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(*S*)-3-[(*S*)-5-(4-Fluorophenyl)-5-hydroxypentanoyl]-4-phenyloxazolidin-2-one 0.67-hydrate

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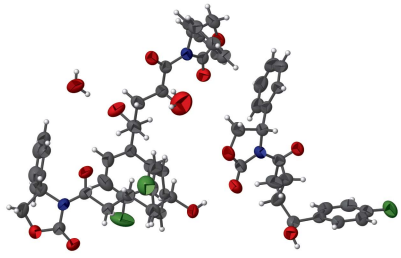
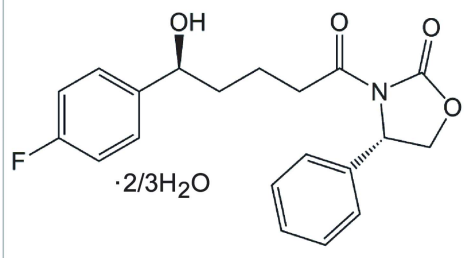
Structural data: full structural data are available from iucrdata.iucr.org

The asymmetric unit of the title compound, $3C_{20}H_{20}FNO_4\cdot 2H_2O$, consists of three (*S*)-3-[(*S*)-5-(4-fluorophenyl)-5-hydroxypentanoyl]-4-phenyloxazolidin-2-one molecules and two solvent water molecules. The carbonyl groups of the oxazolidin-2-one and hydroxypentanoyl units are oriented *anti* to one another in each molecule. In the crystal, O—H···O, C—H···O and C—H···F hydrogen bonds link the molecules into a three-dimensional supramolecular architecture with the organic molecules linked by water molecules and stacked along the *b*-axis direction.

3D view



Chemical scheme



Structure description

Phenyloxazolidinones have received much attention due to their applications in asymmetric synthesis (Evans *et al.*, 1982). We report here the synthesis and crystal structure of the title oxazolidinone compound (Fig. 1). This is an intermediate in the synthesis of the cholesterol-lowering agent Ezetimibe, which is a novel and potent drug that selectively inhibits cholesterol absorption across the intestinal wall (van Heek *et al.*, 2000). Clinical studies show that Ezetimibe can significantly decrease plasma LDL cholesterol levels and increase plasma HDL levels with an excellent tolerability profile (Wu *et al.* 1999).

The asymmetric unit of the title compound consists of three (*S*)-3-[(*S*)-5-(4-fluorophenyl)-5-hydroxypentanoyl]-4-phenyloxazolidin-2-one molecules (*A* incorporating F1, *B* with F2 and *C* with F3), together with two solvent water molecules. The organic molecules differ mainly in the relative inclination of the two aromatic rings. Each unique molecule has two stereogenic centres, at C7 and C14 for *A*, C27 and C34 for *B* and C47 and C54 for *C*. Each of these adopt an *S* configuration. Bond lengths and angles are

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

D—H···A	D—H	H···A	D···A	D—H···A
O10—H10···O8	0.82	2.02	2.837 (3)	173
O6—H6A···O10^{i}	0.82	1.99	2.797 (3)	167
O1—H1···O14^{ii}	0.82	1.85	2.669 (4)	174
C22—H22···O6^{iii}	0.93	2.65	3.522 (3)	157
C33—H33A···O7^{iv}	0.97	2.57	3.337 (4)	137
C30—H30A···O6	0.97	2.58	3.149 (3)	118
C30—H30B···O8	0.97	2.31	2.860 (4)	115
O14—H14A···O1	0.85	1.88	2.728 (3)	173
O14—H14B···O13^{ii}	0.85	2.01	2.825 (3)	161
C13—H13B···F2^{iv}	0.97	2.51	3.347 (3)	145
C42—H42···O3^{v}	0.93	2.37	3.232 (3)	153
O5—H5B···O3^{v}	0.85	2.09	2.928 (5)	171

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

within normal ranges (Yang *et al.*, 2006). In molecule *A*, the benzene ring is inclined at an angle of 65.36 (12)° to the fluorobenzene ring. The corresponding angles for molecules *B* and *C* are 63.55 (13) and 23.49 (14)°, respectively.

In the crystal, O—H···O, C—H···O and C—H···F hydrogen bonds are found involving both the organic and solvent water molecules (Table 1, Fig. 1). These produce a three-dimensional network with the organic molecules linked by water molecules and stacked along the *b*-axis direction (Fig. 2).

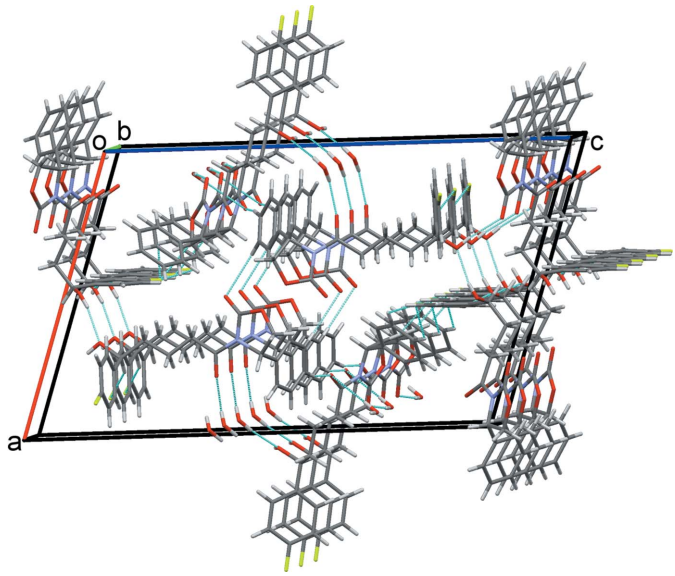


Figure 2
The crystal packing of the title compound, viewed along the *b*-axis direction.

Synthesis and crystallization

The title compound was prepared according to a literature procedure (Fu *et al.* 2003; Bertrand *et al.* 2007). Single crystals suitable for X-ray diffraction were obtained by slow

