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(*S*)-3-Dimethylamino-2-{(4*S*,5*R*)-5-[(*R*)-2,2-dimethyl-1,3-dioxolan-4-yl]-2,2-dimethyl-1,3-dioxolan-4-yl}-2-hydroxypropanoic acid

Sarah F. Jenkinson,[a]* David J. Hotchkiss,[a] Andrew R. Cowley,[b] George W. J. Fleet[a] and David J. Watkin[b]

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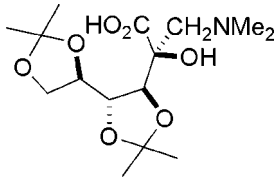
Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.003 Å; R factor = 0.032; wR factor = 0.033; data-to-parameter ratio = 7.8.

The Kiliani reaction on 1-deoxy-(*N*,*N*-dimethylamino)-D-fructose, itself readily available from reaction of dimethylamine and D-glucose, proceeded to give access to the title β-sugar amino acid, $C_{15}H_{27}NO_7$. X-ray crystallography determined the stereochemistry at the newly formed chiral center. There are two molecules in the asymmetric unit; they are related by a pseudo-twofold rotation axis and have very similar geometries, differing only in the conformation of one of the acetonide rings. All the acetonide rings adopt envelope conformations; the flap atom is oxygen in three of the rings, but carbon in one of them. There are two strong hydrogen bonds between the two independent molecules, and further weak hydrogen bonds link the molecules to form infinite chains running parallel to the *a* axis.

Related literature

For related literature see: Risseeuw *et al.* (2007); Hotchkiss *et al.* (2004, 2008); Soengas *et al.* (2005); Parker *et al.* (2006); Simone *et al.* (2007). For the refinement weighting scheme, see: Prince (1982); Watkin (1994).



Experimental

Crystal data

$C_{15}H_{27}NO_7$
M_r = 333.38
Monoclinic, $P2_1$
a = 5.7881 (2) Å
b = 16.7077 (4) Å
c = 17.8572 (5) Å
β = 99.1141 (8)°
V = 1705.09 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 150 K
0.40 × 0.10 × 0.08 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*DENZO*/*SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.96, T_{max} = 1.01
(expected range = 0.943–0.992)
13496 measured reflections
4000 independent reflections
3474 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.033
S = 1.09
3239 reflections
415 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H21···O109	0.90	1.80	2.664 (3)	161
N102—H1021···O9	0.90	1.81	2.675 (3)	162
O110—H1101···O109^{i}	0.81	2.69	3.348 (3)	140
O10—H101···O11^{i}	0.83	2.50	3.187 (3)	141

Symmetry code: (i) $x+1, y, z$.

Data collection: *COLLECT* (Nonius, 2001); cell refinement: *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO*/*SCALEPACK*; 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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CF2175).

References

Altomare, A., Cascarano, G., Giacovazzo, G., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
Betteridge, P. W., Carruthers, J. R., Cooper, R. I., Prout, K. & Watkin, D. J. (2003). *J. Appl. Cryst.* **36**, 1487.
Hotchkiss, D. J., Jenkinson, S. F. & Fleet, G. W. J. (2008). *Tetrahedron Lett.* In preparation.
Hotchkiss, D. J., Soengas, R., Simone, M. I., van Ameijde, J., Hunter, S., Cowley, A. R. & Fleet, G. W. J. (2004). *Tetrahedron Lett.* **45**, 9461–9464.
Nonius (2001). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Parker, S. G., Watkin, D. J., Simone, M. I. & Fleet, G. W. J. (2006). *Acta Cryst.* E**62**, o3961–o3963.
Prince, E. (1982). *Mathematical Techniques in Crystallography and Materials Science*. New York: Springer.

  doi:10.1107/S1600536807066676

Risseeuw, M. D. P., Overhand, M., Fleet, G. W. J. & Simone, M. I. (2007). *Tetrahedron Asymmetry*, **18**, 2001–2010.
Simone, M., Fleet, G. W. J. & Watkin, D. J. (2007). *Acta Cryst.* E**63**, o799–o801.
Soengas, R., Izumori, K., Simone, M. I., Watkin, D. J., Skytte, U. P., Soetaert, W. & Fleet, G. W. J. (2005). *Tetrahedron Lett.* **46**, 5755–5759.
Watkin, D. (1994). *Acta Cryst.* A**50**, 411–437.
Watkin, D. J., Prout, C. K. & Pearce, L. J. (1996). *CAMERON*. Chemical Crystallography Laboratory, Oxford, England.

supporting information

Acta Cryst. (2008). E**64**, o294–o295 [https://doi.org/10.1107/S1600536807066676]

(*S*)-3-Dimethylamino-2-{(4*S*,5*R*)-5-[(*R*)-2,2-dimethyl-1,3-dioxolan-4-yl]-2,2-dimethyl-1,3-dioxolan-4-yl}-2-hydroxypropanoic acid

Sarah F. Jenkinson, David J. Hotchkiss, Andrew R. Cowley, George W. J. Fleet and David J. Watkin

S1. Comment

Sugar amino acids are a versatile class of conformationally biased building blocks, and have use as both glyco- and peptido-mimetics (Risseeuw *et al.*, 2007). The Kiliani reaction on ketoses has been successfully utilized in the synthesis of branched carbohydrate building blocks (Hotchkiss *et al.*, 2004; Soengas *et al.*, 2005; Parker *et al.*, 2006; Simone *et al.*, 2007) to produce, for example, methyl or hydroxymethyl branched lactones. With Amadori products, 1-amino-1-deoxy-ketoses, as substrates, the Kiliani ascension should provide access to *β*-sugar amino acids. The reaction of 1-deoxy-1-(*N,N*-dimethylamino)-D-fructose, **2**, an Amadori product readily available from D-glucose, **1**, with sodium cyanide in water was found to give, after acetonide protection, the title compound, **3**, as the major product. The stereochemistry at C-2 was unequivocally assigned by X-ray crystallography (Fig. 2) and the absolute stereochemistry was determined by the use of D-glucose as the starting material.

