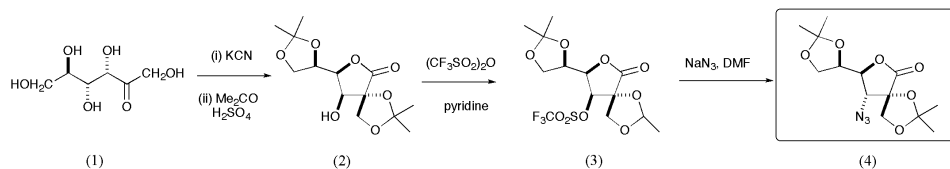


Christopher C. Harding,<sup>a\*</sup>  
Howard Shallard-Brown,<sup>a</sup>  
David J. Watkin,<sup>a</sup> Raquel  
Soengas<sup>b</sup> and George W. J. Fleet<sup>b</sup><sup>a</sup>Department of Chemical Crystallography,  
Chemical Research Laboratory, Oxford  
University, Mansfield Road, Oxford OX1 3TA,  
England, and <sup>b</sup>Department of Organic  
Chemistry, Chemical Research Laboratory,  
Oxford University, Mansfield Road, Oxford  
OX1 3TA, EnglandCorrespondence e-mail:  
christopher.harding@seh.ox.ac.uk

## Key indicators

Single-crystal X-ray study  
T = 120 K  
Mean  $\sigma(\text{C}-\text{C}) = 0.005 \text{ \AA}$   
R factor = 0.045  
wR factor = 0.108  
Data-to-parameter ratio = 9.3For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.3-Azido-3-deoxy-2,2':5,6-di-O-isopropylidene-  
2-C-hydroxymethyl-D-gulono-1,4-lactoneThe title azidolactone,  $\text{C}_{13}\text{H}_{19}\text{O}_6\text{N}_3$ , formed by  $\text{S}_{\text{N}}2$  displacement of the trifluoromethanesulfonate with sodium azide, is the first example of a branched  $\beta$ -sugar amino acid scaffold.Received 22 October 2004  
Accepted 10 November 2004  
Online 20 November 2004

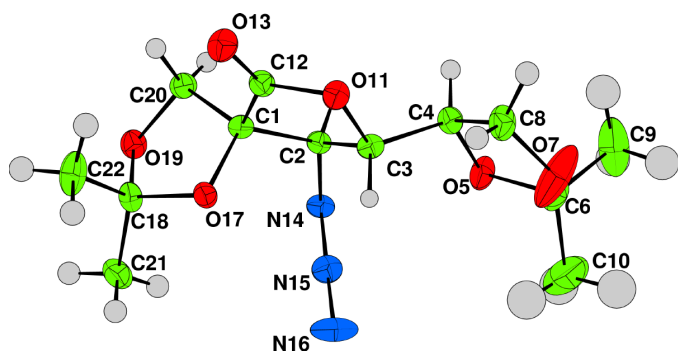
## Comment

Gellman (Lai & Gellman, 2003; Hayen *et al.*, 2004) and Seebach (Lelais & Seebach, 2003; Rueping *et al.*, 2004) have pioneered studies on the exploitation of  $\beta$ -amino acids in their adoption of secondary structural features in short chains. Although sugar amino acids (SAA) have been extensively investigated as peptidomimetics and dipeptide isosteres (Schweizer, 2002; Chakraborty *et al.*, 2004), there have been very few studies of  $\beta$ -SAAs (Jenkinson & Fleet, 2004; Johnson *et al.*, 2004), even though some oxetane-derived  $\beta$ -SAAs exhibited novel helical structures (Barker *et al.*, 2001). This paper reports the structure of the  $\beta$ -azidolactone, (4), which is a novel  $\beta$ -SAA scaffold containing a branched carbon chain.

D-Fructose may be readily converted into the diacetonide (2) (Hotchkiss *et al.*, 2004); esterification of (2) with triflic anhydride gave the corresponding stable trifluoromethanesulfonate. Reaction of (3) with sodium azide in DMF gave an organic azide in good yield. The structure of this azide is fraught with uncertainties; the trifluoromethanesulfonate in (3) has two  $\beta$ -O atoms and the adjacent  $\alpha$ -C atom is trisubstituted, so the efficiency of the  $\text{S}_{\text{N}}2$  reaction is surprising. There is considerable ambiguity in the stereochemistry of the product, since there may well be neighbouring group participation by the O atom; it is also possible that some rearrangement that maintained the same connectivity of CH atoms may have occurred. However, X-ray crystallographic analysis of the product of the reaction showed that the anticipated inverted azide, (4), had indeed been produced.