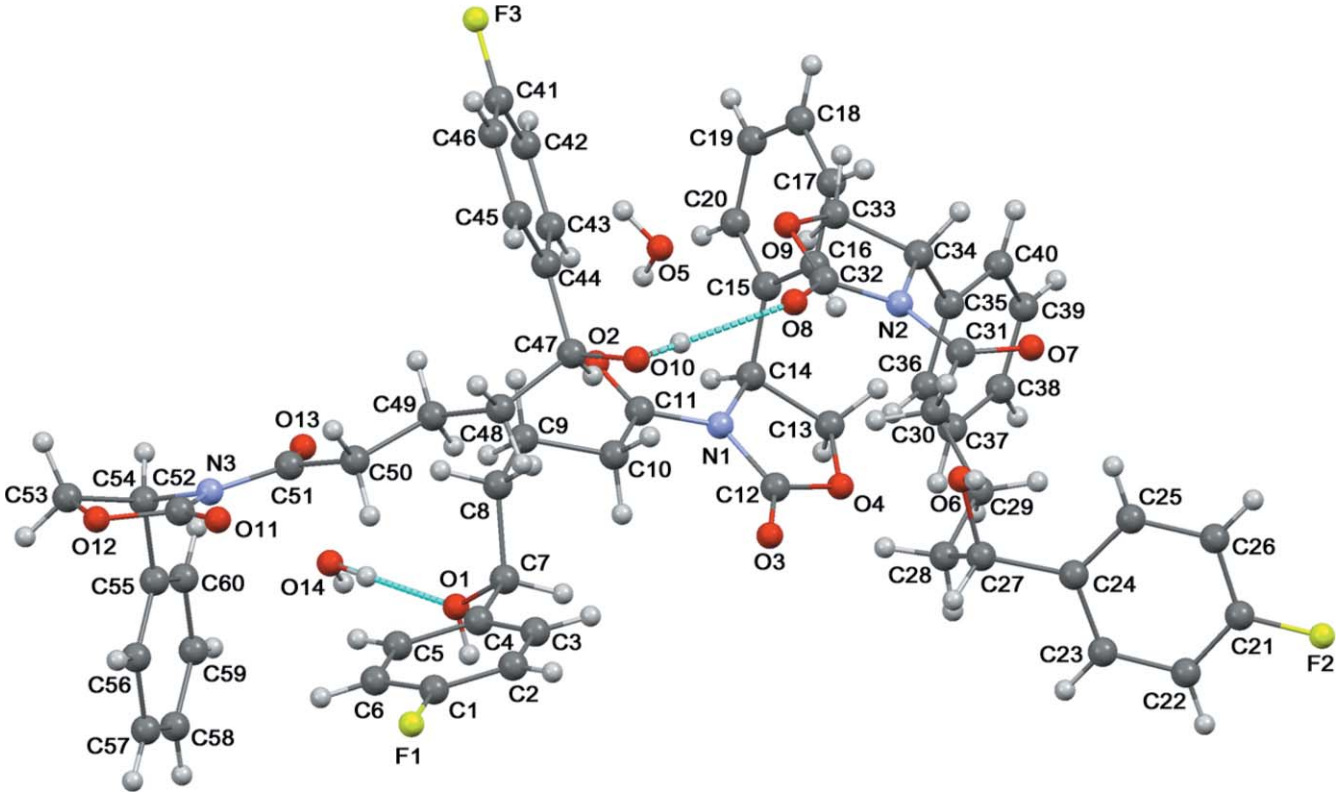


Figure 1
A view of the title compound showing the three-dimensional supramolecular architecture linked by pairs of O—H···O hydrogen bonds. Displacement ellipsoids are drawn at the 50% probability level.

Table 2
Experimental details.

Crystal data	
Chemical formula	$3C_{20}H_{20}FNO_4{\cdot}2H_2O$
M_r	1108.14
Crystal system, space group	Monoclinic, $P2_1$
Temperature (K)	293
a, b, c (Å)	15.4975 (10), 7.8125 (5), 24.1875 (18)
β (°)	107.517 (8)
V (Å^3)	2792.7 (3)
Z	2
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.84
Crystal size (mm)	0.42 × 0.26 × 0.22
Data collection	
Diffractometer	Rigaku Xcalibur Eos Gemini
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku, 2015)
T_{min}, T_{max}	0.691, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	29311, 9875, 8563
R_{int}	0.033
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.042, 0.119, 1.06
No. of reflections	9875
No. of parameters	727
No. of restraints	1
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.20, −0.31
Absolute structure	Flack (1983)
Absolute structure parameter	0.07 (13)

Computer programs: *CrysAlis PRO* (Rigaku, 2015), *SHELXS97* and *SHELXL97* (Sheldrick, 2008) and *OLEX2* (Dolomanov *et al.*, 2009).

evaporation of an ethanol solution at room temperature over a period of 30 days.

Refinement

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

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References

Bertrand, B., Durassier, S., Frein, S. & Burgos, A. (2007). *Tetrahedron Lett.* **48**, 2123–2125.

Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.

Evans, D. A., Nelson, J. V. & Taber, T. (1982). *Top. Stereochem.* **13**, 1–115.

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.

Fu, X., McAllister, T. L., Thiruvengadam, T. K., Tann, C. & Su, D. (2003). *Tetrahedron Lett.* **44**, 801–804.

Heek, M. van, Farley, C., Compton, D. S., Hoos, L., Alton, K. B., Sybertz, E. J. & Davis, H. R. (2000). *Br. J. Pharmacol.* **129**, 1748–1754.

Rigaku (2015). *CrysAlis PRO*. Rigaku Corporation, Tokyo, Japan.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Wu, G., Wong, Y., Chen, X. & Ding, Z. (1999). *J. Org. Chem.* **64**, 3714–3718.

Yang, R., Nie, L., Zhang, M.-S., Wang, J. & Lin, J. (2006). *Acta Cryst.* E**62**, o3618–o3619.

full crystallographic data

IUCrData (2017). **2**, x170213 [https://doi.org/10.1107/S2414314617002139]

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Crystal data

$3C_{20}H_{20}FNO_4 \cdot 2H_2O$
M_r = 1108.14
Monoclinic, $P2_1$
a = 15.4975 (10) Å
b = 7.8125 (5) Å
c = 24.1875 (18) Å
β = 107.517 (8)°
V = 2792.7 (3) Å^3
Z = 2
$F(000)$ = 1168
D_x = 1.318 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 10080 reflections
θ = 5.5–72.3°
μ = 0.84 mm^{-1}
T = 293 K
Needle, colourless
0.42 × 0.26 × 0.22 mm

Data collection

Rigaku Xcalibur Eos Gemini diffractometer
Radiation source: fine-focus sealed X-ray tube, Enhance (Cu) X-ray Source
Graphite monochromator
Detector resolution: 16.0355 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku, 2015)
T_{min} = 0.691, T_{max} = 1.000
29311 measured reflections
9875 independent reflections
8563 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 66.6°, θ_{min} = 5.5°
h = −18→17
k = −9→9
l = −27→28

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.119
S = 1.06
9875 reflections
727 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0733P)^2 + 0.0055P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}
Absolute structure: Flack (1983), **??? Friedel pairs**
Absolute structure parameter: 0.07 (13)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\text{sigma}(F^2)$ 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}
O4	0.22186 (13)	0.5917 (2)	0.27097 (8)	0.0651 (4)
O9	−0.10736 (13)	−0.0727 (2)	0.08775 (9)	0.0701 (5)
O13	−0.22269 (11)	−0.1772 (3)	0.41117 (8)	0.0670 (5)
O10	−0.34846 (12)	−0.0634 (3)	0.13565 (8)	0.0663 (5)
H10	−0.3154	−0.0384	0.1160	0.099*
N1	0.19278 (13)	0.3495 (2)	0.30979 (8)	0.0507 (4)
O2	0.20106 (12)	0.1166 (2)	0.36458 (8)	0.0634 (4)
N2	−0.13039 (13)	0.1743 (2)	0.04055 (8)	0.0511 (4)
N3	−0.34780 (13)	−0.2339 (3)	0.43586 (8)	0.0528 (5)
O6	−0.47429 (13)	0.4274 (2)	−0.06100 (9)	0.0674 (5)
H6A	−0.5229	0.4237	−0.0867	0.101*
O1	−0.05047 (15)	0.3350 (3)	0.44097 (10)	0.0830 (6)
H1	−0.0519	0.4361	0.4497	0.124*
O7	−0.13564 (12)	0.3822 (2)	−0.02380 (8)	0.0643 (4)
C44	−0.25569 (15)	−0.2934 (3)	0.18945 (9)	0.0503 (5)
C51	−0.30383 (15)	−0.1882 (3)	0.39561 (10)	0.0506 (5)
F2	−0.44779 (13)	1.0810 (2)	−0.20943 (9)	0.0877 (5)
O3	0.08350 (12)	0.5607 (2)	0.27908 (9)	0.0698 (5)
C24	−0.45010 (14)	0.7244 (3)	−0.08478 (10)	0.0494 (5)
C11	0.15345 (16)	0.2291 (3)	0.33788 (10)	0.0504 (5)
C55	−0.27078 (15)	−0.0930 (3)	0.52883 (9)	0.0497 (5)
C4	−0.20339 (16)	0.3232 (3)	0.37583 (10)	0.0527 (5)
C10	0.05462 (17)	0.2476 (3)	0.33214 (11)	0.0570 (5)
H10A	0.0198	0.2355	0.2916	0.068*
H10B	0.0438	0.3615	0.3445	0.068*
O8	−0.24876 (14)	0.0229 (3)	0.05862 (9)	0.0759 (5)
C50	−0.35969 (15)	−0.1553 (3)	0.33420 (9)	0.0506 (5)
H50A	−0.3941	−0.0506	0.3325	0.061*
H50B	−0.4022	−0.2485	0.3209	0.061*
C14	0.28594 (16)	0.3244 (3)	0.30862 (10)	0.0532 (5)
H14	0.3257	0.3060	0.3482	0.064*
C19	0.37247 (17)	−0.0785 (3)	0.25634 (12)	0.0610 (6)
H19	0.4177	−0.1594	0.2707	0.073*
C21	−0.44538 (17)	0.9637 (3)	−0.16697 (13)	0.0594 (6)
C26	−0.45009 (19)	0.7944 (3)	−0.18126 (12)	0.0611 (6)

