (2)	153
O5—H5B···O7^{ii}	0.97 (3)	1.70 (3)	2.6648 (15)	169 (2)

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL-Plus* (Sheldrick, 2008); software used to prepare material for publication: *SHELXL97*.

This work was supported by the Science and Technology Foundation of Southwest University (SWUB2007035).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FF2003).

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supporting information

Acta Cryst. (2011). E**67**, o909 [doi:10.1107/S1600536811009524]

4-(3-Carboxy-1-ethyl-6-fluoro-4-oxo-1,4-dihydroquinolin-7-yl)piperazin-1-ium 4-carboxybenzoate–benzene-1,4-dicarboxylic acid (2/1)

Shi-Wei Yan, Hai-Yan Chen, Guang-Ju Zhang, Qin Liao and Yan-Chen Liang

S1. Comment

Norfloxacin [1–Ethyl–6–fluoro–1,4–dihydro–4–oxo–7–(piperazin–1–yl)quinoline–3–carbo–xylic acid] is member of a class of quinolones used to treat infections (Xiao *et al.*, 2005; Barbas *et al.*, 2006; Basavoju *et al.*, 2006). In this paper,the structure of the title compound, 1, is described (Fig. 1). In compound 1, benzene-1,4-dicarboxylic acid is located on the centre of symmetry. The molecules and the ions are linked by intermolecular C—H···O and O—H···O hydrogen-bonding interactions (C···O = 3.273 (2) Å, O···O = 2.6648 (18) Å) and π—π stacking between the benzene ring of $[H_2norf]^+$ and aromatic ring of 1,4-H_2bdc with the centroid-centroid distance of 3.402 (2) Å, generating a three-dimensional supramolecular structure.

S2. Experimental

A mixture of $Mn(CH_3COO)_2.4H_2O$ (0.061 g, 0.25 mmol), Norfloxacin (0.080 g, 0.25 mmol) and distilled water (10 ml) was stirred for 20 min. in air. The mixture was then transferred to a 23 ml Teflon-lined hydrothermal bomb. The bomb was kept at 393 K for 96 h under autogenous pressure. Upon cooling, yellow block of 1 were obtained from the reaction mixture.

S3. Refinement

The H atoms bonded to C atoms were positioned geometrically and refined using a riding model approximation [aromatic C—H = 0.93 Å, aliphatic C—H = 0.97 Å], with U_{iso}(H) = 1.2–1.5 U_{eq}(C). The H on N atoms were located in a difference Fourier map, and refined with distances restraint of N—H = 0.90 Å–0.95 Å, and with U_{iso}(H) = 1.5–1.7 U_{eq}(N). The H atoms bonded to O atoms were located in a difference Fourier maps and with U_{iso}(H) = 1.4, 1.9 and 2.1 U_{eq}(O) for carboxyl groups of $[H_2norf]^+$, $[1,4\text{-}Hbdc]^-$ and 1,4-H_2bdc, respectively. The O—H bonds are 0.87 Å, 0.97 Å and 1.01 Å in carboxyl groups of $[H_2norf]^+$,$[1,4–Hbdc]^-$ and 1,4–H_2bdc.

