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Pentafluorophenyl (3*R*,4*R*,5*R*)-5-{[(3*R*,4*R*,5*R*)-5-azidomethyl-3,4-dimethoxy-2,3,4,5-tetrahydrofuran-3-carbonylamino]methyl}-3,4-dimethoxy-2,3,4,5-tetrahydrofuran-3-carboxylate

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.004 Å
R factor = 0.036
wR factor = 0.081
Data-to-parameter ratio = 11.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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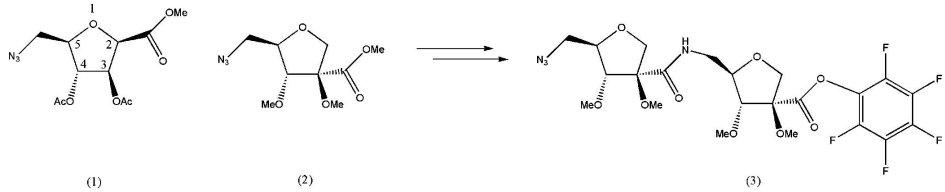
The crystal structure of the title compound, $C_{22}H_{25}F_5N_4O_9$, an important intermediate in the synthesis of novel biopolymers containing branched carbon chains, establishes the relative stereochemistry at all six chiral centres of the dipeptide. The structure may indicate a predisposition to the organization of secondary structure by novel dipeptide isosteres. An intermolecular hydrogen bond between the NH group and one of the N atoms of the azide group contributes to the stabilization of the packing.

Comment

Sugar amino acids (SAAs) have been extensively studied as peptidomimetics (Chakraborty *et al.*, 2005). δ-Tetrahydrofuran (THF) SAAs such as (1) (Smith *et al.*, 2003; Chakraborty *et al.*, 2004) have become well established as dipeptide isosteres (Grotenberg *et al.*, 2004). Such systems continue to provide an increased understanding of the factors inducing secondary structure and insight into the complex nature of protein folding (Billing & Nilsson, 2005; Claridge *et al.*, 2005; Long *et al.*, 1999) with potential chemotherapeutic activities as integrin antagonists (van Well *et al.*, 2004), enkephalin analogues (Montero *et al.*, 2004) and somatastatin mimics (Gruner *et al.*, 2002). In the past, almost all THF SAAs have contained linear carbon chains, since the only carbohydrate building blocks from which they can be derived have unbranched chains (Bols, 1996). Knowledge of the predisposition of monomers to adopt particular secondary structural motif may allow the design of bioactive peptidomimetic libraries.



(1) (2) (3)

However, new classes of branched carbohydrates suitable for short syntheses of branched carbon chain SAAs have recently become available by Kiliani or other procedures (Soengas *et al.*, 2005; Hotchkiss *et al.*, 2004,2006). The Ho crossed aldol (Ho, 1979, 1985) was the crucial step in the synthesis of branched SAAs such as (2) (Simone *et al.*, 2005). The azidoester (2) was converted by standard peptide procedures into the dimeric pentafluorophenyl ester (3) as a key intermediate for the generation of homooligomers having the branched *trans*-δ-SAA scaffold (2) as a component.

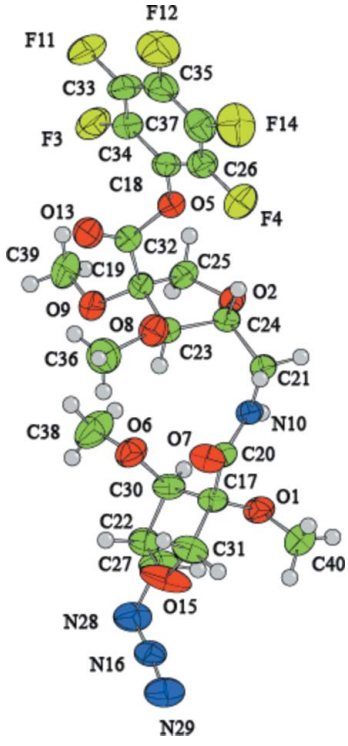


Figure 1
The structure of (3), with displacement ellipsoids drawn at the 50% probability level. H-atom radii are arbitrary.

The crystal structure reported in this paper firmly establishes the relative configuration of the six stereogenic centres in (3); the absolute configuration is consistent with the one determined by the use of D-ribose as the starting material for the synthesis. An intermolecular hydrogen bond between the H atom connected to N10 and N28, the first nitrogen of the azide chain, contributes to the stabilization of the packing.