The asymmetric unit contains of two crystallographically distinct molecules which are related by a pseudo-2-fold rotation axis. These are similar in geometry with the exception of one of the isopropylidene rings: in the first molecule the atoms C20, O13, O14 and C28 are approximately coplanar while C21 is displaced from this plane, whereas in the second molecule C120, C121, O113 and C128 are approximately coplanar while O114 is displaced. The r.m.s. bond length deviation for the two molecules, excluding hydrogen atoms, is 0.007 Å.

Hydrogen bonding links molecules to form infinite chains running parallel to the crystallographic *a* axis (Fig. 3). There are two weak hydrogen bonds between the layers and two strong hydrogen bonds linking the two molecules in the asymmetric unit (Fig. 4).

S2. Experimental

The title compound was prepared as described by Hotchkiss *et al.* (2008) and shown in the reaction scheme of Fig. 1, and was recrystallized from ethyl acetate. m.p.: 453 K decomposed; $[\alpha]_D^{23}$ +19.3 (*c*, 1.0 in water).

S3. Refinement

In the absence of significant anomalous scattering, Friedel pairs were merged and the absolute configuration was assigned from the starting material. The refinement, on F values, used only data for which $F^2 > 3\sigma(F^2)$.

The H atoms were all located in a difference map, but those attached to carbon atoms were repositioned geometrically. The H atoms were initially refined with soft restraints on the bond lengths and angles to regularize their geometry (C—H in the range 0.93–0.98, N—H in the range 0.86–0.89 N—H to 0.86 O—H = 0.82 Å) and U_{iso}(H) (in the range 1.2–1.5 times U_{eq} of the parent atom), after which the positions were refined with riding constraints.

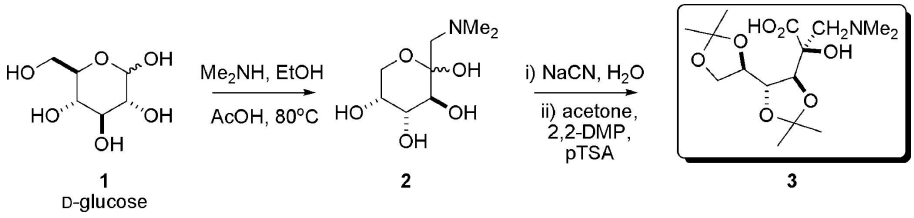


Figure 1

The synthesis of the title compound.

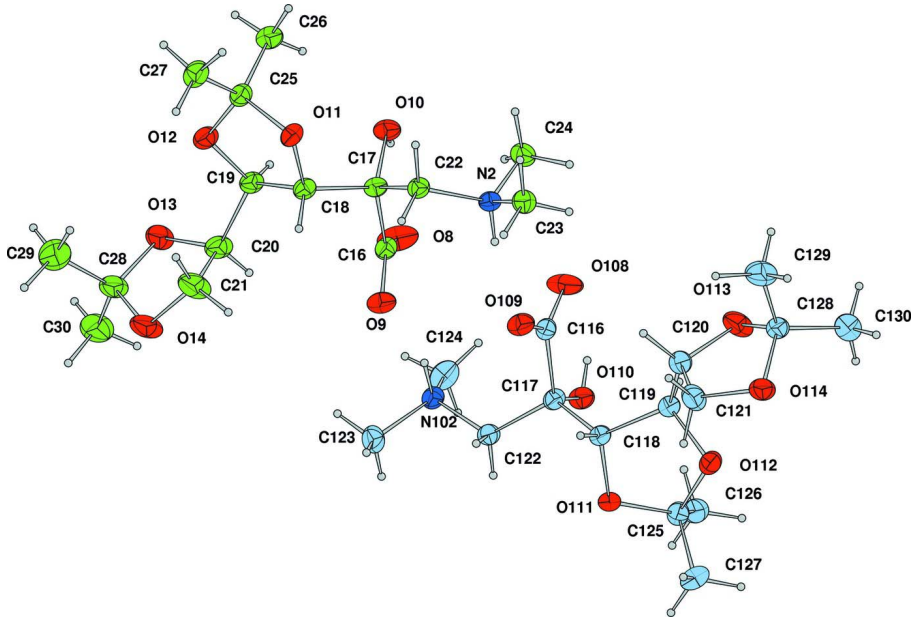


Figure 2

The title compound with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitary radius.