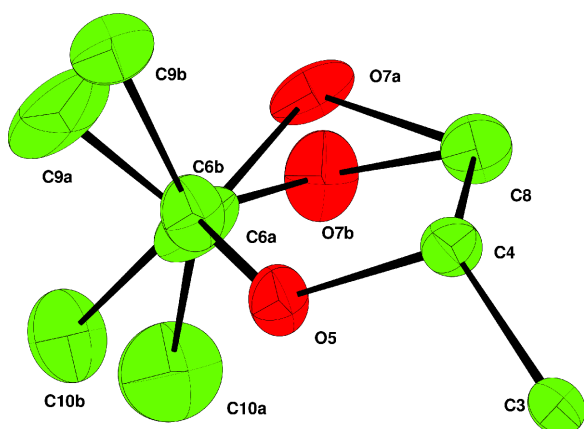
## Experimental

The full preparative method is not available for publication as yet. The sample was crystallized from diethyl ether by inward diffusion of *n*-hexane to give lath-shaped colourless crystals.



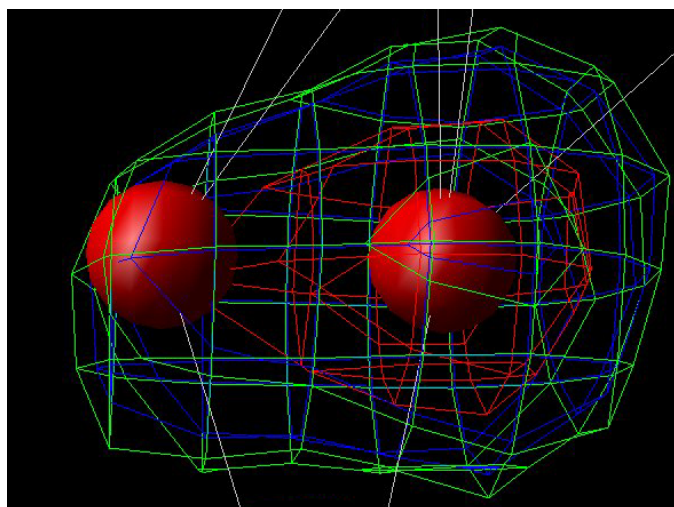
**Figure 1**

The title molecule at 120 K, with displacement ellipsoids drawn at the 50% probability level. The large ellipsoids at atoms O7, C9 and C10 are discussed in the text. H atoms are shown as spheres of arbitrary radii.



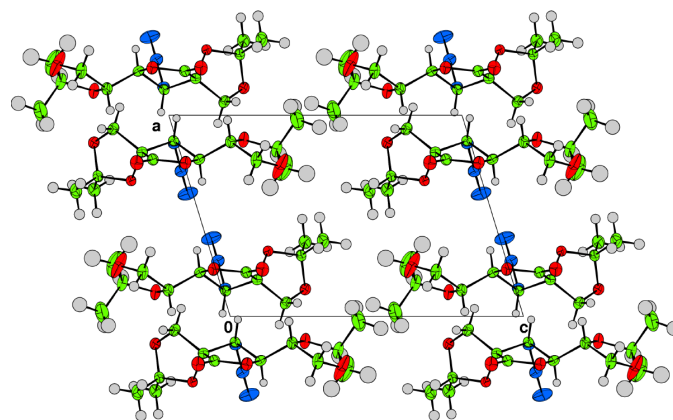
**Figure 2**

The disordered fragment of the title molecule at 120 K, displayed as a 'split atom' model. Even with firm anisotropic displacement parameter similarity restraints, the ellipsoids do not conform to any reasonable physical model.



**Figure 3**

$F_o$  electron density map viewed perpendicular to the line through atoms O7a and O7b, computed excluding phasing information derived from these two partial atoms. There are no distinct lobes near the two atom positions proposed by the 'split atom' refinement, suggesting that there still remains substantial dynamic disorder.