Experimental

Compound (3) was crystallized by dissolving it in dichloromethane, adding a few drops of cyclohexane and allowing the slow competitive evaporation of the two solvents until clear colourless crystals formed.

Crystal data

$C_{22}H_{25}F_5N_4O_9$
M_r = 584.45
Orthorhombic, $P2_12_12_1$
a = 7.18471 (11) Å
b = 11.04142 (15) Å
c = 32.6727 (5) Å
V = 2591.91 (7) Å^3
Z = 4
D_x = 1.498 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 11003 reflections
θ = 4.2–69.3°
μ = 1.22 mm^{-1}
T = 120 K
Lath, colourless
0.50 × 0.20 × 0.10 mm

Data collection

Oxford Diffraction Gemini R CCD diffractometer
$\omega/2\theta$ scans
Absorption correction: multi-scan (CrysAlis; Oxford Diffraction, 2005)
T_{min} = 0.783, T_{max} = 0.885
11003 measured reflections
4281 independent reflections
3387 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 69.3°
$h = -6 \rightarrow 8$
$k = -9 \rightarrow 13$
$l = -39 \rightarrow 33$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.081
S = 0.92
4281 reflections
362 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F^2) + (0.04P)^2 + 0.55P]$ where $P = [\max(F_o^2,0) + 2F_c^2]/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}
Absolute structure: Flack (1983), 1237 Friedel pairs
Flack parameter: 0.00 (16)

Table 1
Selected geometric parameters (Å, °).

O1—C17	1.425 (3)	O15—C22	1.422 (3)
O1—C40	1.418 (4)	O15—C31	1.403 (3)
O2—C24	1.431 (3)	N16—N28	1.183 (3)
O2—C25	1.424 (3)	N16—N29	1.146 (3)
F3—C34	1.334 (3)	C17—C20	1.537 (3)
F4—C26	1.342 (3)	C17—C30	1.532 (4)
O5—C18	1.390 (3)	C17—C31	1.515 (3)
O5—C32	1.387 (3)	C18—C26	1.383 (4)
O6—C30	1.411 (3)	C18—C34	1.376 (3)
O6—C38	1.421 (4)	C19—C23	1.540 (3)
O7—C20	1.222 (3)	C19—C25	1.532 (4)
O8—C23	1.404 (3)	C19—C32	1.522 (3)
O8—C36	1.434 (4)	C21—C24	1.514 (3)
O9—C19	1.409 (3)	C22—C27	1.502 (4)
O9—C39	1.437 (4)	C22—C30	1.546 (4)
N10—C20	1.336 (3)	C23—C24	1.517 (3)
N10—C21	1.450 (3)	C26—C37	1.355 (4)
F11—C33	1.338 (3)	C27—N28	1.495 (4)
F12—C35	1.333 (3)	C33—C34	1.382 (4)
O13—C32	1.180 (3)	C33—C35	1.364 (5)
F14—C37	1.342 (3)	C35—C37	1.369 (4)

