

5-Azido-4-benzyloxy-2-methoxy-6-methylperhydropyran-3-ol

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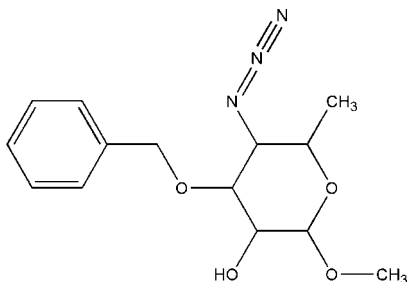
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.049; wR factor = 0.103; data-to-parameter ratio = 8.9.

In the title compound, $\text{C}_{14}\text{H}_{19}\text{N}_3\text{O}_4$, the perhydropyran ring adopts a chair conformation. An intramolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bond generates an $S(6)$ ring motif. In the crystal packing, molecules are linked by $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds, forming infinite chains along $[100]$.

Related literature

For background to D-perosamine, see: Jacquinet (2006). For the synthesis of D-perosamine, see: Krishna & Agrawal (2000). For metabolites, see: Grond *et al.* (2000). For ring conformations, see: Cremer & Pople (1975). For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For bond-length data, see: Allen *et al.* (1987). For the stability of the temperature controller used for the data collection, see: Cosier & Glazer (1986).



Experimental

Crystal data

$\text{C}_{14}\text{H}_{19}\text{N}_3\text{O}_4$

$M_r = 293.32$

Orthorhombic, $P2_12_12_1$
 $a = 4.6662$ (2) Å
 $b = 15.3356$ (8) Å
 $c = 20.9273$ (12) Å
 $V = 1497.54$ (13) Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.10$ mm⁻¹
 $T = 100$ K
 $0.27 \times 0.11 \times 0.08$ mm

Data collection

Bruker SMART APEXII CCD
 area-detector diffractometer
 Absorption correction: multi-scan
 (SADABS; Bruker, 2005)
 $T_{\min} = 0.773$, $T_{\max} = 0.980$

13029 measured reflections
 1742 independent reflections
 1334 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.096$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.049$
 $wR(F^2) = 0.103$
 $S = 1.09$
 1742 reflections
 196 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.23$ e Å⁻³
 $\Delta\rho_{\min} = -0.20$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O2}-\text{H1O2}\cdots\text{O2}^i$	0.88 (4)	1.91 (3)	2.757 (2)	161 (3)
$\text{O2}-\text{H1O2}\cdots\text{O3}^i$	0.88 (3)	2.49 (3)	3.063 (3)	124 (3)
$\text{C7}-\text{H7B}\cdots\text{O2}$	0.97	2.58	3.180 (4)	120

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

Data collection: APEX2 (Bruker, 2005); cell refinement: SAINT (Bruker, 2005); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL and PLATON (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2616).

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supplementary materials

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5-Azido-4-benzyloxy-2-methoxy-6-methylperhydropyran-3-ol

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Comment

4-Amino-4,6-dideoxy-D-mannose (*D*-perosamine) was first discovered in the polyene macrolide antibiotic perimycin and was later recognized to be present in the lipopolysaccharide (LPS) of *Vivrio cholera* (Jacquinet, 2006). Methyl 3-benzyloxy-4-azido- α -D-rhamnopyranoside is an important intermediate in the synthesis of *D*-perosamine (Krishna & Agrawal, 2000). Rhamnopyranosides were detected as metabolites from five different strains of *Streptomyces* (Grond *et al.*, 2000).

The bond lengths (Allen *et al.*, 1987) and angles in the molecule (Fig. 1) are within normal ranges. The perhydropyran ring adopts a chair conformation. The puckering parameters (Cremer & Pople, 1975) are $Q = 0.540$ (3) Å; $\Theta = 8.0$ (3) $^\circ$ and $\varphi = 8.0$ (2) $^\circ$. Intramolecular C7—H7B \cdots O2 hydrogen bonds formed a six-membered ring, producing an *S*(6) ring motif (Bernstein *et al.*, 1995). The dihedral angle formed between the benzene (C1—C6) and perhydropyran (C8—C10/O4/C11/C12) rings is 57.32 (16) $^\circ$.

