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(2*S*,3*R*,4*R*,5*S*)-3,4-*O*-Isopropylidene-2-methyl-1-oxa-6,9-diazaspiro[4.5]decane-7,10-dione

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Key indicators

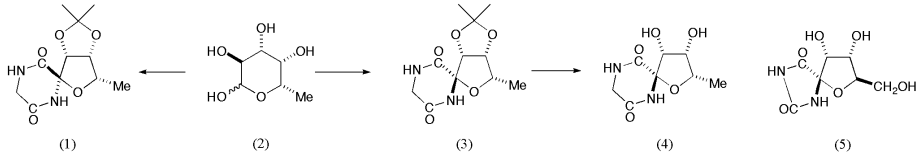
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.009 Å
Disorder in main residue
R factor = 0.073
wR factor = 0.139
Data-to-parameter ratio = 7.1

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

The title spirocarbopeptoid, $C_{11}H_{13}N_2O_5$, was prepared from L-fucose in a sequence that gave this and another anomer. The crystal structure determination removes ambiguities in the synthetic sequence.

Comment

Sugar amino acids (SAAs) provide an extensive family of peptidomimetics (Baron *et al.*, 2004; Chakraborty *et al.*, 2004), an important sub-class of which incorporate an α-amino acid at the anomeric position of a carbohydrate. Such SAAs may form spiro derivatives, some of which have been demonstrated to possess significant biological activity. Thus hydantocidin, (5), which is a potent and environmentally friendly herbicide, is a spirohydantoin of ribose (Nakajima *et al.*, 1991; Harumaya *et al.*, 1991); similar derivatives of glucose have been shown to be potent inhibitors of glycogen phosphorylase (Watson *et al.*, 1994; Bichard *et al.*, 1995).



In an investigation of spiro carbopeptide analogues retaining the footprint of the furanose form of L-fucose, (2), the sugar (2) was transformed into a separable anomeric mixture of the acetonides (1) and (3); one of these anomers,

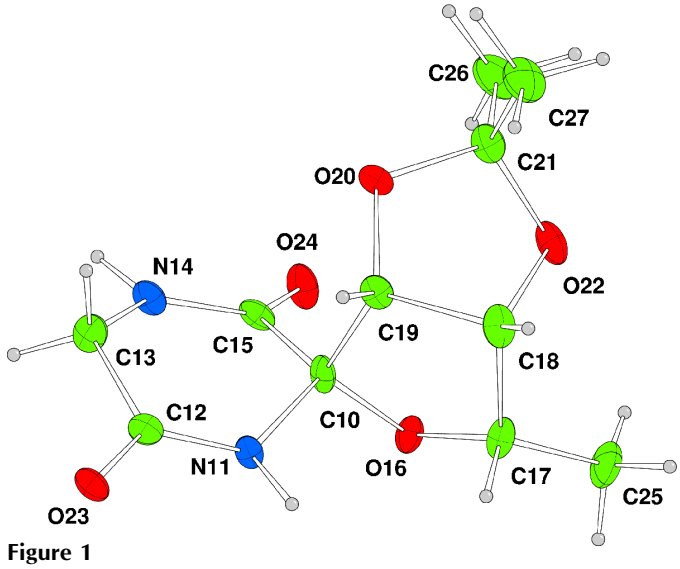


Figure 1
The ordered molecule, with displacement ellipsoids drawn at the 50% probability level. H atoms are of arbitrary radii.