**Figure 4**

Packing diagram of the title compound, viewed along the  $b$  axis.

#### Crystal data

$C_{13}H_{19}N_3O_6$

$M_r = 313.31$

Monoclinic,  $P2_1$

$a = 8.7755$  (2) Å

$b = 7.6452$  (2) Å

$c = 12.2232$  (3) Å

$\beta = 106.9659$  (11)°

$V = 784.37$  (3) Å<sup>3</sup>

$Z = 2$

$D_x = 1.326$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation

Cell parameters from 1734

reflections

$\theta = 5$ – $27^\circ$

$\mu = 0.11$  mm<sup>-1</sup>

$T = 120$  K

Lath, colourless

$0.30 \times 0.20 \times 0.10$  mm

#### Data collection

Nonius KappaCCD diffractometer

$\omega$  scans

Absorption correction: multi-scan

(*DENZO/SCALEPACK*;

Otwinowski & Minor, 1997)

$T_{\min} = 0.98$ ,  $T_{\max} = 0.99$

3320 measured reflections

1869 independent reflections

1655 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.015$

$\theta_{\max} = 27.5^\circ$

$h = -11 \rightarrow 11$

$k = -9 \rightarrow 9$

$l = -15 \rightarrow 15$

#### Refinement

Refinement on  $F^2$

$R[F^2 > 2\sigma(F^2)] = 0.045$

$wR(F^2) = 0.109$

$S = 0.93$

1866 reflections

200 parameters

H-atom parameters constrained

$w = 1/[\sigma^2(F) + (0.0522p)^2 + 0.571p]$ ,

where  $p = [\max(F_o^2, 0) + 2F^2]/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.65$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.42$  e Å<sup>-3</sup>

Extinction correction: Larson

(1970)

Extinction coefficient:  $3.0$  (5)  $\times 10^2$

**Table 1**

Selected interatomic distances (Å).

O5—C6	1.433 (3)	C6—C10	1.486 (7)
C6—O7	1.370 (5)	O7—C8	1.384 (4)
C6—C9	1.503 (6)		

An initial data set was collected at 190 K. This gave reasonable refinement [ $N_{\text{measured}} = 2766$ ,  $R_{\text{int}} = 0.02$ ,  $N_{\text{ref}} = 1748$ ,  $R_w(2\sigma) = 0.081$ ,  $R(2\sigma) = 0.034$ ], though atoms O7, C9 and C10 had very elongated displacement ellipsoids. It was unclear whether the C6/O7/C9/C10 fragment should be modelled with large anisotropic displacement parameters (ADPs) or with 'split atoms'. Refinement was continued with this fragment represented by 'split atoms'. However, the ADPs could not be explained by a rational physical model. As with the unsplit model, the bond lengths deviated unacceptably from averages drawn by *MOGUL* (Bruno *et al.*, 2004) from the Cambridge Structural Database (CSD; Version 5.25; Allen, 2002), even with the application of firm bond length similarity restraints.

In order to resolve this issue, data were recollected at 120 K. It is the result of this refinement that is reported in the CIF. Even at this temperature, the ADPs of the problematic group remained large, though not as large as in the 190 K data set. Both the large ADP and the 'split atom' refinements continued to give unacceptable bond lengths and ellipsoids (see Fig. 2). Fig. 3, plotted with *MCE* (Hušák & Kratochvíl, 2003), shows the observed electron density perpendicular to the line connecting atoms *O7a* and *O7b*. The map is phased by all of the structure except these two atoms, which are included for illustrative purposes at the positions they refine to in the 'split atom' model. There is a smooth transition in electron density between the two sites, with no evident build-up of density at either site.

One interpretation of these observations is that neither the split atom model nor the large ADP model really represents what is occurring in this structure. It is clearly something more complicated than simply having the envelope flap (*O7/O7a*) distributed over two sites on opposite sides of a plane through the other ring atoms. Of 29 structures in the CSD containing this moiety some clearly have the atom corresponding to *O7* as the 'flap', some have *C6* as the flap, but there are also a number in which no four atoms form a convincing plane. It seems that, even in the solid state, there is a continuum between an O-flap and a C-flap geometry. The amount of material available was insufficient to enable low-temperature solid-state NMR measurements to be carried out.