C17—O1—C40	114.6 (2)	C19—C23—O8	115.53 (19)
C24—O2—C25	110.12 (19)	C19—C23—C24	103.4 (2)
C18—O5—C32	115.64 (19)	O8—C23—C24	109.2 (2)
C30—O6—C38	113.3 (2)	C23—C24—C21	115.6 (2)
C23—O8—C36	113.0 (2)	C23—C24—O2	104.3 (2)
C19—O9—C39	114.5 (2)	C21—C24—O2	108.6 (2)
C20—N10—C21	121.8 (2)	C19—C25—O2	107.9 (2)
C22—O15—C31	109.17 (19)	C18—C26—F4	118.9 (2)
N28—N16—N29	172.9 (3)	C18—C26—C37	121.6 (3)
O1—C17—C20	111.3 (2)	F4—C26—C37	119.5 (3)
O1—C17—C30	104.20 (19)	C22—C27—N28	112.0 (3)
C20—C17—C30	113.26 (19)	C27—N28—N16	115.4 (2)
O1—C17—C31	113.9 (2)	C22—C30—C17	103.0 (2)
C20—C17—C31	112.2 (2)	C22—C30—O6	111.7 (2)
C30—C17—C31	101.3 (2)	C17—C30—O6	108.6 (2)
O5—C18—C26	118.8 (2)	C17—C31—O15	105.3 (2)
O5—C18—C34	123.2 (3)	C19—C32—O5	111.3 (2)
C26—C18—C34	118.0 (2)	C19—C32—O13	126.2 (3)
O9—C19—C23	108.06 (19)	O5—C32—O13	122.4 (2)
O9—C19—C25	113.9 (2)	F11—C33—C34	119.2 (3)
C23—C19—C25	102.24 (19)	F11—C33—C35	120.1 (3)
O9—C19—C32	107.94 (19)	C34—C33—C35	120.6 (3)
C23—C19—C32	108.4 (2)	C33—C34—C18	120.2 (3)
C25—C19—C32	115.9 (2)	C33—C34—F3	119.6 (3)
C17—C20—N10	115.5 (2)	C18—C34—F3	120.2 (3)
C17—C20—O7	121.0 (2)	F12—C35—C33	119.9 (3)
N10—C20—O7	123.4 (2)	F12—C35—C37	120.7 (3)
N10—C21—C24	114.3 (2)	C33—C35—C37	119.4 (3)
O15—C22—C27	106.1 (2)	F14—C37—C35	119.6 (3)
O15—C22—C30	106.8 (2)	F14—C37—C26	120.2 (3)
C27—C22—C30	114.9 (2)	C35—C37—C26	120.2 (3)

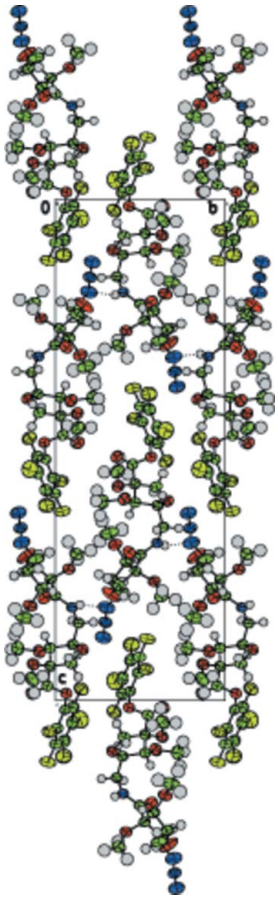


Figure 2
Packing diagram of (3), viewed down the *a* axis. Dashed lines indicate intermolecular hydrogen bonds.

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N10—H1···N28^{i}	0.94	2.23	3.120 (3)	158

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

The H atoms were located in a difference map, but those attached to C atoms were repositioned geometrically. They were initially refined with soft restraints on the bond lengths and angles to regularize their geometry (C—H in the range 0.93–98 Å, N—H in the range 0.86–0.89 Å and U_{iso}(H) in the range 1.2–1.5 times U_{eq} of the parent atom), after which their positions were refined with riding constraints.

Data collection: *CrysAlis* (Oxford Diffraction, 2005); cell refinement: *CrysAlis*; data reduction: *CrysAlis*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *CAMERON* (Watkin *et al.*, 1996); software used to prepare material for publication: *CRYSTALS*.

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supporting information

Acta Cryst. (2006). E**62**, o473–o475 [https://doi.org/10.1107/S1600536805043205]