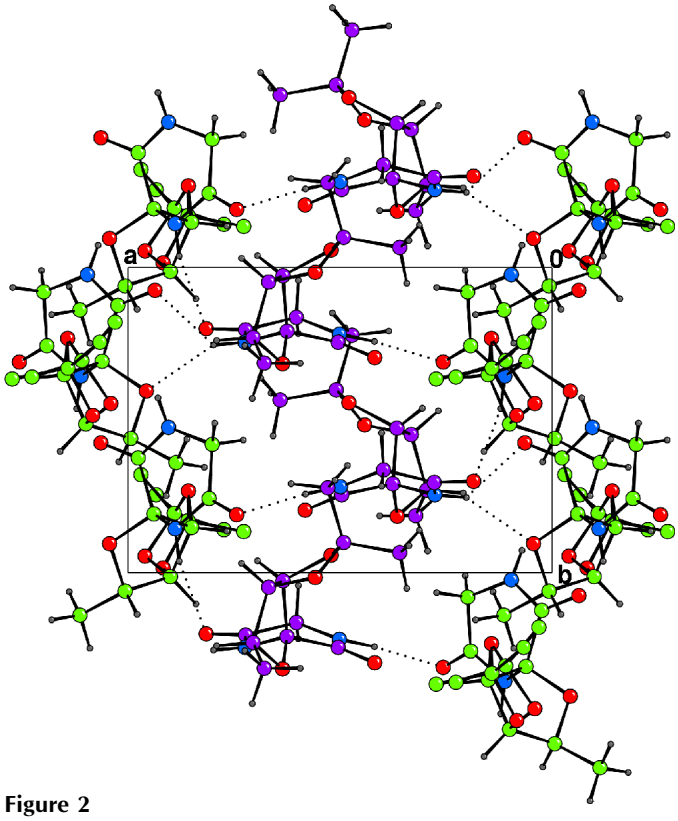


Figure 2
Packing diagram, viewed along the *c* axis. The molecules with the disordered atoms are confined to a single layer in the crystal structure. Dashed lines indicate hydrogen bonds.

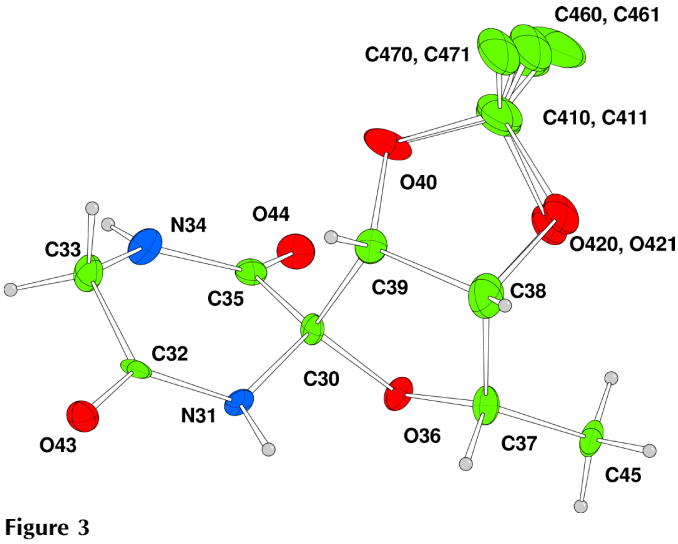


Figure 3
The disordered molecule, with displacement ellipsoids drawn at the 50% probability level. H atoms are of arbitrary radii.

(3), was highly crystalline and its structure has been shown by X-ray crystallographic analysis to have the relative stereochemistry shown in Fig. 1. The acetonide (3) was transformed into the deprotected analogue, (4), for biological evaluation. Fig. 2 is a packing diagram viewed down the *c* axis. The molecules containing the disordered atoms are, as is not uncommon for $Z' = 2$ structures, confined to a distinct layer through the crystal structure. The overall geometry is essentially the same in the ordered and the disordered molecules and is unexceptional.