H26	−0.4518	0.7598	−0.2184	0.073*
C35	0.02871 (15)	0.2855 (3)	0.07010 (9)	0.0487 (5)
C12	0.15785 (17)	0.5051 (3)	0.28622 (10)	0.0539 (5)
C15	0.29514 (15)	0.1764 (3)	0.27074 (10)	0.0493 (5)
F3	−0.14961 (16)	−0.7748 (3)	0.18369 (10)	0.0975 (6)
C48	−0.35387 (16)	−0.1066 (3)	0.23169 (10)	0.0557 (5)
H48A	−0.3815	0.0059	0.2283	0.067*
H48B	−0.4020	−0.1906	0.2195	0.067*
C47	−0.29457 (15)	−0.1167 (3)	0.19187 (10)	0.0541 (5)
H47	−0.2444	−0.0358	0.2058	0.065*
C31	−0.17600 (16)	0.3071 (3)	0.00470 (11)	0.0558 (5)
C27	−0.45808 (16)	0.5962 (3)	−0.03945 (11)	0.0554 (5)
H27	−0.5095	0.6306	−0.0263	0.067*
C32	−0.16962 (17)	0.0417 (3)	0.06248 (11)	0.0584 (6)
C22	−0.44054 (17)	1.0194 (3)	−0.11293 (13)	0.0643 (7)
H22	−0.4358	1.1354	−0.1039	0.077*
C25	−0.45224 (16)	0.6754 (3)	−0.13935 (10)	0.0530 (5)
H25	−0.4552	0.5595	−0.1486	0.064*
O11	−0.50008 (14)	−0.2329 (4)	0.38475 (9)	0.0960 (9)
C41	−0.1848 (2)	−0.6152 (4)	0.18596 (12)	0.0644 (6)
C16	0.23945 (17)	0.1628 (3)	0.21409 (11)	0.0575 (6)
H16	0.1941	0.2434	0.1995	0.069*
C43	−0.16286 (16)	−0.3218 (4)	0.20818 (11)	0.0603 (6)
H43	−0.1238	−0.2304	0.2219	0.072*
C60	−0.18220 (16)	−0.0352 (3)	0.54343 (10)	0.0544 (5)
H60	−0.1387	−0.1003	0.5337	0.065*
C23	−0.44282 (17)	0.8985 (3)	−0.07185 (12)	0.0603 (6)
H23	−0.4394	0.9341	−0.0346	0.072*
C34	−0.03547 (16)	0.1386 (3)	0.04692 (10)	0.0497 (5)
H34	−0.0294	0.1012	0.0096	0.060*
C20	0.36096 (16)	0.0527 (3)	0.29121 (10)	0.0552 (5)
H20	0.3981	0.0586	0.3294	0.066*
C17	0.25083 (18)	0.0304 (4)	0.17918 (11)	0.0636 (6)
H17	0.2133	0.0228	0.1412	0.076*
C49	−0.30037 (16)	−0.1390 (4)	0.29458 (10)	0.0559 (5)
H49A	−0.2580	−0.0457	0.3082	0.067*
H49B	−0.2656	−0.2434	0.2969	0.067*
C59	−0.1581 (2)	0.1179 (4)	0.57229 (12)	0.0675 (7)
H59	−0.0984	0.1549	0.5820	0.081*
C40	0.10942 (16)	0.2918 (3)	0.05677 (11)	0.0566 (6)
H40	0.1215	0.2106	0.0320	0.068*
C54	−0.29644 (17)	−0.2617 (3)	0.49713 (10)	0.0551 (6)
H54	−0.2420	−0.3294	0.5000	0.066*
C18	0.31706 (18)	−0.0901 (3)	0.20003 (13)	0.0630 (6)
H18	0.3245	−0.1790	0.1763	0.076*
C8	−0.07815 (18)	0.1388 (3)	0.36260 (14)	0.0646 (6)
H8A	−0.1133	0.1226	0.3224	0.078*
H8B	−0.0955	0.0494	0.3850	0.078*

C45	−0.31084 (16)	−0.4325 (4)	0.16908 (10)	0.0576 (6)
H45	−0.3732	−0.4166	0.1563	0.069*
C57	−0.3098 (2)	0.1605 (4)	0.57261 (13)	0.0714 (7)
H57	−0.3530	0.2260	0.5826	0.086*
F1	−0.47752 (14)	0.3445 (4)	0.35090 (15)	0.1285 (9)
C46	−0.27641 (19)	−0.5951 (4)	0.16702 (11)	0.0640 (6)
H46	−0.3145	−0.6876	0.1532	0.077*
C52	−0.4393 (2)	−0.2631 (4)	0.42661 (12)	0.0703 (7)
C9	0.02166 (19)	0.1162 (3)	0.36768 (13)	0.0649 (6)
H9A	0.0573	0.1264	0.4081	0.078*
H9B	0.0310	0.0022	0.3547	0.078*
O12	−0.45079 (15)	−0.3331 (4)	0.47472 (9)	0.0893 (7)
C28	−0.37471 (18)	0.5924 (4)	0.01321 (12)	0.0681 (7)
H28A	−0.3861	0.5140	0.0413	0.082*
H28B	−0.3666	0.7054	0.0306	0.082*
C37	0.0741 (2)	0.5354 (4)	0.12905 (12)	0.0771 (9)
H37	0.0616	0.6187	0.1530	0.092*
C6	−0.3316 (3)	0.2844 (5)	0.41097 (18)	0.0855 (9)
H6	−0.3555	0.2536	0.4405	0.103*
C39	0.17225 (19)	0.4186 (4)	0.08024 (15)	0.0758 (8)
H39	0.2268	0.4214	0.0717	0.091*
C1	−0.3870 (2)	0.3373 (4)	0.35868 (17)	0.0790 (9)
C58	−0.2215 (2)	0.2165 (4)	0.58683 (12)	0.0732 (8)
H58	−0.2050	0.3200	0.6061	0.088*
C5	−0.2391 (2)	0.2772 (4)	0.41952 (13)	0.0725 (7)
H5	−0.2007	0.2410	0.4551	0.087*
C3	−0.26199 (18)	0.3771 (4)	0.32441 (11)	0.0628 (6)
H3	−0.2392	0.4105	0.2946	0.075*
C29	−0.28564 (17)	0.5395 (4)	0.00236 (13)	0.0683 (7)
H29A	−0.2863	0.5793	−0.0357	0.082*
H29B	−0.2358	0.5958	0.0307	0.082*
C33	−0.01930 (19)	−0.0126 (4)	0.08834 (14)	0.0678 (7)
H33A	0.0137	−0.1023	0.0756	0.081*
H33B	0.0154	0.0226	0.1271	0.081*
C7	−0.10324 (17)	0.3109 (3)	0.38279 (12)	0.0612 (6)
H7	−0.0876	0.4008	0.3592	0.073*
C30	−0.26961 (19)	0.3539 (4)	0.00576 (18)	0.0821 (9)
H30A	−0.3136	0.2989	−0.0266	0.098*
H30B	−0.2786	0.3107	0.0412	0.098*
C36	0.0118 (2)	0.4079 (3)	0.10679 (11)	0.0643 (6)
H36	−0.0418	0.4042	0.1165	0.077*
O14	0.05185 (19)	0.1703 (4)	0.53805 (15)	0.1212 (12)
H14A	0.0164	0.2172	0.5081	0.182*
H14B	0.0957	0.2374	0.5534	0.182*
C53	−0.3653 (2)	−0.3691 (4)	0.51606 (12)	0.0766 (9)
H53A	−0.3511	−0.4900	0.5159	0.092*
H53B	−0.3655	−0.3372	0.5548	0.092*
C38	0.1539 (2)	0.5403 (4)	0.11621 (14)	0.0808 (10)