In the crystal packing (Fig. 2), the molecules are linked by intermolecular O2—H1O2 \cdots O2 and O2—H1O2 \cdots O3 hydrogen bonds (Table 1) into an infinite one-dimensional chains along the [100] direction.

Experimental

To a stirred mixture of methyl 3-benzyloxy-4-methoxysulfonyl- α -D-rhamnopyranoside (0.50 g, 1.4 mmol) in DMF (5.0 ml) was added sodium azide (0.18 g, 2.8 mmol). The reaction mixture was stirred further at room temperature for 12 h. TLC (30% EtOAc/hexane, R_f-0.5) analysis showed complete conversion. The reaction mixture was concentrated under vacuum and the residue was purified by column chromatography using 25% ethylacetate in petroleum ether to get pure product as colourless crystals (yield: 300.0 mg, 71%, *M.p.* 376–378 K).

Refinement

Atom H1O2 was located in a difference map and was refined freely. Other H atoms were positioned geometrically [C—H = 0.93 to 0.98 Å] and was refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2$ or 1.5 $U_{\text{eq}}(\text{C})$. A rotating-group model was applied for the methyl groups. In the absence of significant anomalous dispersion, 1187 Friedel pairs were merged for the final refinement.

Figures

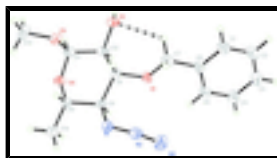


Fig. 1. The molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atom numbering scheme. Intramolecular interaction is shown by dashed line.

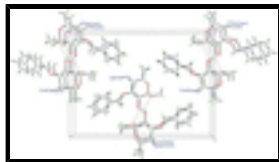


Fig. 2. The crystal packing of the title compound, viewed along a axis. Intermolecular hydrogen bonds are shown by dashed lines.

5-Azido-4-benzyloxy-2-methoxy-6-methylperhydropyran-3-ol

Crystal data

$C_{14}H_{19}N_3O_4$	$F_{000} = 624$
$M_r = 293.32$	$D_x = 1.301 \text{ Mg m}^{-3}$
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Hall symbol: P 2ac 2ab	Cell parameters from 1880 reflections
$a = 4.6662 (2) \text{ \AA}$	$\theta = 2.4\text{--}29.9^\circ$
$b = 15.3356 (8) \text{ \AA}$	$\mu = 0.10 \text{ mm}^{-1}$
$c = 20.9273 (12) \text{ \AA}$	$T = 100 \text{ K}$
$V = 1497.54 (13) \text{ \AA}^3$	Block, colourless
$Z = 4$	$0.27 \times 0.11 \times 0.08 \text{ mm}$

Data collection

Bruker SMART APEXII CCD area-detector diffractometer	1742 independent reflections
Radiation source: fine-focus sealed tube	1334 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.096$
$T = 100 \text{ K}$	$\theta_{\text{max}} = 26.0^\circ$
φ and ω scans	$\theta_{\text{min}} = 2.0^\circ$
Absorption correction: multi-scan (SADABS; Bruker, 2005)	$h = -5 \rightarrow 5$
$T_{\text{min}} = 0.773$, $T_{\text{max}} = 0.980$	$k = -18 \rightarrow 17$
13029 measured reflections	$l = -25 \rightarrow 25$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.049$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.103$	$w = 1/[\sigma^2(F_o^2) + (0.0499P)^2]$
$S = 1.09$	where $P = (F_o^2 + 2F_c^2)/3$
1742 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
196 parameters	$\Delta\rho_{\text{max}} = 0.23 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.20 \text{ e \AA}^{-3}$
	Extinction correction: none