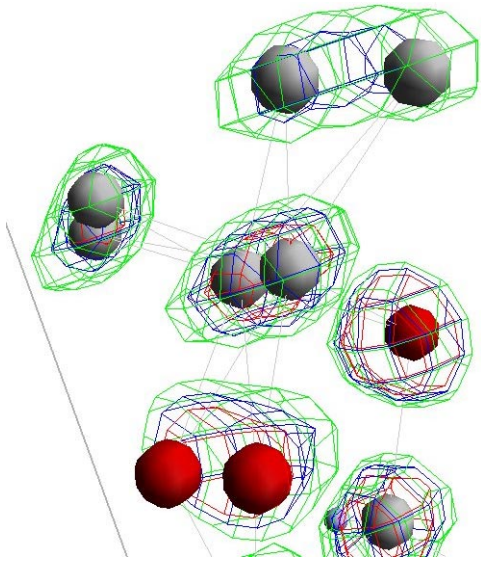


Figure 4
F_o electron density map in the region of the disordered atoms, contoured at 2 (green), 4 (blue) and 6 e Å^{-3} (red). Note the continuous electron density at each disordered atom. A model with ordered atoms and very large displacement parameters is an alternative interpretation.

Experimental

The title material (Blèriot *et al.*, 2004) was crystallized by vapour diffusion of hexane into a solution in ethyl acetate to give small chunky crystals.

Crystal data

$C_{11}H_{13}N_2O_5$
$M_r = 253.24$
Monoclinic, $P2_1$
$a = 11.100$ (2) Å
$b = 7.994$ (2) Å
$c = 13.895$ (2) Å
$\beta = 93.00$ (2)°
$V = 1231.3$ (4) Å^3
$Z = 4$
$D_x = 1.366$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5932 reflections
$\theta = 2.0$–26.6°
$\mu = 0.11$ mm^{-1}
$T = 293$ K
Block, colourless
0.10 × 0.10 × 0.10 mm

Data collection

Nonius DIP2000 diffractometer
ω scans
Absorption correction: none
5932 measured reflections
2417 independent reflections
2404 reflections with $I > -3\sigma(I)$
$R_{int} = 0.035$
$\theta_{max} = 26.6°$
$h = -13 \rightarrow 13$
$k = -8 \rightarrow 8$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma F^2)] = 0.073$
$wR(F^2) = 0.139$
$S = 1.07$
2404 reflections
338 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F) + 0.034P^2 + 0.093P]$, where $P = [\max(F_o^2,0) + 2F_c^2]/3$
$(\Delta/\sigma)_{max} = 0.004$
$\Delta\rho_{max} = 0.39$ e Å^{-3}
$\Delta\rho_{min} = -0.37$ e Å^{-3}

The data were collected on a single-axis image plate diffractometer, leading to missing reflections in the cusp volume. In the absence of significant anomalous scattering effects, Friedel pairs were merged. The absolute configuration was assigned on the basis of the known configuration of the starting material. Part of one molecule was modelled with disordered atoms (Fig. 3). The occupancies of the

two disorder components were refined to 0.566 (13) and 0.434 (13). Anisotropic displacement parameters were constrained to be equal for corresponding atoms, and restraints were applied to the geometry of the disordered portions to keep them similar to the ordered molecules. Fig. 4 is a part of the F_o electron density map in the region of the disordered atoms. Attempts to model the 'split' atoms with electron density distributed along a line (Schröder *et al.*, 2004) led to slightly higher R values, possibly because the disorder trajectory is actually curved. It appears that both the split-atom and a very large displacement model would fit equally well. All H atoms were found in difference density syntheses. They were initially refined with soft geometry restraints to regularize their geometry (bond lengths to accepted values, angles either set by symmetry or to accepted values), after which they were refined with riding constraints only. C—H distances are all close to 0.98 Å, N—H distances are 0.83–1.09 Å, and $U_{iso}(H) = 1.2U_{eq}$(carrier atom).