H38	0.1959	0.6259	0.1318	0.097*
C13	0.30596 (19)	0.5005 (3)	0.28677 (14)	0.0649 (6)
H13A	0.3511	0.5610	0.3170	0.078*
H13B	0.3281	0.4877	0.2535	0.078*
C56	−0.33461 (18)	0.0068 (3)	0.54337 (12)	0.0615 (6)
H56	−0.3945	−0.0295	0.5335	0.074*
C2	−0.3539 (2)	0.3830 (4)	0.31565 (15)	0.0778 (8)
H2	−0.3928	0.4184	0.2802	0.093*
C42	−0.12762 (18)	−0.4843 (4)	0.20677 (13)	0.0700 (7)
H42	−0.0655	−0.5027	0.2200	0.084*
O5	0.0698 (3)	−0.0859 (5)	0.23695 (19)	0.1632 (16)
H5A	0.1042	−0.0361	0.2697	0.245*
H5B	0.0698	−0.1913	0.2456	0.245*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O4	0.0691 (11)	0.0534 (9)	0.0741 (11)	−0.0094 (8)	0.0237 (9)	0.0123 (8)
O9	0.0742 (12)	0.0604 (10)	0.0779 (12)	−0.0054 (9)	0.0261 (9)	0.0212 (9)
O13	0.0472 (9)	0.0944 (13)	0.0528 (9)	0.0038 (9)	0.0051 (7)	−0.0029 (9)
O10	0.0555 (9)	0.0905 (13)	0.0534 (9)	0.0090 (9)	0.0172 (7)	0.0222 (9)
N1	0.0535 (10)	0.0470 (9)	0.0535 (10)	−0.0015 (8)	0.0189 (8)	0.0038 (8)
O2	0.0631 (10)	0.0549 (9)	0.0730 (11)	0.0025 (8)	0.0218 (8)	0.0146 (8)
N2	0.0509 (10)	0.0508 (10)	0.0558 (11)	−0.0036 (8)	0.0225 (8)	0.0028 (8)
N3	0.0497 (10)	0.0594 (11)	0.0432 (10)	−0.0019 (8)	0.0048 (8)	0.0031 (8)
O6	0.0623 (11)	0.0611 (10)	0.0728 (12)	−0.0002 (8)	0.0114 (8)	0.0094 (9)
O1	0.0727 (12)	0.0783 (13)	0.0756 (13)	0.0090 (10)	−0.0114 (10)	−0.0061 (10)
O7	0.0593 (10)	0.0644 (10)	0.0721 (11)	0.0057 (8)	0.0241 (8)	0.0202 (9)
C44	0.0429 (11)	0.0715 (14)	0.0369 (10)	−0.0055 (10)	0.0127 (8)	0.0036 (10)
C51	0.0512 (12)	0.0498 (11)	0.0449 (11)	0.0017 (9)	0.0054 (9)	−0.0059 (9)
F2	0.0934 (12)	0.0706 (10)	0.1084 (14)	−0.0012 (9)	0.0446 (10)	0.0278 (10)
O3	0.0584 (11)	0.0632 (10)	0.0812 (12)	0.0030 (8)	0.0107 (8)	0.0207 (9)
C24	0.0372 (10)	0.0566 (12)	0.0542 (12)	0.0056 (8)	0.0136 (8)	−0.0053 (10)
C11	0.0556 (12)	0.0443 (11)	0.0516 (12)	−0.0027 (9)	0.0166 (9)	0.0007 (9)
C55	0.0538 (12)	0.0508 (11)	0.0390 (10)	0.0055 (9)	0.0055 (8)	0.0079 (9)
C4	0.0586 (13)	0.0465 (11)	0.0514 (12)	−0.0046 (9)	0.0140 (10)	0.0041 (9)
C10	0.0570 (13)	0.0541 (12)	0.0589 (13)	−0.0052 (10)	0.0160 (10)	0.0040 (10)
O8	0.0686 (12)	0.0860 (13)	0.0778 (13)	−0.0171 (10)	0.0292 (9)	0.0154 (11)
C50	0.0486 (11)	0.0548 (12)	0.0440 (11)	0.0019 (9)	0.0070 (8)	−0.0024 (9)
C14	0.0500 (11)	0.0569 (13)	0.0514 (12)	−0.0048 (10)	0.0135 (9)	−0.0001 (10)
C19	0.0557 (13)	0.0449 (11)	0.0757 (16)	0.0053 (10)	0.0094 (11)	0.0066 (11)
C21	0.0471 (12)	0.0567 (13)	0.0766 (17)	−0.0011 (10)	0.0219 (11)	0.0076 (12)
C26	0.0650 (15)	0.0640 (14)	0.0588 (14)	−0.0010 (12)	0.0257 (11)	0.0002 (11)
C35	0.0519 (12)	0.0457 (11)	0.0454 (11)	0.0007 (9)	0.0100 (9)	0.0048 (8)
C12	0.0582 (14)	0.0515 (12)	0.0481 (12)	−0.0054 (10)	0.0104 (9)	0.0045 (9)
C15	0.0464 (11)	0.0486 (11)	0.0518 (12)	−0.0036 (9)	0.0133 (9)	0.0054 (9)
F3	0.1119 (15)	0.0768 (11)	0.1071 (15)	0.0185 (11)	0.0380 (12)	0.0004 (11)
C48	0.0505 (12)	0.0663 (14)	0.0486 (12)	0.0008 (10)	0.0126 (9)	0.0026 (10)