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cyrosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.0881 (4)	0.40074 (13)	0.13521 (10)	0.0229 (5)
O2	0.0911 (5)	0.28256 (14)	0.02574 (11)	0.0251 (5)
O3	0.3176 (5)	0.39026 (14)	-0.06657 (9)	0.0236 (5)
O4	-0.0200 (5)	0.49881 (14)	-0.04434 (10)	0.0250 (5)
N1	0.3572 (6)	0.56890 (17)	0.10102 (12)	0.0260 (7)
N2	0.3482 (6)	0.56020 (18)	0.16023 (14)	0.0306 (7)
N3	0.3680 (9)	0.5600 (2)	0.21422 (15)	0.0537 (11)
C1	0.2228 (8)	0.2487 (2)	0.26646 (17)	0.0341 (9)
H1A	0.3517	0.2100	0.2479	0.041*
C2	0.1203 (8)	0.2318 (2)	0.32682 (17)	0.0389 (9)
H2A	0.1795	0.1820	0.3485	0.047*
C3	-0.0706 (8)	0.2888 (3)	0.35523 (17)	0.0379 (9)
H3A	-0.1405	0.2776	0.3960	0.045*
C4	-0.1561 (9)	0.3619 (2)	0.32269 (16)	0.0362 (9)
H4A	-0.2856	0.4003	0.3413	0.043*
C5	-0.0489 (8)	0.3786 (2)	0.26165 (16)	0.0314 (9)
H5A	-0.1051	0.4289	0.2402	0.038*
C6	0.1390 (7)	0.3216 (2)	0.23289 (15)	0.0245 (7)
C7	0.2586 (8)	0.3380 (2)	0.16733 (16)	0.0309 (9)
H7A	0.4541	0.3590	0.1708	0.037*
H7B	0.2607	0.2840	0.1431	0.037*
C8	0.1803 (7)	0.4219 (2)	0.07207 (14)	0.0209 (7)
H8A	0.3837	0.4073	0.0675	0.025*
C9	0.1415 (7)	0.51995 (19)	0.06387 (14)	0.0193 (7)
H9A	-0.0498	0.5361	0.0791	0.023*
C10	0.1727 (7)	0.5493 (2)	-0.00515 (14)	0.0215 (7)
H10A	0.3703	0.5397	-0.0193	0.026*
C11	0.0416 (7)	0.4086 (2)	-0.04432 (15)	0.0241 (8)
H11A	-0.0965	0.3794	-0.0723	0.029*
C12	0.0088 (7)	0.37161 (19)	0.02287 (15)	0.0214 (7)

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H12A	-0.1941	0.3754	0.0347	0.026*
C13	0.3436 (8)	0.4054 (3)	-0.13393 (15)	0.0361 (9)
H13A	0.5309	0.3880	-0.1480	0.054*
H13B	0.3159	0.4662	-0.1427	0.054*
H13C	0.2012	0.3720	-0.1562	0.054*
C14	0.0949 (9)	0.6437 (2)	-0.01442 (16)	0.0364 (9)
H14A	0.1146	0.6588	-0.0587	0.055*
H14B	0.2203	0.6795	0.0107	0.055*
H14C	-0.0997	0.6530	-0.0012	0.055*
H1O2	-0.060 (8)	0.251 (2)	0.0151 (15)	0.028 (10)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0225 (12)	0.0219 (12)	0.0243 (11)	0.0014 (10)	0.0055 (10)	0.0037 (10)
O2	0.0200 (12)	0.0185 (12)	0.0368 (13)	-0.0030 (11)	0.0015 (11)	-0.0055 (10)
O3	0.0222 (11)	0.0283 (13)	0.0205 (11)	0.0038 (11)	-0.0013 (10)	-0.0046 (10)
O4	0.0249 (12)	0.0218 (12)	0.0282 (12)	0.0030 (11)	-0.0041 (10)	-0.0033 (10)
N1	0.0301 (16)	0.0255 (17)	0.0224 (16)	-0.0079 (14)	0.0009 (14)	-0.0004 (12)
N2	0.0360 (17)	0.0250 (17)	0.0307 (19)	-0.0096 (14)	-0.0032 (15)	-0.0024 (13)
N3	0.084 (3)	0.049 (2)	0.0274 (18)	-0.028 (2)	-0.0075 (19)	-0.0015 (15)
C1	0.036 (2)	0.032 (2)	0.035 (2)	0.0072 (17)	0.0014 (17)	0.0053 (17)
C2	0.044 (2)	0.032 (2)	0.040 (2)	0.003 (2)	0.000 (2)	0.0127 (18)
C3	0.040 (2)	0.045 (2)	0.0295 (19)	-0.002 (2)	0.0026 (18)	0.0106 (18)
C4	0.040 (2)	0.036 (2)	0.0329 (19)	0.0034 (19)	0.0131 (19)	0.0041 (17)
C5	0.038 (2)	0.0257 (19)	0.0300 (19)	0.0002 (18)	0.0039 (18)	0.0061 (16)
C6	0.0246 (18)	0.0220 (18)	0.0267 (17)	-0.0019 (16)	-0.0029 (16)	0.0018 (15)
C7	0.0305 (19)	0.031 (2)	0.0315 (19)	0.0135 (17)	0.0029 (16)	0.0033 (16)
C8	0.0170 (16)	0.0213 (18)	0.0244 (17)	-0.0001 (15)	0.0013 (15)	0.0015 (14)
C9	0.0197 (16)	0.0150 (16)	0.0233 (16)	0.0001 (14)	0.0031 (15)	-0.0037 (13)
C10	0.0238 (17)	0.0185 (17)	0.0224 (16)	-0.0001 (15)	0.0005 (15)	-0.0037 (14)
C11	0.0224 (16)	0.0213 (18)	0.0286 (18)	-0.0019 (15)	-0.0045 (15)	-0.0047 (15)
C12	0.0153 (15)	0.0157 (16)	0.0333 (18)	0.0017 (14)	0.0013 (15)	-0.0062 (14)
C13	0.035 (2)	0.052 (2)	0.0214 (17)	0.004 (2)	-0.0043 (18)	-0.0014 (17)
C14	0.052 (2)	0.028 (2)	0.0291 (19)	0.0020 (19)	0.0024 (19)	0.0017 (16)