Data collection: *XPRESS* (MacScience, 1989); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SIR*92 (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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(2*S*,3*R*,4*R*,5S)-3,4-*O*-isopropylidene-2-methyl-6,9-diazo-1-oxaspiro -[4.5]-decane-7,10-dione

Crystal data

$C_{11}H_{13}N_2O_5$
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Monoclinic, $P2_1$
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$c = 13.895$ (2) Å
$\beta = 93.00$ (2)°
$V = 1231.3$ (4) Å^3
$Z = 4$
$F(000) = 532$
$D_x = 1.366$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å
Cell parameters from 5932 reflections
$\theta = 2.0$–26.6°
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Block, colourless
0.10 × 0.10 × 0.10 mm

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Nonius DIP2000 diffractometer
Graphite monochromator
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5932 measured reflections
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$\theta_{max} = 26.6°$, $\theta_{min} = 1.5°$
$h = -13 \rightarrow 13$
$k = -8 \rightarrow 8$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.073$
$wR(F^2) = 0.139$
$S = 1.07$
2404 reflections
338 parameters
97 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: difference Fourier map
H-atom parameters constrained
$w = 1/[\sigma^2(F) + 0.034 + 0.093P]$, where $P = (\max(F_o^2,0) + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.004$
$\Delta\rho_{max} = 0.39$ e Å^{-3}
$\Delta\rho_{min} = -0.37$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C10	0.6248 (4)	0.2082 (9)	0.6666 (4)	0.0158	
C12	0.7226 (5)	0.2036 (8)	0.5118 (4)	0.0149	
C13	0.6028 (5)	0.1630 (8)	0.4622 (4)	0.0188	
C15	0.5042 (4)	0.2440 (8)	0.6119 (4)	0.0150	
C17	0.7048 (5)	0.2288 (9)	0.8220 (4)	0.0189	

C18	0.6682 (5)	0.0470 (8)	0.8120 (4)	0.0187
C19	0.6345 (5)	0.0261 (8)	0.7049 (4)	0.0151
C21	0.4913 (5)	−0.0993 (8)	0.7964 (4)	0.0202
C25	0.6786 (6)	0.3126 (10)	0.9176 (4)	0.0271
C26	0.3590 (6)	−0.0663 (10)	0.8065 (5)	0.0324
C27	0.5286 (6)	−0.2786 (9)	0.8189 (5)	0.0292
C30	0.0611 (4)	0.3067 (8)	0.6517 (4)	0.0126
C32	0.1914 (5)	0.2561 (7)	0.5182 (3)	0.0127
C33	0.1988 (5)	0.0793 (8)	0.5485 (4)	0.0188
C35	0.0240 (5)	0.1234 (8)	0.6505 (4)	0.0140
C37	−0.0066 (5)	0.5598 (8)	0.7108 (4)	0.0168
C45	−0.1133 (6)	0.6385 (8)	0.7556 (4)	0.0224
N11	0.7240 (4)	0.2443 (7)	0.6038 (3)	0.0132
N14	0.5011 (4)	0.2180 (7)	0.5170 (3)	0.0163
N31	0.1111 (4)	0.3555 (6)	0.5609 (3)	0.0125
N34	0.0956 (4)	0.0230 (7)	0.6009 (3)	0.0162
O16	0.6352 (3)	0.3146 (6)	0.7470 (2)	0.0164
O20	0.5211 (3)	−0.0578 (6)	0.7007 (3)	0.0197
O22	0.5585 (4)	0.0154 (6)	0.8567 (3)	0.0221
O23	0.8155 (3)	0.2001 (6)	0.4644 (3)	0.0165
O24	0.4178 (3)	0.2948 (6)	0.6545 (3)	0.0209
O36	−0.0433 (3)	0.4043 (5)	0.6651 (3)	0.0153
O43	0.2563 (3)	0.3095 (6)	0.4554 (3)	0.0165
O44	−0.0638 (3)	0.0743 (6)	0.6916 (3)	0.0179
H11	0.7983	0.2535	0.6409	0.0378*
H14	0.4185	0.2419	0.4732	0.0493*
H31	0.1209	0.4620	0.5495	0.0144*
H34	0.0705	−0.0735	0.5925	0.0424*
H171	0.7908	0.2385	0.8093	0.0228*
H131	0.5981	0.0415	0.4530	0.0224*
H132	0.5983	0.2157	0.3983	0.0218*
H181	0.7320	−0.0309	0.8332	0.0220*
H191	0.6946	−0.0392	0.6718	0.0179*
H251	0.7070	0.4285	0.9164	0.0330*
H252	0.7245	0.2550	0.9701	0.0330*
H253	0.5939	0.3132	0.9344	0.0330*
H261	0.3424	0.0512	0.7902	0.0382*
H262	0.3374	−0.0888	0.8731	0.0377*
H263	0.3126	−0.1393	0.7613	0.0378*
H271	0.6151	−0.2921	0.8095	0.0350*
H272	0.5117	−0.3033	0.8861	0.0348*
H273	0.4822	−0.3517	0.7740	0.0352*
H331	0.2718	0.0649	0.5904	0.0241*
H332	0.2044	0.0096	0.4909	0.0241*
H371	0.0215	0.6362	0.6617	0.0210*
H381	0.1601	0.6019	0.7824	0.0391*
H391	0.2333	0.3635	0.7183	0.0178*
H451	−0.1801	0.6539	0.7077	0.0269*

