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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

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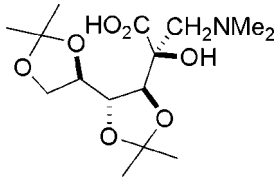
Key indicators: single-crystal X-ray study; $T$ = 150 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.032; $wR$ factor = 0.033; data-to-parameter ratio = 7.8.

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 $\beta$-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
$\beta$ = 99.1141 (8)°
$V$ = 1705.09 (9) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 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).

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# supplementary materials

*Acta Cryst.* (2008). E**64**, o294-o295 [ **doi: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

**S. F. Jenkinson, D. J. Hotchkiss, A. R. Cowley, G. W. J. Fleet and D. J. Watkin**

## 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*-dimethyl-amino)-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).

## 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).

## 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.

**Figures**

Fig. 1. The synthesis of the title compound.

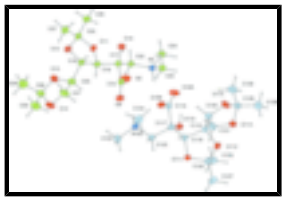

Fig. 2. The title compound with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitary radius.

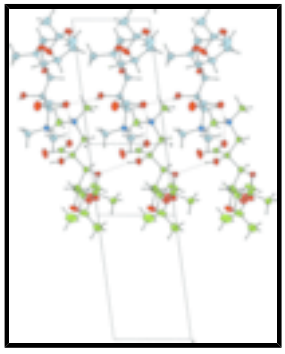

Fig. 3. The packing diagram for the molecule showing the infinite hydrogen bonded chains of molecules lying parallel to the *a* axis.

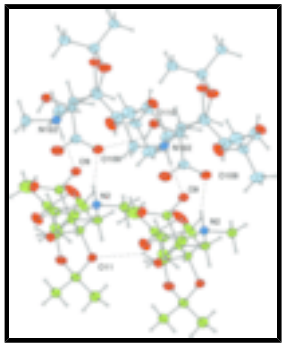

Fig. 4. The two molecules of the asymmetric unit are linked by two strong hydrogen bonds, O9···H21—N2 and O109···H1021-N102 and these dimeric units are linked by weak hydrogen bonds, O11···H101—O10 and O109···H1101-O110, to form a hydrogen bonded column.

**(*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**

*Crystal data*

| | |
|---|---|
| $C_{15}H_{27}N_1O_7$ | $F_{000}$ = 720 |
| $M_r$ = 333.38 | $D_x$ = 1.299 Mg m$^{-3}$ |
| Monoclinic, $P2_1$ | Mo $K\alpha$ radiation<br>λ = 0.71073 Å |
| $a$ = 5.7881 (2) Å | Cell parameters from 13496 reflections |
| $b$ = 16.7077 (4) Å | θ = 5–28º |
| $c$ = 17.8572 (5) Å | μ = 0.10 mm$^{-1}$ |
| β = 99.1141 (8)º | $T$ = 150 K |
| $V$ = 1705.09 (9) Å$^3$ | Fragment, colourless |
| $Z$ = 4 | 0.40 × 0.10 × 0.08 mm |

*Data collection*

| | |
|---|---|
| Nonius KappaCCD diffractometer | 3474 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int} = 0.034$ |
| $T = 150$ K | $\theta_{max} = 27.5^o$ |
| ω scans | $\theta_{min} = 5.1^o$ |
| Absorption correction: multi-scan (DENZO/SCALEPACK; Otwinowski & Minor, 1997) | $h = -7 \rightarrow 7$ |
| $T_{min} = 0.96$, $T_{max} = 1.01$ | $k = -20 \rightarrow 21$ |
| 13496 measured reflections | $l = -23 \rightarrow 23$ |
| 4000 independent reflections | |