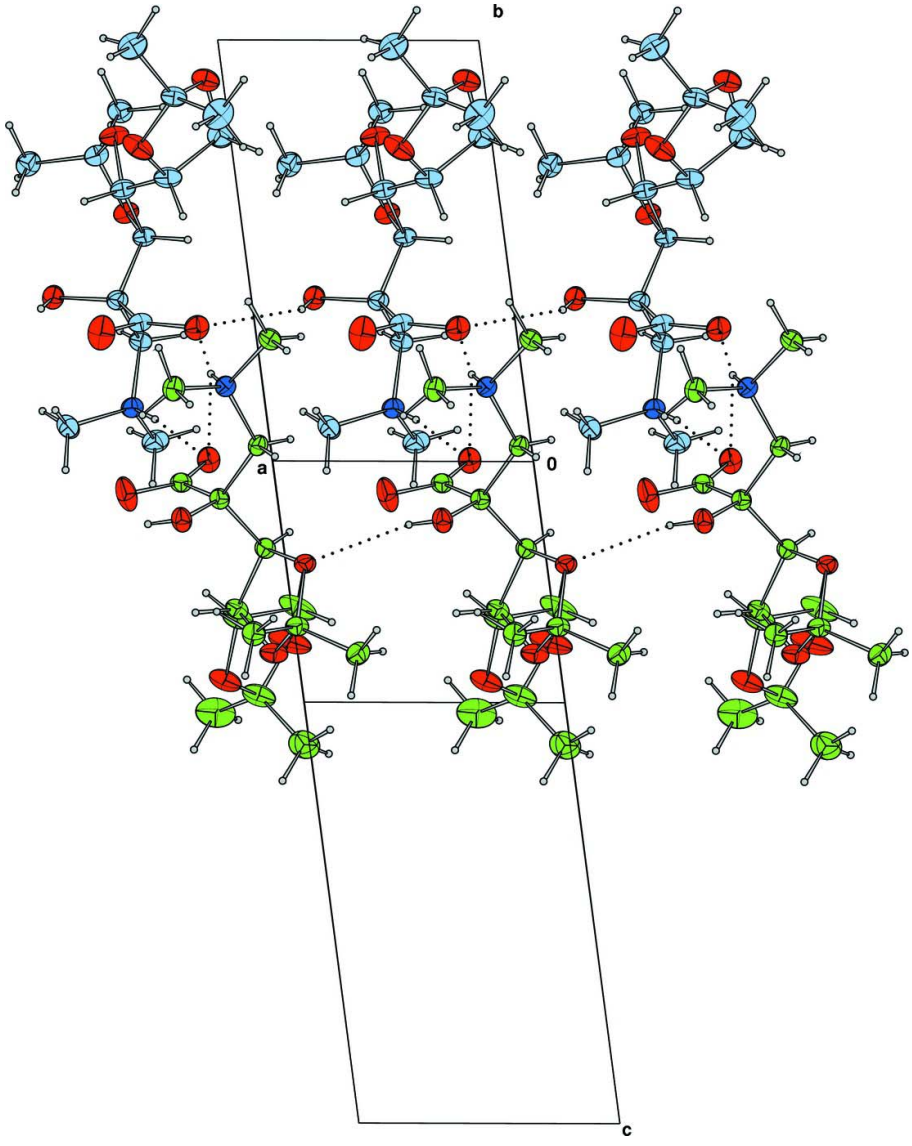


Figure 3

The packing diagram for the molecule showing the infinite hydrogen bonded chains of molecules lying parallel to the *a* axis.