Geometric parameters (\AA , $^\circ$)

O1—C7	1.418 (4)	C5—H5A	0.9300
O1—C8	1.427 (4)	C6—C7	1.502 (4)
O2—C12	1.420 (3)	C7—H7A	0.9700
O2—H1O2	0.88 (4)	C7—H7B	0.9700
O3—C11	1.398 (4)	C8—C12	1.515 (4)
O3—C13	1.434 (4)	C8—C9	1.524 (4)
O4—C11	1.412 (4)	C8—H8A	0.9800
O4—C10	1.443 (4)	C9—C10	1.520 (4)
N1—N2	1.247 (4)	C9—H9A	0.9800
N1—C9	1.477 (4)	C10—C14	1.505 (4)
N2—N3	1.134 (4)	C10—H10A	0.9800

C1—C2	1.375 (5)	C11—C12	1.524 (4)
C1—C6	1.376 (5)	C11—H11A	0.9800
C1—H1A	0.9300	C12—H12A	0.9800
C2—C3	1.382 (5)	C13—H13A	0.9600
C2—H2A	0.9300	C13—H13B	0.9600
C3—C4	1.371 (5)	C13—H13C	0.9600
C3—H3A	0.9300	C14—H14A	0.9600
C4—C5	1.396 (5)	C14—H14B	0.9600
C4—H4A	0.9300	C14—H14C	0.9600
C5—C6	1.378 (5)		
C7—O1—C8	115.1 (2)	N1—C9—C10	106.5 (2)
C12—O2—H1O2	107 (2)	N1—C9—C8	111.2 (3)
C11—O3—C13	111.9 (2)	C10—C9—C8	112.8 (2)
C11—O4—C10	113.5 (2)	N1—C9—H9A	108.7
N2—N1—C9	116.5 (3)	C10—C9—H9A	108.7
N3—N2—N1	171.1 (4)	C8—C9—H9A	108.7
C2—C1—C6	121.5 (3)	O4—C10—C14	107.0 (3)
C2—C1—H1A	119.2	O4—C10—C9	108.8 (2)
C6—C1—H1A	119.2	C14—C10—C9	112.6 (3)
C1—C2—C3	120.0 (3)	O4—C10—H10A	109.5
C1—C2—H2A	120.0	C14—C10—H10A	109.5
C3—C2—H2A	120.0	C9—C10—H10A	109.5
C4—C3—C2	119.4 (3)	O3—C11—O4	112.6 (3)
C4—C3—H3A	120.3	O3—C11—C12	109.0 (3)
C2—C3—H3A	120.3	O4—C11—C12	110.2 (3)
C3—C4—C5	120.1 (3)	O3—C11—H11A	108.3
C3—C4—H4A	120.0	O4—C11—H11A	108.3
C5—C4—H4A	120.0	C12—C11—H11A	108.3
C6—C5—C4	120.7 (3)	O2—C12—C8	108.5 (2)
C6—C5—H5A	119.6	O2—C12—C11	111.7 (2)
C4—C5—H5A	119.6	C8—C12—C11	112.6 (3)
C1—C6—C5	118.3 (3)	O2—C12—H12A	107.9
C1—C6—C7	119.8 (3)	C8—C12—H12A	107.9
C5—C6—C7	121.9 (3)	C11—C12—H12A	107.9
O1—C7—C6	109.8 (3)	O3—C13—H13A	109.5