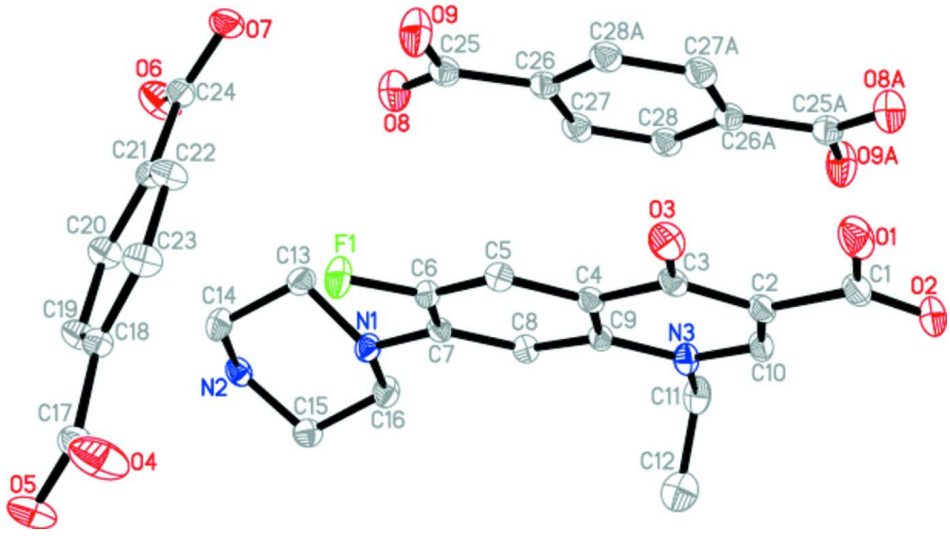

Figure 1

The structure of 1. Displacement ellipsoids are drawn at the 30% probability level. H atoms havebeen omitted for clarity [Symmetry code: -x+2, -y, -z+1].

4-(3-Carboxy-1-ethyl-6-fluoro-4-oxo-1,4-dihydroquinolin-7-yl)piperazin-1-ium 4-carboxybenzoate–benzene-1,4-dicarboxylic acid (2/1)

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$\beta = 74.672\ (2)°$
$\gamma = 71.677\ (2)°$
$V = 1263.5\ (3)$ Å^3
$Z = 2$
$F(000) = 594$
$D_x = 1.494$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 10704 reflections
$\theta = 2.5–26.4°$
$\mu = 0.12$ mm^{-1}
$T = 296$ K
Block, yellow
0.46 × 0.45 × 0.36 mm

Data collection

Bruker APEX CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.948$, $T_{max} = 0.959$
10704 measured reflections
5143 independent reflections
4031 reflections with $I > 2\sigma(I)$
$R_{int} = 0.024$
$\theta_{max} = 26.4°$, $\theta_{min} = 2.5°$
$h = -12 \rightarrow 12$
$k = -12 \rightarrow 12$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.137$
$S = 1.00$
5143 reflections
394 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map	$w = 1/[\sigma^2(F_o^2) + (0.095P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
Hydrogen site location: inferred from neighbouring sites	$(\Delta/\sigma)_{max} < 0.001$
H atoms treated by a mixture of independent and constrained refinement	$\Delta\rho_{max} = 0.26$ e Å^{-3}
	$\Delta\rho_{min} = -0.28$ e Å^{-3}

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
F1	0.63577 (11)	0.18180 (9)	0.27698 (7)	0.0431 (3)
C1	0.63370 (16)	−0.02848 (19)	0.79008 (11)	0.0389 (4)
C2	0.65495 (15)	−0.05532 (16)	0.68003 (10)	0.0307 (3)
C3	0.63043 (15)	0.05539 (15)	0.61573 (11)	0.0290 (3)
C4	0.66237 (14)	0.01485 (14)	0.50870 (10)	0.0258 (3)
C5	0.64110 (15)	0.11621 (15)	0.43912 (10)	0.0275 (3)
H5A	0.6076	0.2090	0.4619	0.033*
C6	0.66906 (15)	0.07966 (14)	0.33949 (10)	0.0276 (3)
C7	0.72669 (15)	−0.05884 (15)	0.29813 (10)	0.0264 (3)
C8	0.75114 (15)	−0.15922 (15)	0.36672 (10)	0.0277 (3)
H8A	0.7915	−0.2517	0.3427	0.033*
C9	0.71625 (14)	−0.12410 (14)	0.47136 (10)	0.0258 (3)
C10	0.70628 (16)	−0.18915 (16)	0.63933 (11)	0.0320 (3)
H10A	0.7198	−0.2588	0.6836	0.038*
C11	0.79437 (18)	−0.37552 (16)	0.50584 (12)	0.0397 (4)
H11A	0.8377	−0.4277	0.5560	0.048*
H11B	0.8717	−0.3910	0.4423	0.048*
C12	0.6737 (2)	−0.42777 (18)	0.49078 (15)	0.0524 (5)
H12A	0.7149	−0.5242	0.4686	0.079*
H12B	0.6316	−0.3776	0.4403	0.079*
H12C	0.5979	−0.4147	0.5539	0.079*
C13	0.82815 (18)	−0.02111 (17)	0.11838 (11)	0.0367 (4)
H13A	0.9339	−0.0663	0.1060	0.044*
H13B	0.8050	0.0741	0.1427	0.044*
C14	0.78474 (18)	−0.02599 (16)	0.02080 (11)	0.0377 (4)
H14A	0.6795	0.0219	0.0328	0.045*
H14B	0.8381	0.0201	−0.0302	0.045*
C15	0.74848 (17)	−0.24840 (16)	0.06232 (11)	0.0353 (4)
H15A	0.7813	−0.3450	0.0383	0.042*