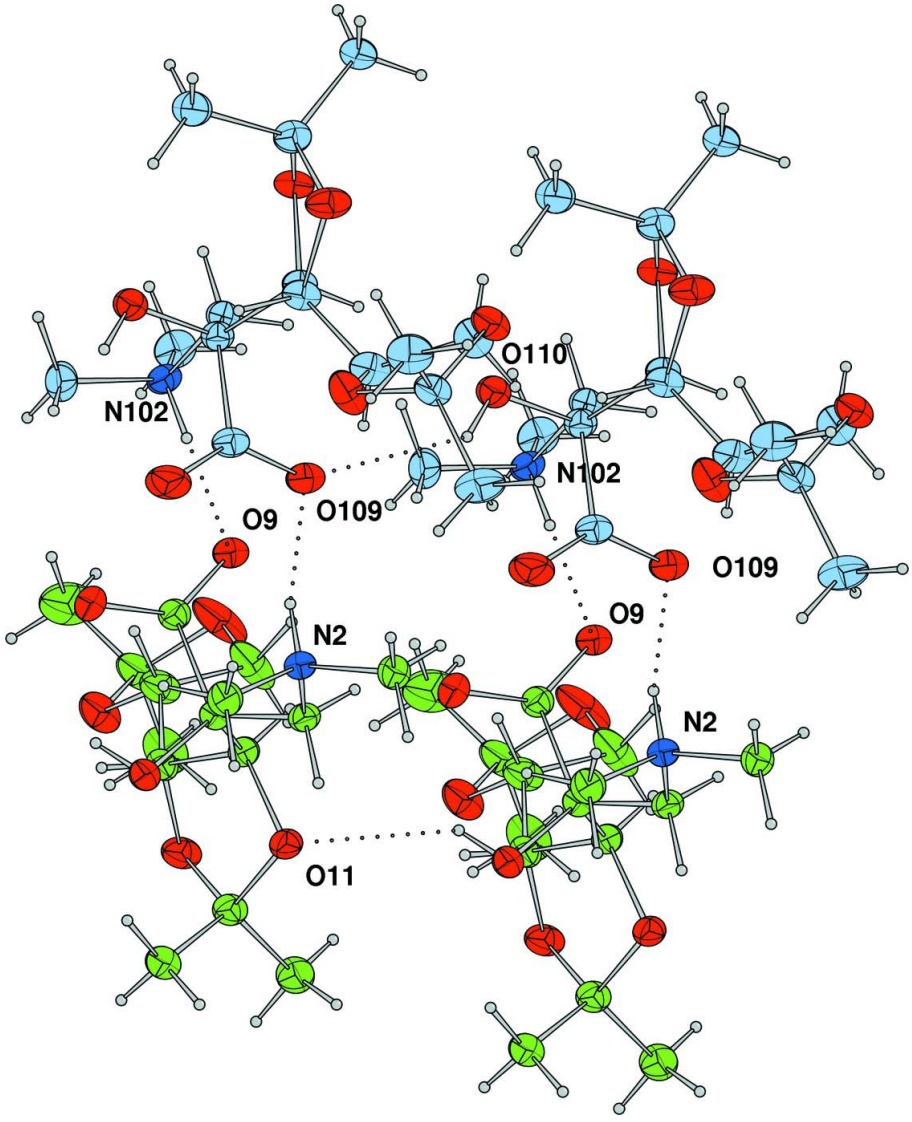


Figure 4

The two molecules of the asymmetric unit are linked by two strong hydrogen bonds, O9···H21—N2 and O109···H1021-N102 and these dimeric units are linked by weak hydrogen bonds, O11···H101—O10 and O109···H1101-O110, to form a hydrogen bonded column.

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$c = 17.8572\ (5)$ Å
$\beta = 99.1141\ (8)°$
$V = 1705.09\ (9)$ Å^3
$Z = 4$
$F(000) = 720$
$D_x = 1.299$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 13496 reflections
$\theta = 5–28°$

μ = 0.10 mm^{-1}
T = 150 K

Fragment, colourless
0.40 × 0.10 × 0.08 mm

Data collection

Nonius KappaCCD diffractometer
Graphite monochromator
ω scans
Absorption correction: multi-scan (*DENZO*/*SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.96, T_{max} = 1.01

13496 measured reflections
4000 independent reflections
3474 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 27.5°, θ_{min} = 5.1°
h = −7→7
k = −20→21
l = −23→23

Refinement

Refinement on F
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.033
S = 1.09
3239 reflections
415 parameters
1 restraint
Primary atom site location: structure-invariant direct methods

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = [1-(F_o-F_c)^2/36\sigma^2(F)]^2/[0.462T_0(x) + 0.141T_1(x) + 0.209T_2(x)]$ where T_i are Chebychev polynomials and x = F_c/F_{max} (*P*rince, 1982; Watkin, 1994)
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C116	0.4385 (4)	0.61358 (13)	0.18160 (11)	0.0229
C117	0.5150 (3)	0.70044 (12)	0.20298 (11)	0.0214
C118	0.3803 (4)	0.75579 (12)	0.14222 (11)	0.0215
C119	0.4380 (4)	0.74189 (12)	0.06211 (12)	0.0245
C120	0.2595 (4)	0.69110 (13)	0.01196 (12)	0.0281
C121	0.0366 (4)	0.73492 (15)	−0.02378 (12)	0.0309
O108	0.5915 (3)	0.56731 (10)	0.16763 (11)	0.0411
O109	0.2243 (3)	0.59883 (9)	0.17987 (9)	0.0291
O110	0.7593 (2)	0.70916 (9)	0.20458 (9)	0.0257
O111	0.4370 (3)	0.83813 (9)	0.15861 (8)	0.0247
O112	0.4449 (3)	0.82134 (10)	0.03206 (8)	0.0302
O113	0.3587 (3)	0.66311 (11)	−0.05185 (9)	0.0356
O114	0.0662 (3)	0.74545 (10)	−0.10091 (9)	0.0292
C122	0.4465 (4)	0.72297 (12)	0.27944 (11)	0.0222
N102	0.5117 (3)	0.66327 (11)	0.34183 (9)	0.0225
C123	0.4395 (4)	0.69477 (15)	0.41273 (12)	0.0308
C124	0.7666 (4)	0.64198 (15)	0.35724 (14)	0.0332
C125	0.5228 (4)	0.87319 (13)	0.09473 (12)	0.0254
C126	0.7880 (4)	0.87748 (14)	0.11015 (14)	0.0305
C127	0.4103 (4)	0.95410 (13)	0.07883 (14)	0.0308
C128	0.1957 (4)	0.67786 (13)	−0.11891 (12)	0.0250
C130	0.3287 (5)	0.69882 (16)	−0.18191 (15)	0.0372
C129	0.0342 (5)	0.60661 (16)	−0.13784 (16)	0.0413