H452	−0.0900	0.7472	0.7830	0.0272*	
H453	−0.1388	0.5621	0.8055	0.0273*	
C38	0.0997 (6)	0.5125 (9)	0.7776 (4)	0.0330	
C39	0.1502 (5)	0.3509 (8)	0.7378 (4)	0.0156	
O40	0.1423 (4)	0.2319 (6)	0.8117 (3)	0.0271	
O420	0.0403 (11)	0.4453 (15)	0.8612 (7)	0.0390	0.434 (13)
O421	0.0830 (10)	0.4840 (12)	0.8745 (5)	0.0390	0.566 (13)
C410	0.1090 (11)	0.3046 (13)	0.8960 (6)	0.0350	0.434 (13)
C411	0.1417 (9)	0.3292 (11)	0.8994 (5)	0.0350	0.566 (13)
C460	0.0306 (17)	0.178 (3)	0.9470 (12)	0.0468	0.434 (13)
C461	0.0664 (13)	0.244 (2)	0.9742 (10)	0.0468	0.566 (13)
C470	0.2264 (17)	0.358 (2)	0.9576 (12)	0.0387	0.434 (13)
C471	0.2730 (13)	0.3647 (19)	0.9350 (9)	0.0387	0.566 (13)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C10	0.010 (2)	0.028 (4)	0.010 (2)	0.002 (2)	0.0035 (19)	0.001 (2)
C12	0.017 (3)	0.008 (3)	0.019 (3)	0.002 (2)	0.002 (2)	0.001 (2)
C13	0.015 (3)	0.024 (4)	0.018 (3)	0.003 (2)	0.004 (2)	−0.003 (2)
C15	0.009 (3)	0.015 (3)	0.020 (3)	0.002 (2)	0.004 (2)	0.005 (2)
C17	0.019 (3)	0.025 (4)	0.012 (3)	0.004 (3)	−0.002 (2)	−0.002 (2)
C18	0.021 (3)	0.019 (4)	0.017 (3)	0.010 (2)	0.000 (2)	0.003 (2)
C19	0.011 (3)	0.017 (4)	0.017 (3)	0.003 (2)	0.003 (2)	0.002 (2)
C21	0.021 (3)	0.021 (4)	0.018 (3)	0.002 (3)	0.002 (2)	0.003 (3)
C25	0.039 (4)	0.028 (4)	0.014 (3)	0.001 (3)	0.000 (2)	−0.007 (3)
C26	0.024 (3)	0.037 (5)	0.037 (4)	−0.001 (3)	0.009 (3)	0.009 (3)
C27	0.039 (4)	0.019 (4)	0.029 (3)	0.003 (3)	0.003 (3)	0.007 (3)
C30	0.010 (2)	0.016 (3)	0.012 (2)	0.003 (2)	0.0045 (19)	−0.001 (2)
C32	0.016 (3)	0.015 (3)	0.006 (2)	0.001 (2)	−0.0033 (19)	0.003 (2)
C33	0.021 (3)	0.018 (4)	0.019 (3)	0.003 (2)	0.009 (2)	0.000 (2)
C35	0.012 (3)	0.017 (3)	0.012 (3)	0.002 (2)	−0.003 (2)	0.000 (2)
C37	0.018 (3)	0.014 (3)	0.018 (3)	−0.002 (2)	0.003 (2)	−0.006 (2)
C45	0.034 (3)	0.015 (4)	0.019 (3)	0.011 (3)	0.010 (2)	−0.001 (2)
N11	0.008 (2)	0.018 (3)	0.013 (2)	0.0028 (18)	0.0010 (16)	0.0035 (19)
N14	0.011 (2)	0.021 (3)	0.018 (2)	0.002 (2)	0.0043 (17)	0.004 (2)
N31	0.018 (2)	0.005 (3)	0.016 (2)	−0.0001 (17)	0.0047 (17)	0.0040 (17)
N34	0.015 (2)	0.010 (3)	0.024 (3)	−0.0036 (19)	0.0033 (19)	0.000 (2)
O16	0.021 (2)	0.016 (2)	0.0118 (18)	0.0034 (18)	0.0009 (15)	−0.0041 (17)
O20	0.021 (2)	0.020 (3)	0.019 (2)	−0.0065 (17)	0.0038 (16)	0.0026 (17)
O22	0.026 (2)	0.026 (3)	0.016 (2)	0.0033 (19)	0.0108 (17)	0.0024 (18)
O23	0.0139 (19)	0.018 (3)	0.018 (2)	0.0006 (16)	0.0085 (15)	0.0016 (17)
O24	0.0142 (19)	0.033 (3)	0.0162 (19)	0.0074 (18)	0.0054 (15)	0.0043 (18)
O36	0.0117 (19)	0.015 (2)	0.019 (2)	0.0030 (16)	0.0016 (14)	−0.0025 (16)
O43	0.0158 (19)	0.020 (2)	0.0137 (18)	0.0020 (17)	0.0039 (14)	0.0025 (17)
O44	0.014 (2)	0.019 (3)	0.020 (2)	−0.0036 (16)	0.0026 (15)	0.0028 (17)
C38	0.036 (3)	0.035 (4)	0.027 (3)	0.012 (3)	−0.010 (3)	−0.014 (3)
C39	0.010 (3)	0.019 (4)	0.018 (3)	−0.001 (2)	−0.002 (2)	−0.003 (2)