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Crystal data

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$a = 7.18471\ (11)$ Å
$b = 11.04142\ (15)$ Å
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$h = -6 \rightarrow 8$
$k = -9 \rightarrow 13$
$l = -39 \rightarrow 33$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.081$
$S = 0.92$
4281 reflections
362 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F^2) + (0.04P)^2 + 0.55P]$ where $P = [\max(F_o^2,0) + 2F_c^2]/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -0.14$ e Å^{-3}
Absolute structure: Flack (1983), 1237 Friedel pairs
Absolute structure parameter: 0.00 (16)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.2249 (3)	0.58178 (16)	0.76221 (5)	0.0567
O2	0.1532 (3)	0.66323 (16)	0.60192 (5)	0.0545
F3	0.0797 (3)	0.46794 (18)	0.43728 (5)	0.0810
F4	−0.3609 (2)	0.65310 (16)	0.52301 (5)	0.0728
O5	−0.0124 (3)	0.56503 (15)	0.51413 (5)	0.0521
O6	0.3012 (2)	0.35309 (18)	0.68901 (5)	0.0613
O7	−0.1177 (2)	0.43686 (17)	0.70491 (5)	0.0585
O8	−0.1422 (3)	0.40677 (17)	0.59803 (5)	0.0604
O9	0.2815 (3)	0.37069 (16)	0.57241 (5)	0.0609
N10	0.0521 (3)	0.59896 (18)	0.68589 (6)	0.0432
F11	−0.1759 (3)	0.45764 (19)	0.37705 (5)	0.1000
F12	−0.5202 (3)	0.5498 (2)	0.38863 (6)	0.1035
O13	0.0116 (4)	0.36179 (17)	0.51341 (6)	0.0782
F14	−0.6115 (3)	0.64942 (19)	0.46177 (7)	0.0972
O15	0.2700 (3)	0.3302 (2)	0.78835 (7)	0.0899
N16	0.5502 (3)	0.2964 (2)	0.84412 (7)	0.0578
C17	0.1661 (3)	0.4742 (2)	0.74192 (7)	0.0464
C18	−0.1368 (4)	0.5586 (2)	0.48162 (7)	0.0499
C19	0.1615 (4)	0.4710 (2)	0.56830 (7)	0.0490
C20	0.0187 (3)	0.5017 (2)	0.70912 (7)	0.0433
C21	−0.0763 (3)	0.6374 (2)	0.65421 (7)	0.0470
C22	0.4169 (4)	0.3390 (3)	0.75934 (8)	0.0601
C23	0.0248 (4)	0.4698 (2)	0.60462 (7)	0.0466
C24	−0.0180 (4)	0.6029 (2)	0.61125 (7)	0.0464
C25	0.2586 (4)	0.5935 (3)	0.57352 (7)	0.0535
C26	−0.3142 (4)	0.6050 (3)	0.48672 (8)	0.0569
C27	0.5819 (4)	0.3878 (3)	0.78228 (8)	0.0669
N28	0.6467 (3)	0.3025 (3)	0.81486 (8)	0.0720
N29	0.4711 (4)	0.2836 (3)	0.87426 (8)	0.0762
C30	0.3459 (4)	0.4209 (3)	0.72428 (7)	0.0509
C31	0.1054 (4)	0.3741 (3)	0.77071 (9)	0.0595
C32	0.0506 (4)	0.4548 (2)	0.52899 (7)	0.0536
C33	−0.2226 (6)	0.5058 (3)	0.41321 (8)	0.0713
C34	−0.0917 (4)	0.5098 (3)	0.44416 (8)	0.0604
C35	−0.3964 (5)	0.5523 (3)	0.41902 (9)	0.0712
C36	−0.1239 (5)	0.2786 (3)	0.60382 (10)	0.0801
C37	−0.4410 (4)	0.6028 (3)	0.45602 (10)	0.0664
C38	0.4599 (5)	0.3180 (4)	0.66591 (10)	0.0950
C39	0.4293 (5)	0.3663 (3)	0.54282 (11)	0.0832
C40	0.0981 (6)	0.6264 (3)	0.79185 (10)	0.0845
H211	−0.0899	0.7268	0.6563	0.0663*
H212	−0.1990	0.5981	0.6594	0.0663*
H221	0.4413	0.2586	0.7484	0.0856*
H231	0.0889	0.4387	0.6294	0.0651*
H241	−0.1121	0.6319	0.5909	0.0654*