C16	0.3613 (4)	0.46972 (13)	0.33186 (11)	0.0221
C17	0.2219 (3)	0.39235 (12)	0.30686 (11)	0.0207
C18	0.0715 (3)	0.37226 (12)	0.36854 (11)	0.0214
C19	0.2109 (4)	0.35053 (13)	0.44690 (12)	0.0232
C20	0.2117 (4)	0.41812 (14)	0.50374 (13)	0.0321
C21	−0.0232 (5)	0.45509 (17)	0.51083 (14)	0.0445
O8	0.5763 (3)	0.46309 (11)	0.34502 (12)	0.0422
O9	0.2444 (3)	0.53219 (9)	0.33683 (9)	0.0284
O10	0.3781 (2)	0.32903 (9)	0.29844 (9)	0.0251
O11	−0.0735 (2)	0.30306 (9)	0.34789 (8)	0.0232
O12	0.0913 (3)	0.28280 (9)	0.47036 (9)	0.0286
O13	0.2906 (3)	0.38992 (10)	0.57957 (8)	0.0345
O14	0.0240 (4)	0.48999 (11)	0.58459 (10)	0.0500
C22	0.0472 (3)	0.40421 (12)	0.23363 (11)	0.0210
N2	0.1429 (3)	0.44201 (10)	0.16870 (9)	0.0204
C23	−0.0510 (4)	0.45073 (13)	0.10267 (12)	0.0247
C24	0.3421 (4)	0.39788 (14)	0.14406 (13)	0.0268
C25	−0.0112 (4)	0.24115 (13)	0.40337 (12)	0.0239
C26	0.1574 (4)	0.18220 (13)	0.37676 (13)	0.0283
C27	−0.2313 (4)	0.20088 (15)	0.41883 (14)	0.0309
C28	0.1693 (5)	0.43430 (14)	0.63045 (13)	0.0353
C29	0.0209 (5)	0.37758 (18)	0.66863 (17)	0.0465
C30	0.3447 (6)	0.48027 (18)	0.68589 (16)	0.0518
H1181	0.2144	0.7487	0.1423	0.0252*
H1191	0.5917	0.7168	0.0646	0.0293*
H1201	0.2207	0.6450	0.0408	0.0337*
H1211	0.0253	0.7867	−0.0002	0.0376*
H1212	−0.1033	0.7032	−0.0199	0.0368*
H1221	0.5202	0.7735	0.2962	0.0258*
H1222	0.2768	0.7284	0.2732	0.0254*
H1231	0.4711	0.6537	0.4509	0.0459*
H1233	0.5303	0.7422	0.4267	0.0453*
H1232	0.2760	0.7072	0.4021	0.0450*
H1241	0.7978	0.6080	0.4012	0.0490*
H1243	0.8570	0.6911	0.3649	0.0489*
H1242	0.8073	0.6134	0.3141	0.0483*
H1261	0.8415	0.9000	0.0662	0.0460*
H1262	0.8342	0.9121	0.1535	0.0454*
H1263	0.8507	0.8252	0.1202	0.0451*
H1271	0.4619	0.9783	0.0352	0.0453*
H1272	0.4513	0.9888	0.1220	0.0449*
H1273	0.2425	0.9481	0.0687	0.0443*
H1301	0.2217	0.7150	−0.2268	0.0566*
H1302	0.4370	0.7422	−0.1661	0.0565*
H1303	0.4179	0.6536	−0.1945	0.0565*
H1291	−0.0809	0.6189	−0.1822	0.0617*
H1292	−0.0451	0.5955	−0.0955	0.0615*
H1293	0.1258	0.5604	−0.1487	0.0622*

H181	−0.0296	0.4172	0.3728	0.0237*
H191	0.3710	0.3357	0.4429	0.0264*
H201	0.3210	0.4583	0.4911	0.0357*
H211	−0.0701	0.4960	0.4730	0.0524*
H212	−0.1466	0.4145	0.5069	0.0521*
H222	−0.0791	0.4377	0.2456	0.0239*
H221	−0.0117	0.3514	0.2167	0.0247*
H231	0.0118	0.4763	0.0624	0.0361*
H232	−0.1710	0.4831	0.1193	0.0354*
H233	−0.1099	0.3991	0.0872	0.0355*
H241	0.3790	0.4232	0.0985	0.0408*
H242	0.4751	0.3989	0.1848	0.0403*
H243	0.2889	0.3436	0.1335	0.0399*
H262	0.2116	0.1456	0.4172	0.0424*
H261	0.2886	0.2090	0.3608	0.0418*
H263	0.0743	0.1521	0.3350	0.0414*
H272	−0.1849	0.1570	0.4539	0.0465*
H271	−0.3277	0.2390	0.4395	0.0470*
H273	−0.3152	0.1817	0.3708	0.0462*
H292	−0.0685	0.4070	0.7011	0.0699*
H291	0.1215	0.3398	0.6993	0.0692*
H293	−0.0862	0.3518	0.6295	0.0696*
H302	0.2616	0.5129	0.7176	0.0760*
H301	0.4457	0.4426	0.7178	0.0759*
H303	0.4375	0.5141	0.6576	0.0749*
H21	0.1925	0.4920	0.1815	0.0296*
H1021	0.4300	0.6182	0.3304	0.0330*
H1101	0.8115	0.6662	0.1944	0.0382*
H101	0.5088	0.3466	0.3164	0.0387*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C116	0.0305 (11)	0.0199 (9)	0.0184 (9)	−0.0035 (8)	0.0043 (8)	0.0006 (8)
C117	0.0232 (9)	0.0189 (9)	0.0218 (9)	−0.0018 (8)	0.0026 (7)	0.0014 (8)
C118	0.0266 (10)	0.0171 (9)	0.0207 (9)	−0.0018 (8)	0.0038 (7)	0.0003 (7)
C119	0.0334 (11)	0.0191 (10)	0.0216 (10)	−0.0009 (9)	0.0057 (8)	0.0004 (8)
C120	0.0402 (12)	0.0235 (11)	0.0205 (10)	0.0002 (9)	0.0045 (8)	−0.0002 (8)
C121	0.0356 (12)	0.0336 (12)	0.0238 (11)	0.0030 (10)	0.0057 (9)	−0.0032 (9)
O108	0.0392 (9)	0.0231 (8)	0.0630 (12)	0.0017 (7)	0.0145 (8)	−0.0123 (8)
O109	0.0315 (8)	0.0211 (7)	0.0347 (8)	−0.0040 (6)	0.0052 (6)	−0.0009 (6)
O110	0.0237 (7)	0.0214 (7)	0.0329 (8)	0.0007 (6)	0.0066 (6)	0.0003 (6)
O111	0.0337 (8)	0.0163 (7)	0.0252 (7)	−0.0011 (6)	0.0083 (6)	−0.0015 (6)
O112	0.0463 (9)	0.0225 (8)	0.0215 (7)	−0.0075 (7)	0.0049 (6)	0.0010 (6)
O113	0.0433 (9)	0.0360 (9)	0.0246 (8)	0.0158 (8)	−0.0041 (7)	−0.0071 (7)
O114	0.0378 (8)	0.0225 (7)	0.0265 (8)	0.0059 (7)	0.0023 (6)	−0.0008 (6)
C122	0.0258 (10)	0.0191 (9)	0.0211 (10)	0.0003 (8)	0.0019 (8)	0.0001 (8)
N102	0.0253 (8)	0.0209 (8)	0.0205 (8)	−0.0055 (7)	0.0009 (6)	0.0000 (7)