*Refinement*

| | |
|---|---|
| Refinement on $F$ | H-atom parameters constrained |
| Least-squares matrix: full | $w = [1-(F_o-F_c)^2/36\sigma^2(F)]^2/[0.462T_0(x) + 0.141T_1(x) + 0.209T_2(x)]$ where $T_i$ are Chebychev polynomials and $x = F_c/F_{max}$ (*P*rince, 1982; Watkin, 1994) |
| $R[F^2 > 2\sigma(F^2)] = 0.032$ | $(\Delta/\sigma)_{max} = 0.003$ |
| $wR(F^2) = 0.033$ | $\Delta\rho_{max} = 0.18$ e Å$^{-3}$ |
| $S = 1.09$ | $\Delta\rho_{min} = -0.15$ e Å$^{-3}$ |
| 3239 reflections | Extinction correction: None |
| 415 parameters | |
| 1 restraint | |
| Primary atom site location: structure-invariant direct methods | |
| Hydrogen site location: inferred from neighbouring sites | |

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

| | *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 (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| 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, °)*

| | | | |
|---|---|---|---|
| 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, °)*

| *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 codes: (i) *x*+1, *y*, *z*.

**Fig. 1**

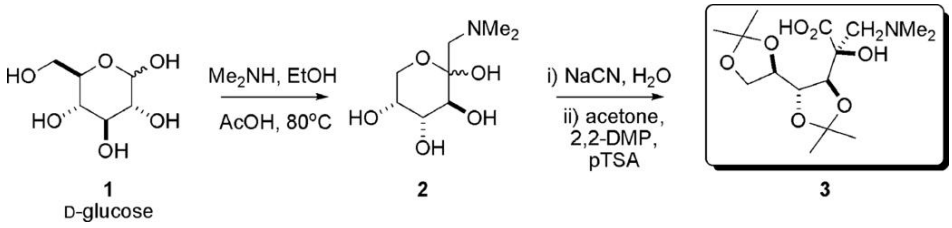
HO
O
OH
HO
OH
OH
1
D-glucose
Me2NH, EtOH
AcOH, 80°C
NMe2
O
OH
HO
OH
OH
2
i) NaCN, H2O
ii) acetone,
2,2-DMP,
pTSA
HO2C
CH2NMe2
OH
O
O
O
3

**Fig. 2**

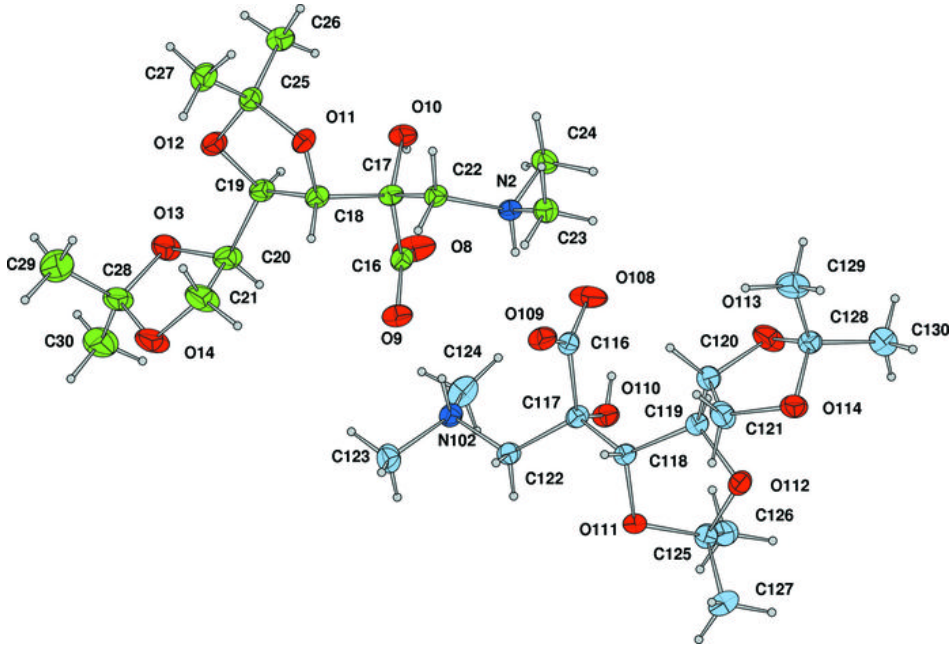
C26
C27
C25
O11
O10
O12
C24
C17
C22
N2
C19
C18
O13
C23
O8
C29
C28
C20
C16
C21
O108
C129
O109
O113
C128
C130
C116
C120
C30
O14
O9
C124
O110
C117
C119
O114
C121
N102
C123
C118
C122
O112
C126
O111
C125
C127