H251	0.2639	0.6369	0.5467	0.0765*
H252	0.3825	0.5806	0.5845	0.0770*
H271	0.6842	0.4000	0.7632	0.0950*
H272	0.5487	0.4654	0.7955	0.0945*
H301	0.4355	0.4855	0.7174	0.0725*
H311	0.0405	0.3081	0.7561	0.0846*
H312	0.0217	0.4038	0.7920	0.0855*
H361	−0.2449	0.2424	0.6020	0.1410*
H362	−0.0467	0.2455	0.5819	0.1409*
H363	−0.0668	0.2635	0.6306	0.1412*
H381	0.4143	0.2762	0.6411	0.1746*
H382	0.5264	0.3928	0.6588	0.1748*
H383	0.5365	0.2637	0.6820	0.1748*
H391	0.4888	0.2887	0.5447	0.1461*
H392	0.3813	0.3773	0.5154	0.1463*
H393	0.5206	0.4291	0.5487	0.1469*
H401	0.1359	0.7089	0.7991	0.1511*
H402	−0.0278	0.6307	0.7815	0.1514*
H403	0.1014	0.5761	0.8165	0.1516*
H1	0.1579	0.6462	0.6911	0.0652*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0701 (12)	0.0580 (11)	0.0418 (9)	−0.0085 (10)	−0.0070 (9)	−0.0026 (8)
O2	0.0681 (12)	0.0481 (9)	0.0473 (9)	−0.0010 (10)	0.0122 (9)	−0.0067 (8)
F3	0.0903 (13)	0.0974 (13)	0.0553 (10)	0.0168 (11)	0.0111 (9)	−0.0099 (9)
F4	0.0689 (10)	0.0816 (11)	0.0678 (10)	−0.0014 (10)	0.0106 (8)	−0.0130 (9)
O5	0.0641 (11)	0.0503 (10)	0.0419 (9)	0.0025 (9)	−0.0037 (8)	−0.0026 (8)
O6	0.0581 (10)	0.0781 (13)	0.0477 (9)	0.0127 (10)	−0.0068 (9)	−0.0090 (9)
O7	0.0490 (10)	0.0670 (11)	0.0596 (11)	−0.0138 (10)	−0.0093 (9)	0.0138 (9)
O8	0.0615 (12)	0.0573 (11)	0.0623 (11)	−0.0055 (10)	−0.0018 (9)	−0.0073 (9)
O9	0.0726 (12)	0.0576 (11)	0.0525 (10)	0.0174 (10)	0.0026 (9)	−0.0050 (9)
N10	0.0485 (11)	0.0456 (11)	0.0356 (9)	−0.0024 (10)	−0.0001 (9)	0.0003 (9)
F11	0.1528 (19)	0.1045 (15)	0.0428 (9)	0.0021 (14)	−0.0053 (10)	−0.0136 (9)
F12	0.1182 (17)	0.1082 (15)	0.0839 (13)	−0.0086 (14)	−0.0500 (13)	0.0019 (12)
O13	0.1300 (19)	0.0507 (11)	0.0540 (11)	−0.0005 (13)	−0.0259 (12)	−0.0108 (10)
F14	0.0682 (12)	0.1017 (14)	0.1216 (16)	0.0050 (12)	−0.0194 (11)	−0.0081 (13)
O15	0.0483 (11)	0.132 (2)	0.0891 (15)	−0.0158 (13)	−0.0161 (11)	0.0689 (15)
N16	0.0603 (14)	0.0699 (16)	0.0431 (13)	0.0128 (13)	−0.0172 (12)	0.0061 (12)
C17	0.0482 (14)	0.0522 (14)	0.0387 (12)	−0.0092 (13)	−0.0016 (11)	0.0063 (11)
C18	0.0648 (17)	0.0462 (13)	0.0386 (12)	−0.0065 (13)	−0.0009 (12)	0.0043 (11)
C19	0.0594 (15)	0.0471 (13)	0.0405 (12)	0.0093 (13)	−0.0029 (12)	−0.0054 (11)
C20	0.0427 (13)	0.0498 (14)	0.0374 (12)	−0.0014 (13)	0.0030 (10)	−0.0009 (11)
C21	0.0549 (15)	0.0478 (14)	0.0382 (12)	0.0078 (12)	−0.0022 (11)	−0.0008 (11)
C22	0.0642 (17)	0.0643 (17)	0.0519 (15)	0.0067 (15)	−0.0124 (13)	0.0048 (14)
C23	0.0526 (15)	0.0478 (13)	0.0394 (12)	0.0009 (13)	−0.0032 (11)	−0.0006 (11)
C24	0.0513 (15)	0.0482 (14)	0.0398 (12)	0.0054 (13)	−0.0047 (11)	0.0018 (11)