H15B	0.6419	−0.2125	0.0739	0.042*
C16	0.78617 (19)	−0.23578 (15)	0.16120 (11)	0.0363 (4)
H16A	0.7325	−0.2810	0.2127	0.044*
H16B	0.8913	−0.2813	0.1517	0.044*
C17	0.17400 (16)	0.50636 (16)	0.20944 (11)	0.0330 (3)
C18	0.33786 (15)	0.48186 (14)	0.18335 (11)	0.0290 (3)
C19	0.43591 (16)	0.39424 (16)	0.10056 (12)	0.0350 (3)
H19A	0.4000	0.3534	0.0571	0.042*
C20	0.58733 (16)	0.36724 (16)	0.08232 (11)	0.0344 (3)
H20A	0.6523	0.3088	0.0264	0.041*
C21	0.64275 (15)	0.42664 (14)	0.14690 (11)	0.0281 (3)
C22	0.54455 (16)	0.51661 (16)	0.22799 (12)	0.0348 (3)
H22A	0.5804	0.5588	0.2707	0.042*
C23	0.39284 (16)	0.54453 (16)	0.24616 (12)	0.0356 (4)
H23A	0.3278	0.6056	0.3007	0.043*
C24	0.80895 (16)	0.38823 (15)	0.12883 (12)	0.0321 (3)
C25	0.97781 (16)	0.15488 (18)	0.32394 (12)	0.0371 (4)
C26	0.98985 (15)	0.07643 (17)	0.41574 (11)	0.0330 (3)
C27	1.04199 (16)	−0.06690 (17)	0.40371 (12)	0.0354 (4)
H27A	1.0697	−0.1117	0.3392	0.042*
C28	1.05276 (16)	−0.14308 (18)	0.48721 (12)	0.0356 (4)
H28	1.087 (2)	−0.246 (2)	0.4790 (14)	0.054 (5)*
N1	0.74725 (14)	−0.09079 (12)	0.19528 (9)	0.0306 (3)
N2	0.81977 (15)	−0.17166 (14)	−0.01670 (9)	0.0327 (3)
H2A	0.923 (2)	−0.2219 (19)	−0.0393 (13)	0.049 (5)*
H2B	0.785 (2)	−0.169 (2)	−0.0715 (15)	0.055 (5)*
N3	0.73831 (13)	−0.22629 (12)	0.54007 (9)	0.0305 (3)
O1	0.60295 (14)	0.10171 (15)	0.82150 (9)	0.0489 (3)
H1A	0.595 (3)	0.154 (3)	0.760 (2)	0.104 (9)*
O2	0.64619 (14)	−0.11994 (15)	0.84810 (9)	0.0522 (3)
O3	0.58611 (12)	0.18033 (11)	0.64870 (8)	0.0391 (3)
O4	0.08777 (13)	0.57308 (16)	0.28487 (10)	0.0622 (4)
O5	0.13363 (12)	0.44329 (13)	0.14372 (9)	0.0466 (3)
H5B	0.030 (3)	0.448 (2)	0.1690 (17)	0.087 (7)*
O6	0.88873 (12)	0.30848 (14)	0.05353 (9)	0.0533 (3)
O7	0.85565 (11)	0.43952 (11)	0.19277 (9)	0.0379 (3)
O8	1.01763 (14)	0.10184 (13)	0.23849 (9)	0.0520 (3)
O9	0.91792 (16)	0.28979 (13)	0.34610 (10)	0.0565 (4)
H9A	0.903 (3)	0.334 (2)	0.2932 (18)	0.080 (8)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.0595 (6)	0.0329 (5)	0.0350 (5)	−0.0096 (4)	−0.0165 (4)	0.0123 (4)
C1	0.0302 (8)	0.0629 (11)	0.0270 (8)	−0.0185 (8)	−0.0093 (6)	0.0033 (8)
C2	0.0241 (7)	0.0454 (9)	0.0242 (7)	−0.0122 (6)	−0.0079 (6)	0.0031 (6)
C3	0.0215 (6)	0.0374 (8)	0.0271 (7)	−0.0080 (6)	−0.0069 (5)	−0.0025 (6)
C4	0.0208 (6)	0.0323 (8)	0.0245 (7)	−0.0080 (6)	−0.0069 (5)	0.0007 (6)