**Fig. 3**

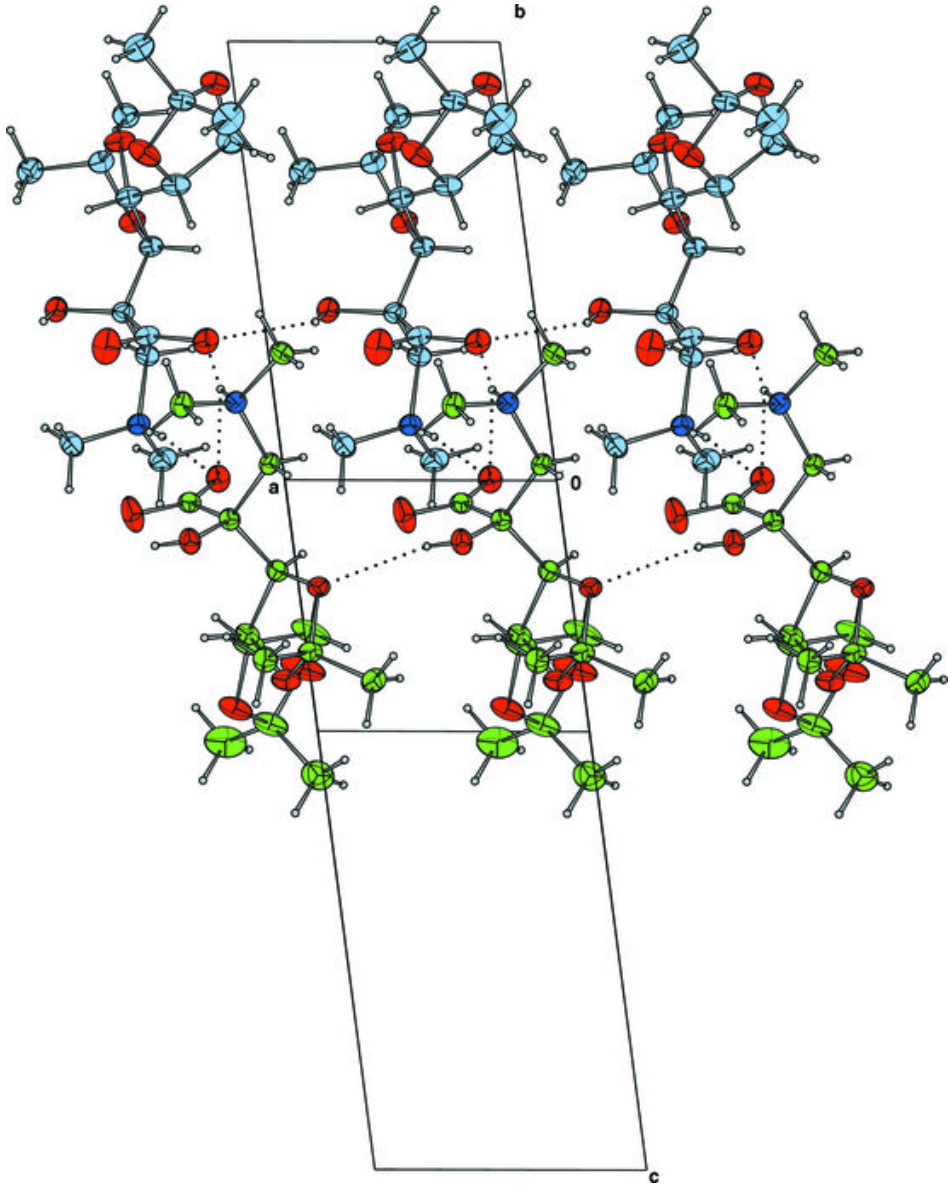

**Fig. 4**