O40	0.034 (2)	0.032 (3)	0.0145 (19)	−0.004 (2)	−0.0063 (16)	0.0070 (19)
O420	0.041 (4)	0.059 (4)	0.015 (3)	0.027 (3)	−0.016 (3)	−0.015 (3)
O421	0.041 (4)	0.059 (4)	0.015 (3)	0.027 (3)	−0.016 (3)	−0.015 (3)
C410	0.029 (4)	0.059 (4)	0.016 (2)	0.016 (3)	−0.006 (3)	0.001 (3)
C411	0.029 (4)	0.059 (4)	0.016 (2)	0.016 (3)	−0.006 (3)	0.001 (3)
C460	0.037 (6)	0.080 (7)	0.023 (5)	0.005 (5)	−0.004 (4)	0.017 (5)
C461	0.037 (6)	0.080 (7)	0.023 (5)	0.005 (5)	−0.004 (4)	0.017 (5)
C470	0.031 (6)	0.064 (6)	0.020 (5)	0.014 (5)	−0.011 (4)	−0.012 (4)
C471	0.031 (6)	0.064 (6)	0.020 (5)	0.014 (5)	−0.011 (4)	−0.012 (4)

Geometric parameters (Å, º)

C10—C15	1.532 (7)	C30—O36	1.417 (6)
C10—C19	1.551 (9)	C30—C39	1.554 (7)
C10—N11	1.469 (6)	C32—C33	1.476 (8)
C10—O16	1.404 (7)	C32—N31	1.354 (7)
C12—C13	1.501 (8)	C32—O43	1.237 (6)
C12—N11	1.319 (7)	C33—N34	1.459 (7)
C12—O23	1.253 (6)	C33—H331	0.980
C13—N14	1.462 (7)	C33—H332	0.980
C13—H131	0.980	C35—N34	1.345 (7)
C13—H132	0.982	C35—O44	1.219 (6)
C15—N14	1.334 (7)	C37—C45	1.504 (7)
C15—O24	1.222 (6)	C37—O36	1.445 (7)
C17—C18	1.514 (9)	C37—H371	0.979
C17—C25	1.529 (8)	C37—C38	1.511 (8)
C17—O16	1.439 (7)	C45—H451	0.978
C17—H171	0.982	C45—H452	0.978
C18—C19	1.525 (8)	C45—H453	0.977
C18—O22	1.419 (7)	N11—H11	0.952
C18—H181	0.977	N14—H14	1.091
C19—O20	1.425 (7)	N31—H31	0.874
C19—H191	0.981	N34—H34	0.827
C21—C26	1.506 (8)	H381—C38	0.980
C21—C27	1.520 (9)	H391—C39	0.980
C21—O20	1.426 (6)	C38—C39	1.523 (8)
C21—O22	1.426 (7)	C38—O421	1.387 (8)
C25—H251	0.979	C38—C39	1.523 (8)
C25—H252	0.983	C38—O420	1.467 (9)
C25—H253	0.980	C39—O40	1.406 (7)
C26—H261	0.981	O40—C411	1.446 (7)
C26—H262	0.985	O40—C410	1.376 (8)
C26—H263	0.983	O420—C410	1.429 (9)
C27—H271	0.982	O421—C411	1.432 (8)
C27—H272	0.982	C410—C460	1.53 (2)
C27—H273	0.981	C410—C470	1.58 (2)
C30—C35	1.522 (9)	C411—C461	1.527 (17)
C30—N31	1.457 (6)	C411—C471	1.542 (16)