For the large ADP model, all H atoms were seen in the difference electron density map (even those on the highly anisotropic *C9* and *C10*). Their positions and  $U_{\text{iso}}(\text{H})$  values were regularized by several cycles of refinement using slack restraints, after which the refinement was completed using 'riding' constraints and all reflections with  $I > -3\sigma(I)$ . Fig. 4 is a packing diagram showing an alternation of highly mobile and highly ordered groups lying in a plane at approximately  $c/2$ .

Data collection: *COLLECT* (Nonius, 1997–2001); 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*.

Financial support (to RS) provided through the European Community's Human Potential Programme under contract HPRN-CT-2002-00173 is gratefully acknowledged.

## References

- Allen, F. H. (2002). *Acta Cryst.* **B58**, 380–388.
- Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
- Barker, S. F., Angus, D., Taillefumier, C., Probert, M. R., Watkin, D. J., Watterson, M. P., Claridge, T. D. W., Hungerford, N. L. & Fleet, G. W. J. (2001). *Tetrahedron Lett.* **42**, 4247–4250.
- Betteridge, P. W., Carruthers, J. R., Cooper, R. I., Prout, K. & Watkin, D. J. (2003). *J. Appl. Cryst.* **36**, 1487.
- Bruno, I. J., Cole, J. C., Kessler, M., Luo, J., Motherwell, W. D. S., Purkis, L. H., Smith, B. R., Taylor, R., Cooper, R. I., Harris, S. E. & Orpen, A. G. (2004). *J. Chem. Inf. Comput. Sci.* In the press.
- Chakraborty, T. K., Srinivasi, P., Tapadar, S. & Mohan, B. K. (2004). *J. Chem. Sci.* **116**, 187–207.
- Hayen, A., Schmitt, M. A., Ngassa, F. N., Thomasson, K. A. & Gellman, S. H. (2004). *Angew. Chem. Int. Ed.* **43**, 505–510.
- Hotchkiss, D., Soengas, R., Simone, M. I., van Ameijde, J., Hunter, S., Cowley, A. R. & Fleet, G. W. J. (2004). *Tetrahedron Lett.* **45**. In the press.
- Hušák, M. & Kratochvíl, B. (2003). *J. Appl. Cryst.* **36**, 1104.
- Jenkinson, S. F. & Fleet, G. W. J. (2004). *Tetrahedron Asymmetry*, **15**, 2667–2679.
- Johnson, S. W., Jenkinson, S. F., Angus, D., Taillefumier, C., Jones, J. H. & Fleet, G. W. J. (2004). *Tetrahedron Asymmetry*, **15**, 2681–2686.
- Lai, J. R. & Gellman, S. H. (2003). *Protein Sci.* **12**, 560–566.
- Larson, A. C. (1970). *Crystallographic Computing*, edited by F. R. Ahmed, S. R. Hall & C. P. Huber, pp. 291–294. Copenhagen: Munksgaard.
- Lelais, G. & Seebach, D. (2003). *Helv. Chim. Acta*, **86**, 4152–4168.
- Nonius (1997–2001). *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.
- Rueping, M., Mahajan, Y. R., Jaun, B. & Seebach, D. (2004). *Chem. Eur. J.* **10**, 1607–1615.
- Schweizer, F. (2002). *Angew. Chem. Int. Ed.* **41**, 230–253.
- Watkin, D. J., Prout, C. K. & Pearce, L. J. (1996). *CAMERON*. Chemical Crystallography Laboratory, Oxford, England.

## supporting information

*Acta Cryst.* (2004). E60, o2334–o2336 [https://doi.org/10.1107/S1600536804029113]

### 3-Azido-3-deoxy-2,2':5,6-di-O-isopropylidene-2-C-hydroxymethyl-D-gulono-1,4-lactone

Christopher C. Harding, Howard Shallard-Brown, David J. Watkin, Raquel Soengas and George W. J. Fleet