C25	0.0562 (15)	0.0605 (15)	0.0439 (13)	0.0022 (14)	0.0000 (12)	−0.0084 (12)
C26	0.0636 (17)	0.0562 (15)	0.0508 (15)	−0.0062 (15)	−0.0011 (13)	−0.0002 (13)
C27	0.0448 (15)	0.107 (3)	0.0484 (15)	0.0027 (17)	−0.0036 (12)	0.0122 (16)
N28	0.0496 (13)	0.109 (2)	0.0576 (14)	0.0228 (15)	−0.0039 (12)	0.0145 (15)
N29	0.0688 (16)	0.102 (2)	0.0578 (15)	−0.0032 (16)	−0.0082 (13)	0.0114 (15)
C30	0.0470 (13)	0.0645 (16)	0.0412 (12)	−0.0043 (14)	−0.0078 (11)	0.0056 (12)
C31	0.0491 (15)	0.0683 (18)	0.0611 (16)	−0.0084 (14)	−0.0060 (13)	0.0204 (14)
C32	0.0702 (18)	0.0486 (15)	0.0420 (13)	0.0041 (14)	0.0014 (12)	−0.0027 (12)
C33	0.106 (3)	0.070 (2)	0.0379 (14)	−0.007 (2)	−0.0035 (16)	0.0016 (13)
C34	0.0738 (19)	0.0621 (17)	0.0452 (14)	0.0075 (15)	0.0051 (13)	0.0040 (13)
C35	0.085 (2)	0.0665 (19)	0.0619 (18)	−0.0048 (18)	−0.0220 (17)	0.0082 (16)
C36	0.100 (3)	0.0616 (19)	0.079 (2)	−0.017 (2)	0.007 (2)	−0.0011 (17)
C37	0.0646 (19)	0.0568 (17)	0.078 (2)	−0.0052 (16)	−0.0054 (16)	0.0020 (16)
C38	0.074 (2)	0.144 (4)	0.067 (2)	0.035 (2)	−0.0031 (17)	−0.027 (2)
C39	0.095 (2)	0.077 (2)	0.077 (2)	0.0235 (19)	0.0241 (19)	−0.0138 (19)
C40	0.125 (3)	0.073 (2)	0.0558 (17)	0.016 (2)	0.0076 (19)	−0.0088 (16)

Geometric parameters (Å, º)

O1—C17	1.425 (3)	C21—C24	1.514 (3)
O1—C40	1.418 (4)	C21—H211	0.994
O2—C24	1.431 (3)	C21—H212	0.997
O2—C25	1.424 (3)	C22—C27	1.502 (4)
F3—C34	1.334 (3)	C22—C30	1.546 (4)
F4—C26	1.342 (3)	C22—H221	0.973
O5—C18	1.390 (3)	C23—C24	1.517 (3)
O5—C32	1.387 (3)	C23—H231	0.991
O6—C30	1.411 (3)	C24—H241	1.000
O6—C38	1.421 (4)	C25—H251	0.999
O7—C20	1.222 (3)	C25—H252	0.970
O8—C23	1.404 (3)	C26—C37	1.355 (4)
O8—C36	1.434 (4)	C27—N28	1.495 (4)
O9—C19	1.409 (3)	C27—H271	0.972
O9—C39	1.437 (4)	C27—H272	0.989
N10—C20	1.336 (3)	C30—H301	0.987
N10—C21	1.450 (3)	C31—H311	0.989
N10—H1	0.938	C31—H312	0.976
F11—C33	1.338 (3)	C33—C34	1.382 (4)
F12—C35	1.333 (3)	C33—C35	1.364 (5)
O13—C32	1.180 (3)	C35—C37	1.369 (4)
F14—C37	1.342 (3)	C36—H361	0.959
O15—C22	1.422 (3)	C36—H362	0.976
O15—C31	1.403 (3)	C36—H363	0.980
N16—N28	1.183 (3)	C38—H381	0.989
N16—N29	1.146 (3)	C38—H382	0.982
C17—C20	1.537 (3)	C38—H383	0.969
C17—C30	1.532 (4)	C39—H391	0.960
C17—C31	1.515 (3)	C39—H392	0.968

C18—C26	1.383 (4)	C39—H393	0.974
C18—C34	1.376 (3)	C40—H401	0.980
C19—C23	1.540 (3)	C40—H402	0.967
C19—C25	1.532 (4)	C40—H403	0.978
C19—C32	1.522 (3)		