C123	0.0378 (12)	0.0339 (12)	0.0208 (10)	−0.0050 (10)	0.0047 (9)	−0.0014 (9)
C124	0.0248 (11)	0.0348 (12)	0.0375 (12)	0.0002 (9)	−0.0026 (9)	0.0089 (10)
C125	0.0307 (11)	0.0238 (10)	0.0220 (10)	−0.0030 (9)	0.0049 (8)	0.0007 (8)
C126	0.0321 (11)	0.0257 (11)	0.0347 (12)	−0.0004 (9)	0.0084 (9)	0.0003 (9)
C127	0.0312 (11)	0.0213 (10)	0.0384 (12)	−0.0015 (9)	0.0012 (9)	0.0061 (9)
C128	0.0330 (11)	0.0203 (9)	0.0210 (9)	0.0019 (9)	0.0023 (8)	0.0004 (8)
C130	0.0447 (14)	0.0343 (13)	0.0346 (12)	−0.0017 (11)	0.0119 (10)	−0.0031 (10)
C129	0.0507 (15)	0.0291 (12)	0.0448 (14)	−0.0097 (11)	0.0097 (12)	−0.0110 (11)
C16	0.0237 (9)	0.0210 (10)	0.0210 (9)	−0.0016 (8)	0.0015 (7)	0.0022 (7)
C17	0.0197 (9)	0.0168 (9)	0.0253 (10)	0.0013 (7)	0.0029 (7)	0.0006 (7)
C18	0.0212 (9)	0.0196 (9)	0.0227 (9)	−0.0017 (8)	0.0009 (7)	0.0007 (8)
C19	0.0250 (10)	0.0200 (9)	0.0233 (10)	0.0024 (8)	−0.0004 (7)	0.0017 (8)
C20	0.0452 (13)	0.0225 (11)	0.0253 (11)	0.0027 (9)	−0.0044 (9)	0.0000 (9)
C21	0.0644 (17)	0.0374 (14)	0.0273 (12)	0.0278 (13)	−0.0060 (11)	−0.0032 (10)
O8	0.0214 (7)	0.0275 (9)	0.0734 (13)	−0.0021 (7)	−0.0059 (8)	−0.0020 (8)
O9	0.0265 (8)	0.0200 (7)	0.0380 (9)	−0.0021 (6)	0.0034 (6)	−0.0022 (6)
O10	0.0206 (7)	0.0203 (7)	0.0342 (8)	0.0025 (6)	0.0041 (6)	−0.0014 (6)
O11	0.0242 (7)	0.0210 (7)	0.0234 (7)	−0.0037 (6)	0.0011 (5)	0.0042 (6)
O12	0.0394 (9)	0.0208 (7)	0.0239 (7)	−0.0009 (6)	−0.0004 (6)	0.0027 (6)
O13	0.0485 (10)	0.0305 (9)	0.0212 (8)	0.0106 (8)	−0.0046 (7)	−0.0022 (6)
O14	0.0876 (15)	0.0320 (9)	0.0267 (9)	0.0296 (10)	−0.0026 (9)	−0.0032 (8)
C22	0.0207 (9)	0.0189 (9)	0.0237 (10)	−0.0026 (8)	0.0044 (7)	0.0000 (8)
N2	0.0216 (8)	0.0173 (8)	0.0222 (8)	−0.0018 (6)	0.0027 (6)	−0.0013 (7)
C23	0.0254 (10)	0.0253 (10)	0.0222 (10)	0.0005 (8)	0.0005 (7)	−0.0006 (8)
C24	0.0252 (10)	0.0264 (10)	0.0298 (11)	0.0013 (9)	0.0072 (8)	−0.0029 (9)
C25	0.0280 (10)	0.0213 (10)	0.0218 (10)	−0.0016 (8)	0.0020 (8)	0.0028 (8)
C26	0.0275 (10)	0.0226 (10)	0.0348 (12)	0.0018 (8)	0.0050 (9)	0.0009 (9)
C27	0.0295 (11)	0.0295 (11)	0.0350 (12)	−0.0010 (9)	0.0089 (9)	0.0061 (9)
C28	0.0532 (14)	0.0247 (11)	0.0253 (11)	0.0118 (10)	−0.0018 (10)	−0.0021 (9)
C29	0.0488 (15)	0.0429 (15)	0.0475 (15)	0.0105 (13)	0.0072 (12)	0.0063 (13)
C30	0.079 (2)	0.0387 (15)	0.0340 (14)	−0.0050 (15)	−0.0020 (13)	−0.0106 (12)