C5	0.0259 (7)	0.0254 (7)	0.0295 (7)	−0.0062 (6)	−0.0071 (6)	−0.0001 (6)
C6	0.0281 (7)	0.0284 (7)	0.0284 (7)	−0.0092 (6)	−0.0113 (6)	0.0079 (6)
C7	0.0265 (7)	0.0333 (8)	0.0221 (7)	−0.0127 (6)	−0.0075 (5)	0.0013 (6)
C8	0.0280 (7)	0.0262 (7)	0.0266 (7)	−0.0058 (6)	−0.0072 (6)	−0.0001 (5)
C9	0.0218 (6)	0.0307 (7)	0.0246 (7)	−0.0077 (6)	−0.0073 (5)	0.0043 (6)
C10	0.0291 (7)	0.0420 (9)	0.0265 (7)	−0.0106 (6)	−0.0114 (6)	0.0087 (6)
C11	0.0431 (9)	0.0297 (8)	0.0338 (8)	0.0027 (7)	−0.0076 (7)	0.0049 (6)
C12	0.0648 (12)	0.0311 (9)	0.0544 (11)	−0.0102 (8)	−0.0109 (9)	−0.0027 (8)
C13	0.0433 (9)	0.0441 (9)	0.0285 (8)	−0.0239 (7)	−0.0079 (6)	0.0049 (6)
C14	0.0460 (9)	0.0390 (9)	0.0270 (8)	−0.0145 (7)	−0.0073 (7)	0.0066 (6)
C15	0.0378 (8)	0.0392 (9)	0.0290 (8)	−0.0145 (7)	−0.0066 (6)	−0.0042 (6)
C16	0.0504 (9)	0.0330 (8)	0.0267 (8)	−0.0152 (7)	−0.0103 (7)	0.0006 (6)
C17	0.0268 (7)	0.0366 (8)	0.0350 (8)	−0.0092 (6)	−0.0087 (6)	0.0019 (6)
C18	0.0255 (7)	0.0310 (7)	0.0320 (8)	−0.0101 (6)	−0.0090 (6)	0.0048 (6)
C19	0.0310 (8)	0.0404 (9)	0.0365 (8)	−0.0125 (7)	−0.0126 (6)	−0.0041 (7)
C20	0.0291 (8)	0.0369 (8)	0.0339 (8)	−0.0078 (6)	−0.0066 (6)	−0.0035 (6)
C21	0.0251 (7)	0.0276 (7)	0.0341 (8)	−0.0099 (6)	−0.0106 (6)	0.0080 (6)
C22	0.0305 (8)	0.0378 (8)	0.0388 (8)	−0.0130 (6)	−0.0116 (6)	−0.0044 (7)
C23	0.0295 (8)	0.0366 (8)	0.0371 (8)	−0.0086 (6)	−0.0053 (6)	−0.0072 (6)
C24	0.0253 (7)	0.0329 (8)	0.0392 (8)	−0.0097 (6)	−0.0108 (6)	0.0093 (6)
C25	0.0283 (7)	0.0475 (10)	0.0373 (9)	−0.0137 (7)	−0.0104 (6)	0.0057 (7)
C26	0.0224 (7)	0.0436 (9)	0.0349 (8)	−0.0123 (6)	−0.0091 (6)	0.0047 (6)
C27	0.0264 (7)	0.0459 (9)	0.0331 (8)	−0.0107 (7)	−0.0079 (6)	−0.0004 (7)
C28	0.0272 (7)	0.0412 (9)	0.0389 (9)	−0.0113 (7)	−0.0093 (6)	0.0020 (7)
N1	0.0415 (7)	0.0314 (7)	0.0225 (6)	−0.0164 (5)	−0.0090 (5)	0.0023 (5)
N2	0.0295 (7)	0.0435 (8)	0.0228 (6)	−0.0085 (6)	−0.0070 (5)	−0.0016 (5)
N3	0.0306 (6)	0.0311 (7)	0.0263 (6)	−0.0045 (5)	−0.0089 (5)	0.0046 (5)
O1	0.0497 (7)	0.0647 (8)	0.0289 (6)	−0.0152 (6)	−0.0086 (5)	−0.0086 (6)
O2	0.0622 (8)	0.0773 (9)	0.0300 (6)	−0.0324 (7)	−0.0226 (6)	0.0168 (6)
O3	0.0424 (6)	0.0386 (6)	0.0321 (6)	−0.0085 (5)	−0.0082 (5)	−0.0078 (5)
O4	0.0288 (6)	0.0954 (11)	0.0531 (8)	−0.0150 (6)	−0.0005 (6)	−0.0288 (7)
O5	0.0263 (6)	0.0604 (8)	0.0531 (7)	−0.0146 (5)	−0.0095 (5)	−0.0155 (6)
O6	0.0265 (6)	0.0708 (9)	0.0519 (8)	−0.0039 (6)	−0.0068 (5)	−0.0122 (6)
O7	0.0282 (5)	0.0403 (6)	0.0514 (7)	−0.0136 (5)	−0.0183 (5)	0.0077 (5)
O8	0.0606 (8)	0.0560 (8)	0.0367 (7)	−0.0143 (6)	−0.0146 (6)	0.0056 (6)
O9	0.0783 (10)	0.0429 (8)	0.0424 (8)	−0.0081 (7)	−0.0208 (7)	0.0090 (6)