C47	0.0478 (11)	0.0629 (13)	0.0497 (12)	−0.0062 (10)	0.0118 (9)	0.0072 (10)
C31	0.0522 (12)	0.0502 (12)	0.0654 (14)	−0.0027 (10)	0.0181 (11)	0.0039 (11)
C27	0.0477 (12)	0.0649 (14)	0.0564 (13)	0.0086 (10)	0.0198 (10)	0.0018 (11)
C32	0.0619 (14)	0.0628 (14)	0.0545 (13)	−0.0113 (11)	0.0235 (10)	0.0032 (11)
C22	0.0509 (13)	0.0473 (12)	0.0906 (19)	0.0009 (10)	0.0149 (12)	−0.0083 (12)
C25	0.0577 (13)	0.0476 (11)	0.0552 (13)	0.0001 (9)	0.0192 (10)	−0.0052 (10)
O11	0.0525 (10)	0.167 (3)	0.0593 (12)	−0.0153 (13)	0.0032 (9)	0.0261 (13)
C41	0.0754 (17)	0.0653 (15)	0.0558 (14)	0.0042 (13)	0.0249 (12)	0.0013 (12)
C16	0.0532 (13)	0.0592 (13)	0.0552 (13)	0.0103 (10)	0.0088 (10)	0.0057 (11)
C43	0.0451 (12)	0.0746 (16)	0.0599 (14)	−0.0100 (11)	0.0136 (10)	−0.0069 (12)
C60	0.0531 (13)	0.0603 (13)	0.0466 (12)	0.0038 (10)	0.0103 (9)	0.0005 (10)
C23	0.0551 (13)	0.0636 (14)	0.0599 (14)	0.0077 (11)	0.0140 (10)	−0.0156 (12)
C34	0.0537 (12)	0.0448 (11)	0.0521 (12)	0.0009 (9)	0.0185 (9)	−0.0002 (9)
C20	0.0529 (12)	0.0538 (12)	0.0525 (12)	−0.0006 (10)	0.0062 (9)	0.0089 (10)
C17	0.0613 (14)	0.0691 (15)	0.0525 (13)	0.0013 (12)	0.0052 (10)	−0.0023 (11)
C49	0.0504 (11)	0.0667 (14)	0.0478 (12)	−0.0045 (10)	0.0102 (9)	−0.0038 (10)
C59	0.0709 (16)	0.0680 (15)	0.0569 (14)	−0.0139 (13)	0.0092 (12)	0.0027 (12)
C40	0.0487 (12)	0.0528 (12)	0.0658 (14)	0.0010 (10)	0.0136 (10)	0.0084 (11)
C54	0.0617 (14)	0.0510 (12)	0.0441 (11)	0.0039 (10)	0.0028 (10)	0.0047 (9)
C18	0.0649 (15)	0.0471 (12)	0.0755 (16)	−0.0036 (11)	0.0188 (12)	−0.0075 (11)
C8	0.0595 (15)	0.0562 (13)	0.0813 (17)	−0.0127 (11)	0.0261 (12)	0.0010 (12)
C45	0.0451 (11)	0.0778 (16)	0.0460 (11)	−0.0076 (11)	0.0078 (9)	0.0041 (11)
C57	0.090 (2)	0.0570 (14)	0.0708 (17)	0.0228 (14)	0.0305 (14)	0.0067 (12)
F1	0.0635 (11)	0.1211 (18)	0.206 (3)	−0.0072 (12)	0.0488 (14)	−0.0150 (19)
C46	0.0725 (16)	0.0675 (15)	0.0504 (13)	−0.0133 (12)	0.0161 (11)	−0.0026 (11)
C52	0.0623 (15)	0.0890 (19)	0.0518 (14)	−0.0156 (14)	0.0054 (12)	0.0103 (13)
C9	0.0651 (15)	0.0510 (12)	0.0813 (18)	−0.0063 (11)	0.0257 (13)	0.0091 (12)
O12	0.0756 (13)	0.127 (2)	0.0581 (11)	−0.0367 (13)	0.0093 (9)	0.0189 (12)
C28	0.0566 (14)	0.0882 (19)	0.0563 (14)	0.0136 (13)	0.0120 (11)	−0.0028 (13)
C37	0.107 (2)	0.0564 (15)	0.0549 (15)	−0.0064 (15)	0.0047 (14)	−0.0062 (12)
C6	0.095 (2)	0.0777 (19)	0.103 (3)	−0.0136 (17)	0.059 (2)	−0.0022 (18)
C39	0.0538 (14)	0.0746 (18)	0.087 (2)	−0.0125 (12)	0.0026 (13)	0.0205 (16)
C1	0.0598 (15)	0.0631 (16)	0.114 (3)	−0.0066 (13)	0.0266 (16)	−0.0117 (16)
C58	0.104 (2)	0.0490 (13)	0.0621 (16)	−0.0026 (14)	0.0178 (14)	−0.0018 (12)
C5	0.0826 (19)	0.0778 (18)	0.0572 (15)	−0.0028 (14)	0.0211 (13)	0.0130 (13)
C3	0.0620 (14)	0.0687 (15)	0.0539 (13)	−0.0025 (12)	0.0115 (11)	0.0076 (11)
C29	0.0503 (13)	0.0754 (17)	0.0754 (17)	0.0052 (12)	0.0131 (11)	0.0078 (14)
C33	0.0664 (16)	0.0569 (14)	0.0807 (18)	0.0027 (11)	0.0227 (13)	0.0158 (12)
C7	0.0556 (13)	0.0575 (13)	0.0635 (15)	−0.0085 (11)	0.0073 (10)	0.0073 (11)
C30	0.0536 (15)	0.0722 (17)	0.122 (3)	0.0019 (13)	0.0294 (15)	0.0130 (18)
C36	0.0781 (17)	0.0610 (14)	0.0534 (13)	−0.0003 (12)	0.0192 (12)	−0.0026 (11)
O14	0.0865 (17)	0.112 (2)	0.126 (2)	−0.0344 (15)	−0.0269 (15)	0.0572 (18)
C53	0.097 (2)	0.0675 (16)	0.0522 (14)	−0.0190 (15)	0.0026 (14)	0.0112 (12)
C38	0.087 (2)	0.0602 (16)	0.0713 (17)	−0.0230 (14)	−0.0125 (15)	0.0084 (14)
C13	0.0673 (16)	0.0571 (13)	0.0785 (17)	−0.0080 (12)	0.0343 (13)	−0.0029 (12)
C56	0.0570 (13)	0.0609 (14)	0.0657 (15)	0.0095 (11)	0.0172 (11)	0.0097 (11)
C2	0.0610 (16)	0.0759 (18)	0.0811 (19)	−0.0009 (14)	−0.0017 (14)	0.0033 (15)
C42	0.0502 (13)	0.092 (2)	0.0668 (16)	0.0086 (13)	0.0159 (11)	−0.0009 (14)

O5	0.190 (4)	0.113 (3)	0.165 (4)	0.010 (3)	0.022 (3)	0.017 (3)

Geometric parameters (Å, º)