Geometric parameters (Å, º)

C116—C117	1.547 (3)	C16—C17	1.552 (3)
C116—O108	1.231 (3)	C16—O8	1.234 (3)
C116—O109	1.260 (3)	C16—O9	1.255 (3)
C117—C118	1.540 (3)	C17—C18	1.545 (3)
C117—O110	1.417 (2)	C17—O10	1.415 (2)
C117—C122	1.528 (3)	C17—C22	1.534 (3)
C118—C119	1.537 (3)	C18—C19	1.544 (3)
C118—O111	1.434 (2)	C18—O11	1.442 (2)
C118—H1181	0.968	C18—H181	0.962
C119—C120	1.515 (3)	C19—C20	1.518 (3)
C119—O112	1.435 (3)	C19—O12	1.424 (3)
C119—H1191	0.978	C19—H191	0.973
C120—C121	1.532 (3)	C20—C21	1.517 (4)
C120—O113	1.433 (3)	C20—O13	1.438 (3)

C120—H1201	0.973	C20—H201	0.973
C121—O114	1.425 (3)	C21—O14	1.427 (3)
C121—H1211	0.969	C21—H211	0.969
C121—H1212	0.979	C21—H212	0.980
O110—H1101	0.810	O10—H101	0.828
O111—C125	1.439 (2)	O11—C25	1.438 (2)
O112—C125	1.431 (3)	O12—C25	1.429 (3)
O113—C128	1.423 (3)	O13—C28	1.439 (3)
O114—C128	1.420 (3)	O14—C28	1.423 (3)
C122—N102	1.499 (3)	C22—N2	1.501 (3)
C122—H1221	0.971	C22—H222	0.970
C122—H1222	0.975	C22—H221	0.976
N102—C123	1.491 (3)	N2—C23	1.500 (3)
N102—C124	1.500 (3)	N2—C24	1.492 (3)
N102—H1021	0.896	N2—H21	0.900
C123—H1231	0.964	C23—H231	0.956
C123—H1233	0.962	C23—H232	0.964
C123—H1232	0.958	C23—H233	0.953
C124—H1241	0.962	C24—H241	0.971
C124—H1243	0.971	C24—H242	0.973
C124—H1242	0.967	C24—H243	0.967
C125—C126	1.518 (3)	C25—C26	1.515 (3)
C125—C127	1.508 (3)	C25—C27	1.504 (3)
C126—H1261	0.964	C26—H262	0.960
C126—H1262	0.969	C26—H261	0.963
C126—H1263	0.952	C26—H263	0.962
C127—H1271	0.966	C27—H272	0.974
C127—H1272	0.964	C27—H271	0.958
C127—H1273	0.965	C27—H273	0.971
C128—C130	1.502 (3)	C28—C29	1.512 (4)
C128—C129	1.518 (3)	C28—C30	1.511 (4)
C130—H1301	0.971	C29—H292	0.970
C130—H1302	0.970	C29—H291	0.968
C130—H1303	0.962	C29—H293	0.960
C129—H1291	0.973	C30—H302	0.967
C129—H1292	0.962	C30—H301	0.978
C129—H1293	0.973	C30—H303	0.974

C117—C116—O108	116.90 (18)	C17—C16—O8	116.28 (18)
C117—C116—O109	115.55 (18)	C17—C16—O9	116.84 (17)
O108—C116—O109	127.5 (2)	O8—C16—O9	126.9 (2)
C116—C117—C118	107.24 (15)	C16—C17—C18	107.57 (16)
C116—C117—O110	110.29 (16)	C16—C17—O10	109.98 (15)
C118—C117—O110	110.14 (16)	C18—C17—O10	110.78 (16)
C116—C117—C122	110.32 (16)	C16—C17—C22	112.36 (16)
C118—C117—C122	107.92 (16)	C18—C17—C22	105.42 (15)
O110—C117—C122	110.84 (16)	O10—C17—C22	110.61 (16)
C117—C118—C119	113.87 (17)	C17—C18—C19	115.13 (16)