#### 3-Azido-3-deoxy-2,2':5,6-di-O-isopropylidene-2-C-hydroxymethyl-D-gulono-1,4-lactone

##### Crystal data

$C_{13}H_{19}N_3O_6$

$M_r = 313.31$

Monoclinic,  $P2_1$

$a = 8.7755$  (2) Å

$b = 7.6452$  (2) Å

$c = 12.2232$  (3) Å

$\beta = 106.9659$  (11)°

$V = 784.37$  (3) Å<sup>3</sup>

$Z = 2$

$F(000) = 332$

$D_x = 1.326$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 1734 reflections

$\theta = 5\text{--}27^\circ$

$\mu = 0.11$  mm<sup>-1</sup>

$T = 120$  K

Plate, colourless

$0.30 \times 0.20 \times 0.10$  mm

##### Data collection

Nonius KappaCCD

diffractometer

Graphite monochromator

$\omega$  scans

Absorption correction: multi-scan

DENZO/SCALEPACK (Otwinowski & Minor, 1997)

$T_{\min} = 0.98$ ,  $T_{\max} = 0.99$

3320 measured reflections

1869 independent reflections

1655 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.015$

$\theta_{\max} = 27.5^\circ$ ,  $\theta_{\min} = 5.2^\circ$

$h = -11 \rightarrow 11$

$k = -9 \rightarrow 9$

$l = -15 \rightarrow 15$

##### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.045$

$wR(F^2) = 0.109$

$S = 0.93$

1866 reflections

200 parameters

1 restraint

Primary atom site location: structure-invariant  
direct methods

Hydrogen site location: inferred from  
neighbouring sites

H-atom parameters constrained

Method: SHELXL97 (Sheldrick, 1997);  $w =$

$1/[\sigma^2(f) + (0.0522p)^2 + 0.571p]$ ,

where  $p = [\max(F_o^2, 0) + 2F_c^2]/3$

$(\Delta/\sigma)_{\max} = 0.000326$

$\Delta\rho_{\max} = 0.65$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.42$  e Å<sup>-3</sup>

Extinction correction: Larson 1970

Crystallographic Computing eq 22

Extinction coefficient: 300 (50)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.8173 (3)	0.1205 (4)	0.8704 (2)	0.0207
C2	0.8728 (3)	0.0356 (4)	0.9895 (2)	0.0197
C3	0.7808 (3)	0.1387 (4)	1.0574 (2)	0.0214
C4	0.8684 (3)	0.1553 (4)	1.1835 (2)	0.0253
O5	0.8788 (3)	-0.0165 (3)	1.22982 (16)	0.0306
C6	0.8328 (4)	-0.0098 (5)	1.3330 (3)	0.0349
O7	0.7463 (5)	0.1409 (5)	1.3271 (3)	0.0777
C8	0.7788 (4)	0.2612 (4)	1.2524 (3)	0.0345
C9	0.9801 (6)	0.0021 (9)	1.4333 (3)	0.0762
C10	0.7390 (8)	-0.1702 (9)	1.3373 (5)	0.0891
O11	0.7642 (2)	0.3122 (3)	1.00698 (16)	0.0251
C12	0.7805 (3)	0.3076 (4)	0.8996 (2)	0.0234
O13	0.7649 (3)	0.4350 (3)	0.84141 (18)	0.0315
N14	0.8584 (3)	-0.1555 (3)	0.9887 (2)	0.0219
N15	0.7245 (3)	-0.2124 (4)	0.9845 (2)	0.0267
N16	0.6106 (4)	-0.2828 (4)	0.9831 (3)	0.0454
O17	0.6748 (2)	0.0412 (3)	0.80306 (15)	0.0223
C18	0.6968 (3)	-0.0131 (4)	0.6948 (2)	0.0246
O19	0.8643 (2)	-0.0228 (3)	0.71573 (16)	0.0250
C20	0.9322 (3)	0.1103 (4)	0.7966 (2)	0.0252
C21	0.6301 (4)	-0.1943 (5)	0.6674 (3)	0.0338
C22	0.6235 (4)	0.1233 (5)	0.6046 (3)	0.0408
H21	0.9848	0.0640	1.0229	0.0240*
H31	0.6712	0.0884	1.0445	0.0251*
H41	0.9753	0.2039	1.1948	0.0304*
H81	0.8497	0.3528	1.2967	0.0438*
H82	0.6789	0.3123	1.2055	0.0432*
H91	0.9408	0.0093	1.5009	0.0912*
H92	1.0438	-0.1057	1.4370	0.0913*
H93	1.0444	0.1073	1.4251	0.0920*
H101	0.7105	-0.1659	1.4094	0.1170*
H102	0.8053	-0.2750	1.3328	0.1173*
H103	0.6413	-0.1563	1.2703	0.1167*
H201	1.0400	0.0778	0.8411	0.0321*
H202	0.9326	0.2218	0.7573	0.0317*
H211	0.6543	-0.2349	0.5979	0.0399*
H212	0.6785	-0.2742	0.7300	0.0403*
H213	0.5151	-0.1924	0.6528	0.0409*
H221	0.6405	0.1021	0.5300	0.0487*
H222	0.6603	0.2431	0.6279	0.0489*
H223	0.5076	0.1206	0.5922	0.0491*