O4—C12	1.341 (3)	C41—C42	1.348 (4)
O4—C13	1.433 (3)	C16—H16	0.9300
O9—C32	1.322 (3)	C16—C17	1.379 (4)
O9—C33	1.439 (3)	C43—H43	0.9300
O13—C51	1.202 (3)	C43—C42	1.387 (4)
O10—H10	0.8200	C60—H60	0.9300
O10—C47	1.427 (3)	C60—C59	1.379 (4)
N1—C11	1.402 (3)	C23—H23	0.9300
N1—C14	1.466 (3)	C34—H34	0.9800
N1—C12	1.382 (3)	C34—C33	1.520 (3)
O2—C11	1.203 (3)	C20—H20	0.9300
N2—C31	1.400 (3)	C17—H17	0.9300
N2—C32	1.385 (3)	C17—C18	1.372 (4)
N2—C34	1.459 (3)	C49—H49A	0.9700
N3—C51	1.393 (3)	C49—H49B	0.9700
N3—C54	1.472 (3)	C59—H59	0.9300
N3—C52	1.386 (4)	C59—C58	1.375 (5)
O6—H6A	0.8200	C40—H40	0.9300
O6—C27	1.412 (3)	C40—C39	1.385 (4)
O1—H1	0.8200	C54—H54	0.9800
O1—C7	1.411 (3)	C54—C53	1.532 (4)
O7—C31	1.213 (3)	C18—H18	0.9300
C44—C47	1.515 (4)	C8—H8A	0.9700
C44—C43	1.390 (3)	C8—H8B	0.9700
C44—C45	1.379 (3)	C8—C9	1.525 (4)
C51—C50	1.499 (3)	C8—C7	1.521 (4)
F2—C21	1.368 (3)	C45—H45	0.9300
O3—C12	1.194 (3)	C45—C46	1.384 (4)
C24—C27	1.517 (4)	C57—H57	0.9300
C24—C25	1.365 (3)	C57—C58	1.378 (5)
C24—C23	1.392 (4)	C57—C56	1.388 (4)
C11—C10	1.503 (3)	F1—C1	1.360 (4)
C55—C60	1.386 (3)	C46—H46	0.9300
C55—C54	1.516 (3)	C52—O12	1.346 (4)
C55—C56	1.385 (4)	C9—H9A	0.9700
C4—C5	1.380 (4)	C9—H9B	0.9700
C4—C3	1.367 (3)	O12—C53	1.427 (4)
C4—C7	1.513 (4)	C28—H28A	0.9700
C10—H10A	0.9700	C28—H28B	0.9700
C10—H10B	0.9700	C28—C29	1.537 (4)
C10—C9	1.523 (4)	C37—H37	0.9300
O8—C32	1.211 (3)	C37—C36	1.378 (4)
C50—H50A	0.9700	C37—C38	1.363 (5)
C50—H50B	0.9700	C6—H6	0.9300

C50—C49	1.520 (4)	C6—C1	1.361 (5)
C14—H14	0.9800	C6—C5	1.387 (5)
C14—C15	1.508 (3)	C39—H39	0.9300
C14—C13	1.538 (4)	C39—C38	1.375 (5)
C19—H19	0.9300	C1—C2	1.339 (5)
C19—C20	1.373 (4)	C58—H58	0.9300
C19—C18	1.377 (4)	C5—H5	0.9300
C21—C26	1.363 (4)	C3—H3	0.9300
C21—C22	1.358 (4)	C3—C2	1.377 (4)
C26—H26	0.9300	C29—H29A	0.9700
C26—C25	1.384 (4)	C29—H29B	0.9700
C35—C34	1.511 (3)	C29—C30	1.469 (4)
C35—C40	1.383 (4)	C33—H33A	0.9700
C35—C36	1.382 (4)	C33—H33B	0.9700
C15—C16	1.386 (3)	C7—H7	0.9800
C15—C20	1.384 (3)	C30—H30A	0.9700
F3—C41	1.368 (4)	C30—H30B	0.9700
C48—H48A	0.9700	C36—H36	0.9300
C48—H48B	0.9700	O14—H14A	0.8497
C48—C47	1.520 (3)	O14—H14B	0.8504
C48—C49	1.519 (3)	C53—H53A	0.9700
C47—H47	0.9800	C53—H53B	0.9700
C31—C30	1.504 (4)	C38—H38	0.9300
C27—H27	0.9800	C13—H13A	0.9700
C27—C28	1.517 (3)	C13—H13B	0.9700
C22—H22	0.9300	C56—H56	0.9300
C22—C23	1.379 (4)	C2—H2	0.9300
C25—H25	0.9300	C42—H42	0.9300
O11—C52	1.181 (3)	O5—H5A	0.9000
C41—C46	1.363 (4)	O5—H5B	0.8495

C12—O4—C13	111.37 (19)	C50—C49—H49B	108.9
C32—O9—C33	110.00 (19)	C48—C49—C50	113.21 (19)
C47—O10—H10	109.5	C48—C49—H49A	108.9
C11—N1—C14	119.60 (19)	C48—C49—H49B	108.9
C12—N1—C11	128.0 (2)	H49A—C49—H49B	107.8
C12—N1—C14	112.1 (2)	C60—C59—H59	119.7
C31—N2—C34	120.80 (19)	C58—C59—C60	120.7 (3)
C32—N2—C31	126.4 (2)	C58—C59—H59	119.7
C32—N2—C34	111.2 (2)	C35—C40—H40	119.9
C51—N3—C54	120.8 (2)	C35—C40—C39	120.2 (3)
C52—N3—C51	128.41 (19)	C39—C40—H40	119.9
C52—N3—C54	110.8 (2)	N3—C54—C55	111.16 (18)
C27—O6—H6A	109.5	N3—C54—H54	110.1
C7—O1—H1	109.5	N3—C54—C53	99.64 (19)
C43—C44—C47	121.2 (2)	C55—C54—H54	110.1
C45—C44—C47	121.4 (2)	C55—C54—C53	115.2 (2)
C45—C44—C43	117.4 (2)	C53—C54—H54	110.1

O13—C51—N3	119.0 (2)	C19—C18—H18	120.2
O13—C51—C50	122.4 (2)	C17—C18—C19	119.5 (2)
N3—C51—C50	118.59 (19)	C17—C18—H18	120.2
C25—C24—C27	122.0 (2)	H8A—C8—H8B	107.5
C25—C24—C23	117.9 (2)	C9—C8—H8A	108.4
C23—C24—C27	120.1 (2)	C9—C8—H8B	108.4
N1—C11—C10	118.1 (2)	C7—C8—H8A	108.4
O2—C11—N1	117.7 (2)	C7—C8—H8B	108.4
O2—C11—C10	124.3 (2)	C7—C8—C9	115.3 (2)
C60—C55—C54	120.0 (2)	C44—C45—H45	118.9
C56—C55—C60	118.7 (2)	C44—C45—C46	122.1 (2)
C56—C55—C54	121.2 (2)	C46—C45—H45	118.9
C5—C4—C7	122.2 (2)	C58—C57—H57	119.9
C3—C4—C5	117.9 (3)	C58—C57—C56	120.2 (3)
C3—C4—C7	119.9 (2)	C56—C57—H57	119.9
C11—C10—H10A	109.0	C41—C46—C45	117.9 (2)
C11—C10—H10B	109.0	C41—C46—H46	121.1
C11—C10—C9	112.9 (2)	C45—C46—H46	121.1
H10A—C10—H10B	107.8	O11—C52—N3	128.3 (3)
C9—C10—H10A	109.0	O11—C52—O12	122.9 (3)
C9—C10—H10B	109.0	O12—C52—N3	108.8 (2)
C51—C50—H50A	109.4	C10—C9—C8	112.4 (2)
C51—C50—H50B	109.4	C10—C9—H9A	109.1
C51—C50—C49	111.13 (19)	C10—C9—H9B	109.1
H50A—C50—H50B	108.0	C8—C9—H9A	109.1
C49—C50—H50A	109.4	C8—C9—H9B	109.1
C49—C50—H50B	109.4	H9A—C9—H9B	107.9
N1—C14—H14	109.5	C52—O12—C53	110.4 (2)
N1—C14—C15	112.83 (18)	C27—C28—H28A	108.2
N1—C14—C13	100.9 (2)	C27—C28—H28B	108.2
C15—C14—H14	109.5	C27—C28—C29	116.3 (2)
C15—C14—C13	114.3 (2)	H28A—C28—H28B	107.4
C13—C14—H14	109.5	C29—C28—H28A	108.2
C20—C19—H19	120.0	C29—C28—H28B	108.2
C20—C19—C18	120.0 (2)	C36—C37—H37	119.7
C18—C19—H19	120.0	C38—C37—H37	119.7
C26—C21—F2	118.2 (3)	C38—C37—C36	120.5 (3)
C22—C21—F2	119.3 (2)	C1—C6—H6	120.5
C22—C21—C26	122.6 (3)	C1—C6—C5	119.0 (3)
C21—C26—H26	120.7	C5—C6—H6	120.5
C21—C26—C25	118.5 (3)	C40—C39—H39	120.0
C25—C26—H26	120.7	C38—C39—C40	120.0 (3)
C40—C35—C34	118.7 (2)	C38—C39—H39	120.0
C36—C35—C34	122.2 (2)	F1—C1—C6	118.5 (4)
C36—C35—C40	118.9 (2)	C2—C1—F1	120.1 (3)
O4—C12—N1	109.1 (2)	C2—C1—C6	121.4 (3)
O3—C12—O4	122.5 (2)	C59—C58—C57	119.4 (3)
O3—C12—N1	128.4 (2)	C59—C58—H58	120.3