C15—C10—C19	112.9 (5)	C32—C33—H331	108.2
C15—C10—N11	109.3 (4)	N34—C33—H331	108.2
C19—C10—N11	110.3 (4)	C32—C33—H332	108.4
C15—C10—O16	108.4 (5)	N34—C33—H332	108.4
C19—C10—O16	107.2 (4)	H331—C33—H332	109.4
N11—C10—O16	108.7 (5)	C30—C35—N34	114.4 (5)
C13—C12—N11	117.6 (5)	C30—C35—O44	121.9 (5)
C13—C12—O23	119.2 (5)	N34—C35—O44	123.8 (6)
N11—C12—O23	123.2 (5)	C45—C37—O36	109.4 (5)
C12—C13—N14	112.7 (5)	C45—C37—H371	108.3
C12—C13—H131	108.3	O36—C37—H371	108.8
N14—C13—H131	109.1	C45—C37—C38	117.2 (5)
C12—C13—H132	108.9	O36—C37—C38	104.3 (5)
N14—C13—H132	109.7	H371—C37—C38	108.4
H131—C13—H132	107.9	C37—C45—H451	110.9
C10—C15—N14	116.0 (4)	C37—C45—H452	109.5
C10—C15—O24	120.6 (5)	H451—C45—H452	109.2
N14—C15—O24	123.4 (5)	C37—C45—H453	107.1
C18—C17—C25	116.0 (5)	H451—C45—H453	108.9
C18—C17—O16	105.0 (5)	H452—C45—H453	111.0
C25—C17—O16	107.5 (5)	C10—N11—C12	123.8 (5)
C18—C17—H171	108.5	C10—N11—H11	110.4
C25—C17—H171	110.6	C12—N11—H11	120.6
O16—C17—H171	108.7	C13—N14—C15	125.6 (5)
C17—C18—C19	104.2 (5)	C13—N14—H14	114.2
C17—C18—O22	111.4 (5)	C15—N14—H14	120.1
C19—C18—O22	103.7 (4)	C30—N31—C32	120.8 (5)
C17—C18—H181	113.3	C30—N31—H31	118.3
C19—C18—H181	111.1	C32—N31—H31	113.5
O22—C18—H181	112.3	C33—N34—C35	124.9 (5)
C10—C19—C18	103.9 (5)	C33—N34—H34	119.0
C10—C19—O20	112.4 (4)	C35—N34—H34	115.1
C18—C19—O20	105.2 (4)	C17—O16—C10	107.8 (4)
C10—C19—H191	112.0	C21—O20—C19	108.6 (4)
C18—C19—H191	112.2	C21—O22—C18	106.9 (4)
O20—C19—H191	110.5	C37—O36—C30	108.6 (4)
C26—C21—C27	113.7 (5)	C37—C38—H381	111.6
C26—C21—O20	108.7 (5)	C37—C38—C39	106.2 (5)
C27—C21—O20	109.8 (5)	H381—C38—C39	112.3