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C1	0.0206 (13)	0.0191 (13)	0.0222 (12)	-0.0012 (11)	0.0061 (10)	-0.0002 (11)
C2	0.0206 (13)	0.0182 (13)	0.0199 (12)	-0.0019 (11)	0.0051 (10)	-0.0009 (10)
C3	0.0215 (13)	0.0186 (15)	0.0256 (13)	0.0009 (11)	0.0090 (10)	-0.0002 (11)
C4	0.0289 (15)	0.0252 (16)	0.0223 (12)	-0.0017 (12)	0.0084 (11)	-0.0032 (12)
O5	0.0445 (12)	0.0282 (11)	0.0235 (9)	0.0034 (10)	0.0166 (9)	0.0017 (9)
C6	0.0495 (19)	0.0342 (17)	0.0279 (14)	-0.0014 (17)	0.0220 (13)	0.0011 (14)
O7	0.118 (3)	0.069 (2)	0.080 (2)	0.051 (2)	0.082 (2)	0.0350 (19)
C8	0.0481 (19)	0.0326 (18)	0.0262 (14)	0.0028 (15)	0.0161 (14)	-0.0044 (13)
C9	0.086 (3)	0.109 (5)	0.0317 (18)	0.019 (4)	0.013 (2)	-0.001 (3)
C10	0.120 (5)	0.089 (5)	0.080 (4)	-0.047 (4)	0.064 (4)	-0.009 (3)
O11	0.0307 (11)	0.0208 (11)	0.0253 (9)	0.0038 (9)	0.0106 (8)	-0.0001 (8)
C12	0.0244 (14)	0.0229 (15)	0.0225 (12)	-0.0005 (12)	0.0063 (10)	-0.0013 (12)
O13	0.0395 (12)	0.0238 (11)	0.0315 (11)	0.0066 (10)	0.0106 (9)	0.0059 (9)
N14	0.0187 (12)	0.0206 (12)	0.0266 (11)	-0.0017 (10)	0.0069 (9)	-0.0019 (10)
N15	0.0279 (13)	0.0217 (12)	0.0320 (13)	0.0018 (11)	0.0109 (10)	-0.0010 (10)
N16	0.0334 (15)	0.0299 (16)	0.078 (2)	-0.0116 (13)	0.0236 (15)	-0.0071 (16)
O17	0.0209 (9)	0.0272 (11)	0.0189 (8)	-0.0018 (8)	0.0057 (7)	-0.0032 (8)
C18	0.0245 (13)	0.0301 (15)	0.0186 (11)	0.0025 (13)	0.0053 (10)	-0.0021 (12)
O19	0.0254 (10)	0.0270 (11)	0.0236 (9)	0.0018 (9)	0.0089 (7)	-0.0026 (8)
C20	0.0287 (14)	0.0239 (14)	0.0259 (13)	-0.0041 (13)	0.0125 (11)	-0.0043 (12)
C21	0.0341 (16)	0.0377 (19)	0.0289 (14)	-0.0073 (15)	0.0084 (12)	-0.0114 (14)
C22	0.0436 (18)	0.054 (2)	0.0262 (14)	0.0194 (18)	0.0116 (13)	0.0104 (15)