Geometric parameters (Å, º)

F1—C6	1.3576 (16)	C15—H15A	0.9700
C1—O2	1.217 (2)	C15—H15B	0.9700
C1—O1	1.321 (2)	C16—N1	1.4604 (18)
C1—C2	1.479 (2)	C16—H16A	0.9700
C2—C10	1.370 (2)	C16—H16B	0.9700
C2—C3	1.426 (2)	C17—O4	1.2012 (18)
C3—O3	1.2607 (18)	C17—O5	1.3225 (18)
C3—C4	1.4490 (19)	C17—C18	1.5025 (19)
C4—C9	1.402 (2)	C18—C19	1.387 (2)

C4—C5	1.404 (2)	C18—C23	1.387 (2)
C5—C6	1.349 (2)	C19—C20	1.388 (2)
C5—H5A	0.9300	C19—H19A	0.9300
C6—C7	1.413 (2)	C20—C21	1.389 (2)
C7—C8	1.391 (2)	C20—H20A	0.9300
C7—N1	1.3936 (17)	C21—C22	1.383 (2)
C8—C9	1.4023 (19)	C21—C24	1.5162 (19)
C8—H8A	0.9300	C22—C23	1.389 (2)
C9—N3	1.4020 (18)	C22—H22A	0.9300
C10—N3	1.3397 (18)	C23—H23A	0.9300
C10—H10A	0.9300	C24—O6	1.2388 (18)
C11—N3	1.4865 (19)	C24—O7	1.2766 (18)
C11—C12	1.513 (3)	C25—O8	1.2048 (19)
C11—H11A	0.9700	C25—O9	1.324 (2)
C11—H11B	0.9700	C25—C26	1.493 (2)
C12—H12A	0.9600	C26—C27	1.390 (2)
C12—H12B	0.9600	C26—C28^{i}	1.396 (2)
C12—H12C	0.9600	C27—C28	1.383 (2)
C13—N1	1.4663 (18)	C27—H27A	0.9300
C13—C14	1.511 (2)	C28—C26^{i}	1.396 (2)
C13—H13A	0.9700	C28—H28	1.00 (2)
C13—H13B	0.9700	N2—H2A	0.95 (2)
C14—N2	1.486 (2)	N2—H2B	0.90 (2)
C14—H14A	0.9700	O1—H1A	1.01 (3)
C14—H14B	0.9700	O5—H5B	0.97 (3)
C15—N2	1.4854 (19)	O9—H9A	0.87 (2)
C15—C16	1.511 (2)		