C26—C21—O22	108.3 (5)	C37—C38—O421	119.8 (6)
C27—C21—O22	110.9 (5)	H381—C38—O421	100.3
O20—C21—O22	105.0 (5)	C39—C38—O421	106.4 (6)
C17—C25—H251	108.9	C37—C38—H381	111.6
C17—C25—H252	108.9	C37—C38—C39	106.2 (5)
H251—C25—H252	107.5	H381—C38—C39	112.3
C17—C25—H253	115.9	C37—C38—O420	102.0 (6)
H251—C25—H253	108.2	H381—C38—O420	123.2

H252—C25—H253	106.9	C39—C38—O420	99.6 (6)
C21—C26—H261	108.5	C30—C39—C38	103.9 (5)
C21—C26—H262	109.9	C30—C39—H391	112.3
H261—C26—H262	109.8	C38—C39—H391	112.5
C21—C26—H263	108.4	C30—C39—O40	110.3 (4)
H261—C26—H263	109.6	C38—C39—O40	105.7 (4)
H262—C26—H263	110.2	H391—C39—O40	111.5
C21—C27—H271	109.4	C39—O40—C411	104.7 (5)
C21—C27—H272	108.8	C39—O40—C410	111.4 (5)
H271—C27—H272	110.0	C38—O420—C410	107.6 (6)
C21—C27—H273	107.6	C38—O421—C411	107.1 (6)
H271—C27—H273	109.4	O420—C410—O40	102.0 (6)
H272—C27—H273	111.4	O420—C410—C460	111.7 (7)
C35—C30—N31	111.3 (4)	O40—C410—C460	107.5 (8)
C35—C30—O36	108.0 (4)	O420—C410—C470	112.3 (7)
N31—C30—O36	108.5 (5)	O40—C410—C470	108.9 (7)
C35—C30—C39	112.9 (5)	C460—C410—C470	113.6 (7)
N31—C30—C39	110.5 (4)	O40—C411—O421	106.5 (5)
O36—C30—C39	105.4 (4)	O40—C411—C461	111.2 (7)
C33—C32—N31	117.7 (5)	O421—C411—C461	106.9 (7)
C33—C32—O43	120.4 (5)	O40—C411—C471	108.8 (6)
N31—C32—O43	121.9 (5)	O421—C411—C471	109.2 (7)
C32—C33—N34	113.9 (5)	C461—C411—C471	113.9 (6)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N11—H11···O36^{i}	0.95	2.14	2.967 (6)	144
N11—H11···O44^{i}	0.95	2.19	2.930 (6)	134
N14—H14···O43	1.09	1.88	2.900 (6)	153
N31—H31···O23^{ii}	0.87	2.04	2.899 (7)	166

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