C16—C15—C14	121.0 (2)	C57—C58—H58	120.3
C20—C15—C14	120.8 (2)	C4—C5—C6	120.7 (3)
C20—C15—C16	118.2 (2)	C4—C5—H5	119.7
H48A—C48—H48B	107.9	C6—C5—H5	119.7
C47—C48—H48A	109.2	C4—C3—H3	119.2
C47—C48—H48B	109.2	C4—C3—C2	121.6 (3)
C49—C48—H48A	109.2	C2—C3—H3	119.2
C49—C48—H48B	109.2	C28—C29—H29A	108.8
C49—C48—C47	111.97 (19)	C28—C29—H29B	108.8
O10—C47—C44	110.8 (2)	H29A—C29—H29B	107.7
O10—C47—C48	107.26 (19)	C30—C29—C28	113.6 (3)
O10—C47—H47	108.5	C30—C29—H29A	108.8
C44—C47—C48	113.3 (2)	C30—C29—H29B	108.8
C44—C47—H47	108.5	O9—C33—C34	106.2 (2)
C48—C47—H47	108.5	O9—C33—H33A	110.5
N2—C31—C30	118.7 (2)	O9—C33—H33B	110.5
O7—C31—N2	117.4 (2)	C34—C33—H33A	110.5
O7—C31—C30	123.9 (2)	C34—C33—H33B	110.5
O6—C27—C24	113.2 (2)	H33A—C33—H33B	108.7
O6—C27—H27	107.7	O1—C7—C4	111.7 (2)
O6—C27—C28	107.7 (2)	O1—C7—C8	108.1 (2)
C24—C27—H27	107.7	O1—C7—H7	108.3
C24—C27—C28	112.7 (2)	C4—C7—C8	111.9 (2)
C28—C27—H27	107.7	C4—C7—H7	108.3
O9—C32—N2	109.8 (2)	C8—C7—H7	108.3
O8—C32—O9	122.4 (2)	C31—C30—H30A	109.0
O8—C32—N2	127.8 (3)	C31—C30—H30B	109.0
C21—C22—H22	121.1	C29—C30—C31	112.8 (2)
C21—C22—C23	117.9 (2)	C29—C30—H30A	109.0
C23—C22—H22	121.1	C29—C30—H30B	109.0
C24—C25—C26	121.4 (2)	H30A—C30—H30B	107.8
C24—C25—H25	119.3	C35—C36—H36	119.8
C26—C25—H25	119.3	C37—C36—C35	120.4 (3)
C46—C41—F3	118.6 (3)	C37—C36—H36	119.8
C42—C41—F3	118.7 (3)	H14A—O14—H14B	109.5
C42—C41—C46	122.6 (3)	C54—C53—H53A	110.7
C15—C16—H16	119.8	C54—C53—H53B	110.7
C17—C16—C15	120.4 (2)	O12—C53—C54	105.4 (2)
C17—C16—H16	119.8	O12—C53—H53A	110.7
C44—C43—H43	119.5	O12—C53—H53B	110.7
C42—C43—C44	121.0 (2)	H53A—C53—H53B	108.8
C42—C43—H43	119.5	C37—C38—C39	120.0 (3)
C55—C60—H60	119.7	C37—C38—H38	120.0
C59—C60—C55	120.5 (2)	C39—C38—H38	120.0
C59—C60—H60	119.7	O4—C13—C14	106.0 (2)
C24—C23—H23	119.2	O4—C13—H13A	110.5
C22—C23—C24	121.7 (2)	O4—C13—H13B	110.5
C22—C23—H23	119.2	C14—C13—H13A	110.5

N2—C34—C35	115.10 (19)	C14—C13—H13B	110.5
N2—C34—H34	109.5	H13A—C13—H13B	108.7
N2—C34—C33	100.31 (19)	C55—C56—C57	120.5 (3)
C35—C34—H34	109.5	C55—C56—H56	119.8
C35—C34—C33	112.7 (2)	C57—C56—H56	119.8
C33—C34—H34	109.5	C1—C2—C3	119.4 (3)
C19—C20—C15	121.3 (2)	C1—C2—H2	120.3
C19—C20—H20	119.4	C3—C2—H2	120.3
C15—C20—H20	119.4	C41—C42—C43	119.0 (2)
C16—C17—H17	119.7	C41—C42—H42	120.5
C18—C17—C16	120.6 (2)	C43—C42—H42	120.5
C18—C17—H17	119.7	H5A—O5—H5B	104.3
C50—C49—H49A	108.9		