O2—C1—O1	121.77 (15)	H15A—C15—H15B	108.0
O2—C1—C2	122.66 (16)	N1—C16—C15	110.42 (12)
O1—C1—C2	115.55 (15)	N1—C16—H16A	109.6
C10—C2—C3	120.06 (13)	C15—C16—H16A	109.6
C10—C2—C1	118.91 (14)	N1—C16—H16B	109.6
C3—C2—C1	120.95 (14)	C15—C16—H16B	109.6
O3—C3—C2	122.78 (13)	H16A—C16—H16B	108.1
O3—C3—C4	121.79 (13)	O4—C17—O5	122.82 (14)
C2—C3—C4	115.42 (13)	O4—C17—C18	123.26 (14)
C9—C4—C5	118.25 (12)	O5—C17—C18	113.87 (13)
C9—C4—C3	121.91 (13)	C19—C18—C23	119.20 (13)
C5—C4—C3	119.84 (13)	C19—C18—C17	121.79 (13)
C6—C5—C4	120.33 (13)	C23—C18—C17	118.96 (13)
C6—C5—H5A	119.8	C18—C19—C20	120.29 (13)
C4—C5—H5A	119.8	C18—C19—H19A	119.9
C5—C6—F1	117.76 (12)	C20—C19—H19A	119.9
C5—C6—C7	123.34 (13)	C19—C20—C21	120.57 (14)
F1—C6—C7	118.88 (12)	C19—C20—H20A	119.7
C8—C7—N1	122.74 (13)	C21—C20—H20A	119.7
C8—C7—C6	116.28 (12)	C22—C21—C20	119.00 (12)

N1—C7—C6	120.84 (12)	C22—C21—C24	121.62 (13)
C7—C8—C9	121.46 (13)	C20—C21—C24	119.35 (13)
C7—C8—H8A	119.3	C21—C22—C23	120.57 (13)
C9—C8—H8A	119.3	C21—C22—H22A	119.7
N3—C9—C4	118.79 (12)	C23—C22—H22A	119.7
N3—C9—C8	120.97 (12)	C18—C23—C22	120.33 (14)
C4—C9—C8	120.22 (13)	C18—C23—H23A	119.8
N3—C10—C2	124.37 (14)	C22—C23—H23A	119.8
N3—C10—H10A	117.8	O6—C24—O7	125.16 (13)
C2—C10—H10A	117.8	O6—C24—C21	117.30 (13)
N3—C11—C12	112.40 (13)	O7—C24—C21	117.54 (13)
N3—C11—H11A	109.1	O8—C25—O9	123.17 (15)
C12—C11—H11A	109.1	O8—C25—C26	123.95 (15)
N3—C11—H11B	109.1	O9—C25—C26	112.88 (14)
C12—C11—H11B	109.1	C27—C26—C28^{i}	119.59 (15)
H11A—C11—H11B	107.9	C27—C26—C25	118.64 (14)
C11—C12—H12A	109.5	C28^{i}—C26—C25	121.76 (15)
C11—C12—H12B	109.5	C28—C27—C26	120.27 (15)
H12A—C12—H12B	109.5	C28—C27—H27A	119.9
C11—C12—H12C	109.5	C26—C27—H27A	119.9
H12A—C12—H12C	109.5	C27—C28—C26^{i}	120.14 (15)
H12B—C12—H12C	109.5	C27—C28—H28	120.3 (11)
N1—C13—C14	109.50 (12)	C26^{i}—C28—H28	119.5 (11)
N1—C13—H13A	109.8	C7—N1—C16	117.61 (12)
C14—C13—H13A	109.8	C7—N1—C13	119.55 (11)
N1—C13—H13B	109.8	C16—N1—C13	110.22 (11)
C14—C13—H13B	109.8	C15—N2—C14	111.24 (11)
H13A—C13—H13B	108.2	C15—N2—H2A	108.2 (11)
N2—C14—C13	109.94 (13)	C14—N2—H2A	114.5 (11)
N2—C14—H14A	109.7	C15—N2—H2B	109.8 (13)
C13—C14—H14A	109.7	C14—N2—H2B	106.8 (12)
N2—C14—H14B	109.7	H2A—N2—H2B	106.1 (15)
C13—C14—H14B	109.7	C10—N3—C9	119.45 (12)
H14A—C14—H14B	108.2	C10—N3—C11	118.90 (12)
N2—C15—C16	111.36 (12)	C9—N3—C11	121.65 (11)
N2—C15—H15A	109.4	C1—O1—H1A	105.0 (15)
C16—C15—H15A	109.4	C17—O5—H5B	111.4 (13)
N2—C15—H15B	109.4	C25—O9—H9A	112.3 (15)
C16—C15—H15B	109.4		