C117—C118—O111	111.02 (15)	C17—C18—O11	111.26 (16)
C119—C118—O111	104.98 (16)	C19—C18—O11	104.04 (15)
C117—C118—H1181	108.5	C17—C18—H181	107.8
C119—C118—H1181	110.2	C19—C18—H181	110.4
O111—C118—H1181	108.1	O11—C18—H181	108.0
C118—C119—C120	113.97 (17)	C18—C19—C20	112.14 (17)
C118—C119—O112	103.37 (16)	C18—C19—O12	104.19 (15)
C120—C119—O112	110.72 (17)	C20—C19—O12	110.16 (18)
C118—C119—H1191	110.7	C18—C19—H191	111.1
C120—C119—H1191	108.5	C20—C19—H191	109.7
O112—C119—H1191	109.5	O12—C19—H191	109.4
C119—C120—C121	115.40 (19)	C19—C20—C21	116.8 (2)
C119—C120—O113	109.34 (18)	C19—C20—O13	110.66 (17)
C121—C120—O113	103.99 (16)	C21—C20—O13	102.13 (19)
C119—C120—H1201	108.8	C19—C20—H201	107.0
C121—C120—H1201	110.5	C21—C20—H201	111.3
O113—C120—H1201	108.6	O13—C20—H201	108.8
C120—C121—O114	103.85 (17)	C20—C21—O14	101.6 (2)
C120—C121—H1211	110.8	C20—C21—H211	112.6
O114—C121—H1211	109.7	O14—C21—H211	110.3
C120—C121—H1212	111.3	C20—C21—H212	111.4
O114—C121—H1212	111.2	O14—C21—H212	112.3
H1211—C121—H1212	109.8	H211—C21—H212	108.6
C117—O110—H1101	108.1	C17—O10—H101	104.7
C118—O111—C125	108.99 (15)	C18—O11—C25	109.17 (14)
C119—O112—C125	106.95 (15)	C19—O12—C25	107.31 (15)
C120—O113—C128	108.48 (16)	C20—O13—C28	108.15 (16)
C121—O114—C128	105.60 (16)	C21—O14—C28	105.86 (17)
C117—C122—N102	115.47 (16)	C17—C22—N2	116.01 (15)
C117—C122—H1221	109.1	C17—C22—H222	107.8
N102—C122—H1221	107.6	N2—C22—H222	108.0
C117—C122—H1222	108.6	C17—C22—H221	107.4
N102—C122—H1222	106.1	N2—C22—H221	107.1
H1221—C122—H1222	109.9	H222—C22—H221	110.6
C122—N102—C123	108.96 (16)	C22—N2—C23	109.03 (15)
C122—N102—C124	114.76 (17)	C22—N2—C24	114.62 (16)
C123—N102—C124	109.14 (17)	C23—N2—C24	109.44 (16)
C122—N102—H1021	109.2	C22—N2—H21	109.7
C123—N102—H1021	106.4	C23—N2—H21	106.1
C124—N102—H1021	108.1	C24—N2—H21	107.6
N102—C123—H1231	107.6	N2—C23—H231	107.9
N102—C123—H1233	107.2	N2—C23—H232	107.3
H1231—C123—H1233	111.6	H231—C23—H232	111.1
N102—C123—H1232	108.1	N2—C23—H233	109.2
H1231—C123—H1232	111.4	H231—C23—H233	110.3
H1233—C123—H1232	110.8	H232—C23—H233	110.8
N102—C124—H1241	110.0	N2—C24—H241	108.3
N102—C124—H1243	108.5	N2—C24—H242	109.1

H1241—C124—H1243	110.9	H241—C24—H242	111.5
N102—C124—H1242	109.1	N2—C24—H243	106.5
H1241—C124—H1242	108.7	H241—C24—H243	110.6
H1243—C124—H1242	109.5	H242—C24—H243	110.7
O111—C125—O112	105.55 (16)	O11—C25—O12	104.59 (16)
O111—C125—C126	109.99 (17)	O11—C25—C26	110.80 (17)
O112—C125—C126	110.79 (18)	O12—C25—C26	112.11 (17)
O111—C125—C127	108.73 (17)	O11—C25—C27	108.73 (17)
O112—C125—C127	108.80 (17)	O12—C25—C27	108.40 (18)
C126—C125—C127	112.70 (18)	C26—C25—C27	111.91 (18)
C125—C126—H1261	108.5	C25—C26—H262	109.2
C125—C126—H1262	108.4	C25—C26—H261	111.6
H1261—C126—H1262	109.5	H262—C26—H261	109.9
C125—C126—H1263	109.6	C25—C26—H263	108.4
H1261—C126—H1263	110.4	H262—C26—H263	108.2
H1262—C126—H1263	110.4	H261—C26—H263	109.5
C125—C127—H1271	110.4	C25—C27—H272	107.4
C125—C127—H1272	110.1	C25—C27—H271	109.5
H1271—C127—H1272	109.0	H272—C27—H271	111.9
C125—C127—H1273	109.5	C25—C27—H273	107.7
H1271—C127—H1273	108.9	H272—C27—H273	111.6
H1272—C127—H1273	109.0	H271—C27—H273	108.6
O113—C128—O114	104.50 (16)	O13—C28—O14	106.07 (18)
O113—C128—C130	108.70 (19)	O13—C28—C29	109.3 (2)
O114—C128—C130	109.61 (18)	O14—C28—C29	110.1 (2)
O113—C128—C129	110.74 (19)	O13—C28—C30	109.3 (2)
O114—C128—C129	110.20 (18)	O14—C28—C30	108.6 (2)
C130—C128—C129	112.7 (2)	C29—C28—C30	113.2 (2)
C128—C130—H1301	110.4	C28—C29—H292	110.3
C128—C130—H1302	109.9	C28—C29—H291	109.3
H1301—C130—H1302	109.4	H292—C29—H291	108.8
C128—C130—H1303	110.4	C28—C29—H293	107.5
H1301—C130—H1303	108.7	H292—C29—H293	108.6
H1302—C130—H1303	108.0	H291—C29—H293	112.4
C128—C129—H1291	109.5	C28—C30—H302	109.0
C128—C129—H1292	109.3	C28—C30—H301	109.4
H1291—C129—H1292	109.3	H302—C30—H301	109.2
C128—C129—H1293	109.3	C28—C30—H303	108.9
H1291—C129—H1293	109.2	H302—C30—H303	110.0
H1292—C129—H1293	110.3	H301—C30—H303	110.4

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H21···O109	0.90	1.80	2.664 (3)	161
N102—H1021···O9	0.90	1.81	2.675 (3)	162

O110—H1101···O109^{i}	0.81	2.69	3.348 (3)	140
O10—H101···O11^{i}	0.83	2.50	3.187 (3)	141

Symmetry code: (i) $x+1, y, z$.