O13—C51—C50—C49	10.0 (3)	C43—C44—C47—C48	−117.3 (2)
N1—C11—C10—C9	−176.3 (2)	C43—C44—C45—C46	0.0 (4)
N1—C14—C15—C16	51.3 (3)	C60—C55—C54—N3	−105.9 (2)
N1—C14—C15—C20	−130.3 (2)	C60—C55—C54—C53	141.7 (2)
N1—C14—C13—O4	−7.7 (3)	C60—C55—C56—C57	−0.7 (4)
O2—C11—C10—C9	4.5 (4)	C60—C59—C58—C57	0.4 (4)
N2—C31—C30—C29	140.7 (3)	C23—C24—C27—O6	173.8 (2)
N2—C34—C33—O9	15.2 (3)	C23—C24—C27—C28	−63.7 (3)
N3—C51—C50—C49	−169.7 (2)	C23—C24—C25—C26	−2.0 (4)
N3—C54—C53—O12	−21.3 (3)	C34—N2—C31—O7	5.2 (3)
N3—C52—O12—C53	−5.9 (4)	C34—N2—C31—C30	−171.9 (2)
O6—C27—C28—C29	63.6 (3)	C34—N2—C32—O9	5.6 (3)
O7—C31—C30—C29	−36.2 (5)	C34—N2—C32—O8	−171.7 (3)
C44—C43—C42—C41	1.1 (4)	C34—C35—C40—C39	−176.0 (2)
C44—C45—C46—C41	0.0 (4)	C34—C35—C36—C37	177.1 (2)
C51—N3—C54—C55	78.2 (3)	C20—C19—C18—C17	0.3 (4)
C51—N3—C54—C53	−159.8 (2)	C20—C15—C16—C17	−1.0 (4)
C51—N3—C52—O11	−11.4 (6)	C49—C48—C47—O10	−172.8 (2)
C51—N3—C52—O12	169.3 (3)	C49—C48—C47—C44	64.7 (3)
C51—C50—C49—C48	179.6 (2)	C40—C35—C34—N2	−154.8 (2)
F2—C21—C26—C25	−176.8 (2)	C40—C35—C34—C33	91.0 (3)
F2—C21—C22—C23	176.8 (2)	C40—C35—C36—C37	0.8 (4)
C24—C27—C28—C29	−61.9 (3)	C40—C39—C38—C37	0.4 (4)
C11—N1—C14—C15	69.8 (3)	C54—N3—C51—O13	2.5 (3)
C11—N1—C14—C13	−167.9 (2)	C54—N3—C51—C50	−177.8 (2)
C11—N1—C12—O4	171.2 (2)	C54—N3—C52—O11	170.0 (4)
C11—N1—C12—O3	−9.2 (4)	C54—N3—C52—O12	−9.4 (3)
C11—C10—C9—C8	178.7 (2)	C54—C55—C60—C59	−179.9 (2)
C55—C60—C59—C58	−0.3 (4)	C54—C55—C56—C57	179.7 (2)
C55—C54—C53—O12	97.7 (3)	C18—C19—C20—C15	−0.9 (4)
C4—C3—C2—C1	1.0 (5)	C45—C44—C47—O10	−58.4 (3)
C14—N1—C11—O2	6.5 (3)	C45—C44—C47—C48	62.1 (3)
C14—N1—C11—C10	−172.8 (2)	C45—C44—C43—C42	−0.5 (4)
C14—N1—C12—O4	−2.4 (3)	F1—C1—C2—C3	179.2 (3)

C14—N1—C12—O3	177.2 (3)	C46—C41—C42—C43	−1.1 (5)
C14—C15—C16—C17	177.5 (2)	C52—N3—C51—O13	−176.0 (3)
C14—C15—C20—C19	−177.2 (2)	C52—N3—C51—C50	3.7 (4)
C21—C26—C25—C24	0.3 (4)	C52—N3—C54—C55	−103.0 (3)
C21—C22—C23—C24	−0.2 (4)	C52—N3—C54—C53	18.9 (3)
C26—C21—C22—C23	−1.6 (4)	C52—O12—C53—C54	17.9 (4)
C35—C34—C33—O9	138.1 (2)	C9—C8—C7—O1	−57.1 (3)
C35—C40—C39—C38	−1.0 (4)	C9—C8—C7—C4	179.5 (2)
C12—O4—C13—C14	7.1 (3)	C28—C29—C30—C31	−170.7 (3)
C12—N1—C11—O2	−166.7 (2)	C6—C1—C2—C3	−0.2 (5)
C12—N1—C11—C10	14.0 (3)	C1—C6—C5—C4	0.1 (5)
C12—N1—C14—C15	−116.0 (2)	C58—C57—C56—C55	0.8 (4)
C12—N1—C14—C13	6.3 (3)	C5—C4—C3—C2	−1.1 (4)
C15—C14—C13—O4	113.6 (2)	C5—C4—C7—O1	−30.1 (3)
C15—C16—C17—C18	0.3 (4)	C5—C4—C7—C8	91.3 (3)
F3—C41—C46—C45	−179.4 (2)	C5—C6—C1—F1	−179.7 (3)
F3—C41—C42—C43	178.8 (3)	C5—C6—C1—C2	−0.3 (5)
C47—C44—C43—C42	178.9 (2)	C3—C4—C5—C6	0.5 (4)
C47—C44—C45—C46	−179.5 (2)	C3—C4—C7—O1	151.6 (2)
C47—C48—C49—C50	−171.7 (2)	C3—C4—C7—C8	−87.0 (3)
C31—N2—C32—O9	171.3 (2)	C33—O9—C32—N2	5.2 (3)
C31—N2—C32—O8	−6.1 (4)	C33—O9—C32—O8	−177.3 (3)
C31—N2—C34—C35	59.3 (3)	C7—C4—C5—C6	−177.7 (3)
C31—N2—C34—C33	−179.5 (2)	C7—C4—C3—C2	177.2 (3)
C27—C24—C25—C26	176.3 (2)	C7—C8—C9—C10	−60.1 (3)
C27—C24—C23—C22	−176.3 (2)	C36—C35—C34—N2	29.0 (3)
C27—C28—C29—C30	−88.3 (4)	C36—C35—C34—C33	−85.2 (3)
C32—O9—C33—C34	−13.3 (3)	C36—C35—C40—C39	0.4 (3)
C32—N2—C31—O7	−159.1 (2)	C36—C37—C38—C39	0.8 (4)
C32—N2—C31—C30	23.7 (4)	C38—C37—C36—C35	−1.4 (4)
C32—N2—C34—C35	−134.1 (2)	C13—O4—C12—N1	−3.2 (3)
C32—N2—C34—C33	−12.9 (3)	C13—O4—C12—O3	177.1 (2)
C22—C21—C26—C25	1.6 (4)	C13—C14—C15—C16	−63.2 (3)
C25—C24—C27—O6	−4.4 (3)	C13—C14—C15—C20	115.2 (3)
C25—C24—C27—C28	118.1 (3)	C56—C55—C60—C59	0.5 (3)
C25—C24—C23—C22	1.9 (4)	C56—C55—C54—N3	73.6 (3)
O11—C52—O12—C53	174.7 (4)	C56—C55—C54—C53	−38.8 (3)
C16—C15—C20—C19	1.3 (4)	C56—C57—C58—C59	−0.6 (4)
C16—C17—C18—C19	0.0 (4)	C42—C41—C46—C45	0.6 (4)
C43—C44—C47—O10	122.1 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O10—H10···O8	0.82	2.02	2.837 (3)	173
O6—H6*A*···O10^{i}	0.82	1.99	2.797 (3)	167
O1—H1···O14^{ii}	0.82	1.85	2.669 (4)	174
C22—H22···O6^{iii}	0.93	2.65	3.522 (3)	157

C33—H33*A*···O7^{iv}	0.97	2.57	3.337 (4)	137
C30—H30*A*···O6	0.97	2.58	3.149 (3)	118
C30—H30*B*···O8	0.97	2.31	2.860 (4)	115
O14—H14*A*···O1	0.85	1.88	2.728 (3)	173
O14—H14*B*···O13^{ii}	0.85	2.01	2.825 (3)	161
C13—H13*B*···F2^{iv}	0.97	2.51	3.347 (3)	145
C42—H42···O3^{v}	0.93	2.37	3.232 (3)	153
O5—H5*B*···O3^{v}	0.85	2.09	2.928 (5)	171

Symmetry codes: (i) −*x*−1, *y*+1/2, −*z*; (ii) −*x*, *y*+1/2, −*z*+1; (iii) *x*, *y*+1, *z*; (iv) −*x*, *y*−1/2, −*z*; (v) *x*, *y*−1, *z*.