*Geometric parameters (Å, °)*

C1—C2	1.538 (4)	C9—H93	1.004
C1—C12	1.531 (4)	C10—H101	0.984
C1—O17	1.417 (3)	C10—H102	1.001
C1—C20	1.539 (3)	C10—H103	1.004
C2—C3	1.533 (4)	O11—C12	1.362 (3)
C2—N14	1.466 (4)	C12—O13	1.190 (4)
C2—H21	0.973	N14—N15	1.240 (3)
C3—C4	1.514 (4)	N15—N16	1.131 (4)
C3—O11	1.452 (3)	O17—C18	1.452 (3)
C3—H31	1.005	C18—O19	1.419 (3)
C4—O5	1.423 (4)	C18—C21	1.504 (5)
C4—C8	1.538 (4)	C18—C22	1.518 (4)
C4—H41	0.981	O19—C20	1.422 (3)
O5—C6	1.433 (3)	C20—H201	0.976
C6—O7	1.370 (5)	C20—H202	0.979
C6—C9	1.503 (6)	C21—H211	0.984
C6—C10	1.486 (7)	C21—H212	0.974
O7—C8	1.384 (4)	C21—H213	0.973
C8—H81	0.987	C22—H221	0.980
C8—H82	0.979	C22—H222	0.985

---

C9—H91	0.985	C22—H223	0.984
C9—H92	0.989		
C2—C1—C12	101.8 (2)	C6—C9—H93	109.937
C2—C1—O17	110.6 (2)	H91—C9—H93	112.584
C12—C1—O17	109.0 (2)	H92—C9—H93	110.192
C2—C1—C20	116.9 (2)	C6—C10—H101	107.044
C12—C1—C20	113.8 (2)	C6—C10—H102	108.749
O17—C1—C20	104.8 (2)	H101—C10—H102	112.303
C1—C2—C3	102.9 (2)	C6—C10—H103	103.516
C1—C2—N14	114.3 (2)	H101—C10—H103	110.346
C3—C2—N14	117.3 (2)	H102—C10—H103	114.253
C1—C2—H21	107.763	C3—O11—C12	111.1 (2)
C3—C2—H21	106.585	C1—C12—O11	109.7 (2)
N14—C2—H21	107.444	C1—C12—O13	128.4 (2)
C2—C3—C4	113.8 (2)	O11—C12—O13	121.9 (3)
C2—C3—O11	104.1 (2)	C2—N14—N15	115.4 (2)
C4—C3—O11	108.3 (2)	N14—N15—N16	172.0 (3)
C2—C3—H31	110.457	C1—O17—C18	108.8 (2)
C4—C3—H31	111.708	O17—C18—O19	104.9 (2)
O11—C3—H31	108.048	O17—C18—C21	108.6 (2)
C3—C4—O5	106.4 (2)	O19—C18—C21	107.9 (2)
C3—C4—C8	114.4 (2)	O17—C18—C22	109.0 (2)
O5—C4—C8	104.6 (2)	O19—C18—C22	111.4 (2)
C3—C4—H41	110.832	C21—C18—C22	114.6 (3)
O5—C4—H41	110.245	C18—O19—C20	106.8 (2)
C8—C4—H41	110.109	C1—C20—O19	103.3 (2)
C4—O5—C6	108.4 (2)	C1—C20—H201	112.385
O5—C6—O7	106.2 (3)	O19—C20—H201	110.314
O5—C6—C9	108.9 (3)	C1—C20—H202	109.886
O7—C6—C9	108.9 (4)	O19—C20—H202	110.026
O5—C6—C10	107.6 (3)	H201—C20—H202	110.659
O7—C6—C10	113.0 (4)	C18—C21—H211	108.339
C9—C6—C10	112.0 (4)	C18—C21—H212	110.490
C6—O7—C8	112.1 (2)	H211—C21—H212	109.050
C4—C8—O7	104.4 (3)	C18—C21—H213	110.056
C4—C8—H81	109.437	H211—C21—H213	108.699
O7—C8—H81	109.119	H212—C21—H213	110.158
C4—C8—H82	113.777	C18—C22—H221	114.558
O7—C8—H82	109.278	C18—C22—H222	113.225
H81—C8—H82	110.606	H221—C22—H222	107.285
C6—C9—H91	105.028	C18—C22—H223	107.279
C6—C9—H92	109.043	H221—C22—H223	106.580
H91—C9—H92	109.895	H222—C22—H223	107.508

---