C17—O1—C40	114.6 (2)	C18—C26—C37	121.6 (3)
C24—O2—C25	110.12 (19)	F4—C26—C37	119.5 (3)
C18—O5—C32	115.64 (19)	C22—C27—N28	112.0 (3)
C30—O6—C38	113.3 (2)	C22—C27—H271	109.1
C23—O8—C36	113.0 (2)	N28—C27—H271	107.9
C19—O9—C39	114.5 (2)	C22—C27—H272	109.8
C20—N10—C21	121.8 (2)	N28—C27—H272	108.0
C20—N10—H1	119.3	H271—C27—H272	110.0
C21—N10—H1	118.8	C27—N28—N16	115.4 (2)
C22—O15—C31	109.17 (19)	C22—C30—C17	103.0 (2)
N28—N16—N29	172.9 (3)	C22—C30—O6	111.7 (2)
O1—C17—C20	111.3 (2)	C17—C30—O6	108.6 (2)
O1—C17—C30	104.20 (19)	C22—C30—H301	112.1
C20—C17—C30	113.26 (19)	C17—C30—H301	111.0
O1—C17—C31	113.9 (2)	O6—C30—H301	110.2
C20—C17—C31	112.2 (2)	C17—C31—O15	105.3 (2)
C30—C17—C31	101.3 (2)	C17—C31—H311	111.9
O5—C18—C26	118.8 (2)	O15—C31—H311	109.9
O5—C18—C34	123.2 (3)	C17—C31—H312	112.0
C26—C18—C34	118.0 (2)	O15—C31—H312	110.0
O9—C19—C23	108.06 (19)	H311—C31—H312	107.6
O9—C19—C25	113.9 (2)	C19—C32—O5	111.3 (2)
C23—C19—C25	102.24 (19)	C19—C32—O13	126.2 (3)
O9—C19—C32	107.94 (19)	O5—C32—O13	122.4 (2)
C23—C19—C32	108.4 (2)	F11—C33—C34	119.2 (3)
C25—C19—C32	115.9 (2)	F11—C33—C35	120.1 (3)
C17—C20—N10	115.5 (2)	C34—C33—C35	120.6 (3)
C17—C20—O7	121.0 (2)	C33—C34—C18	120.2 (3)
N10—C20—O7	123.4 (2)	C33—C34—F3	119.6 (3)
N10—C21—C24	114.3 (2)	C18—C34—F3	120.2 (3)
N10—C21—H211	107.7	F12—C35—C33	119.9 (3)
C24—C21—H211	109.9	F12—C35—C37	120.7 (3)
N10—C21—H212	108.2	C33—C35—C37	119.4 (3)
C24—C21—H212	107.1	O8—C36—H361	108.7
H211—C21—H212	109.4	O8—C36—H362	108.9
O15—C22—C27	106.1 (2)	H361—C36—H362	108.3
O15—C22—C30	106.8 (2)	O8—C36—H363	108.9
C27—C22—C30	114.9 (2)	H361—C36—H363	111.4
O15—C22—H221	108.4	H362—C36—H363	110.6
C27—C22—H221	111.6	F14—C37—C35	119.6 (3)
C30—C22—H221	108.7	F14—C37—C26	120.2 (3)
C19—C23—O8	115.53 (19)	C35—C37—C26	120.2 (3)

C19—C23—C24	103.4 (2)	O6—C38—H381	107.3
O8—C23—C24	109.2 (2)	O6—C38—H382	106.6
C19—C23—H231	109.6	H381—C38—H382	111.1
O8—C23—H231	110.5	O6—C38—H383	109.6
C24—C23—H231	108.2	H381—C38—H383	110.2
C23—C24—C21	115.6 (2)	H382—C38—H383	111.8
C23—C24—O2	104.3 (2)	O9—C39—H391	108.4
C21—C24—O2	108.6 (2)	O9—C39—H392	110.8
C23—C24—H241	110.6	H391—C39—H392	109.3
C21—C24—H241	110.4	O9—C39—H393	110.0
O2—C24—H241	106.9	H391—C39—H393	108.8
C19—C25—O2	107.9 (2)	H392—C39—H393	109.5
C19—C25—H251	110.1	O1—C40—H401	108.1
O2—C25—H251	109.4	O1—C40—H402	112.3
C19—C25—H252	109.3	H401—C40—H402	107.3
O2—C25—H252	109.0	O1—C40—H403	110.4
H251—C25—H252	111.1	H401—C40—H403	108.8
C18—C26—F4	118.9 (2)	H402—C40—H403	109.8

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N10—H1···N28^{i}	0.94	2.23	3.120 (3)	158

Symmetry code: (i) −*x*+1, *y*+1/2, −*z*+3/2.