O2—C1—C2—C10	−7.3 (2)	C18—C19—C20—C21	0.4 (2)
O1—C1—C2—C10	171.42 (13)	C19—C20—C21—C22	−2.0 (2)
O2—C1—C2—C3	176.10 (13)	C19—C20—C21—C24	176.19 (13)
O1—C1—C2—C3	−5.2 (2)	C20—C21—C22—C23	1.6 (2)
C10—C2—C3—O3	−178.58 (13)	C24—C21—C22—C23	−176.53 (14)
C1—C2—C3—O3	−2.0 (2)	C19—C18—C23—C22	−1.9 (2)
C10—C2—C3—C4	0.41 (19)	C17—C18—C23—C22	175.66 (14)
C1—C2—C3—C4	176.99 (12)	C21—C22—C23—C18	0.3 (2)

O3—C3—C4—C9	177.89 (12)	C22—C21—C24—O6	−178.99 (14)
C2—C3—C4—C9	−1.10 (19)	C20—C21—C24—O6	2.9 (2)
O3—C3—C4—C5	−1.3 (2)	C22—C21—C24—O7	1.6 (2)
C2—C3—C4—C5	179.68 (12)	C20—C21—C24—O7	−176.52 (13)
C9—C4—C5—C6	1.4 (2)	O8—C25—C26—C27	−3.9 (2)
C3—C4—C5—C6	−179.33 (12)	O9—C25—C26—C27	175.87 (13)
C4—C5—C6—F1	175.05 (11)	O8—C25—C26—C28^{i}	177.53 (15)
C4—C5—C6—C7	−3.0 (2)	O9—C25—C26—C28^{i}	−2.7 (2)
C5—C6—C7—C8	1.4 (2)	C28^{i}—C26—C27—C28	−0.5 (2)
F1—C6—C7—C8	−176.64 (12)	C25—C26—C27—C28	−179.12 (13)
C5—C6—C7—N1	177.13 (13)	C26—C27—C28—C26^{i}	0.5 (2)
F1—C6—C7—N1	−0.92 (19)	C8—C7—N1—C16	4.3 (2)
N1—C7—C8—C9	−173.88 (12)	C6—C7—N1—C16	−171.11 (13)
C6—C7—C8—C9	1.8 (2)	C8—C7—N1—C13	−133.83 (14)
C5—C4—C9—N3	179.84 (12)	C6—C7—N1—C13	50.73 (19)
C3—C4—C9—N3	0.61 (19)	C15—C16—N1—C7	158.69 (12)
C5—C4—C9—C8	1.64 (19)	C15—C16—N1—C13	−59.52 (16)
C3—C4—C9—C8	−177.59 (12)	C14—C13—N1—C7	−157.19 (13)
C7—C8—C9—N3	178.57 (12)	C14—C13—N1—C16	61.86 (16)
C7—C8—C9—C4	−3.3 (2)	C16—C15—N2—C14	−53.27 (17)
C3—C2—C10—N3	0.8 (2)	C13—C14—N2—C15	55.27 (16)
C1—C2—C10—N3	−175.81 (13)	C2—C10—N3—C9	−1.4 (2)
N1—C13—C14—N2	−59.34 (17)	C2—C10—N3—C11	179.53 (13)
N2—C15—C16—N1	55.06 (16)	C4—C9—N3—C10	0.64 (19)
O4—C17—C18—C19	174.08 (16)	C8—C9—N3—C10	178.82 (12)
O5—C17—C18—C19	−3.4 (2)	C4—C9—N3—C11	179.68 (12)
O4—C17—C18—C23	−3.4 (2)	C8—C9—N3—C11	−2.1 (2)
O5—C17—C18—C23	179.09 (14)	C12—C11—N3—C10	98.46 (16)
C23—C18—C19—C20	1.5 (2)	C12—C11—N3—C9	−80.58 (17)
C17—C18—C19—C20	−175.97 (14)		

Symmetry code: (i) −*x*+2, −*y*, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C15—H15*B*···O1^{ii}	0.97	2.38	3.274 (2)	153
O5—H5*B*···O7^{iii}	0.97 (3)	1.70 (3)	2.6648 (15)	169 (2)

Symmetry codes: (ii) −*x*+1, −*y*, −*z*+1; (iii) *x*−1, *y*, *z*.