O1—C7—H7A	109.7	O3—C13—H13B	109.5
C6—C7—H7A	109.7	H13A—C13—H13B	109.5
O1—C7—H7B	109.7	O3—C13—H13C	109.5
C6—C7—H7B	109.7	H13A—C13—H13C	109.5
H7A—C7—H7B	108.2	H13B—C13—H13C	109.5
O1—C8—C12	110.8 (2)	C10—C14—H14A	109.5
O1—C8—C9	107.0 (2)	C10—C14—H14B	109.5
C12—C8—C9	111.3 (3)	H14A—C14—H14B	109.5
O1—C8—H8A	109.2	C10—C14—H14C	109.5
C12—C8—H8A	109.2	H14A—C14—H14C	109.5
C9—C8—H8A	109.2	H14B—C14—H14C	109.5
C9—N1—N2—N3	-177 (2)	C12—C8—C9—C10	-47.1 (4)
C6—C1—C2—C3	-0.2 (6)	C11—O4—C10—C14	175.5 (3)

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C1—C2—C3—C4	0.0 (6)	C11—O4—C10—C9	-62.7 (3)
C2—C3—C4—C5	-0.5 (6)	N1—C9—C10—O4	176.0 (2)
C3—C4—C5—C6	1.2 (6)	C8—C9—C10—O4	53.7 (3)
C2—C1—C6—C5	0.9 (5)	N1—C9—C10—C14	-65.6 (3)
C2—C1—C6—C7	179.4 (3)	C8—C9—C10—C14	172.2 (3)
C4—C5—C6—C1	-1.4 (5)	C13—O3—C11—O4	-70.6 (3)
C4—C5—C6—C7	-179.8 (3)	C13—O3—C11—C12	166.9 (2)
C8—O1—C7—C6	-179.9 (3)	C10—O4—C11—O3	-59.5 (3)
C1—C6—C7—O1	163.7 (3)	C10—O4—C11—C12	62.4 (3)
C5—C6—C7—O1	-17.8 (5)	O1—C8—C12—O2	-71.0 (3)
C7—O1—C8—C12	98.3 (3)	C9—C8—C12—O2	170.1 (3)
C7—O1—C8—C9	-140.2 (3)	O1—C8—C12—C11	164.8 (2)
N2—N1—C9—C10	173.4 (3)	C9—C8—C12—C11	45.9 (3)
N2—N1—C9—C8	-63.3 (4)	O3—C11—C12—O2	-51.3 (3)
O1—C8—C9—N1	72.2 (3)	O4—C11—C12—O2	-175.3 (2)
C12—C8—C9—N1	-166.7 (2)	O3—C11—C12—C8	71.1 (3)
O1—C8—C9—C10	-168.2 (2)	O4—C11—C12—C8	-52.9 (3)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O2—H1O2 \cdots O2 ⁱ	0.88 (4)	1.91 (3)	2.757 (2)	161 (3)
O2—H1O2 \cdots O3 ⁱ	0.88 (3)	2.49 (3)	3.063 (3)	124 (3)
C7—H7B \cdots O2	0.97	2.58	3.180 (4)	120

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

Fig. 1

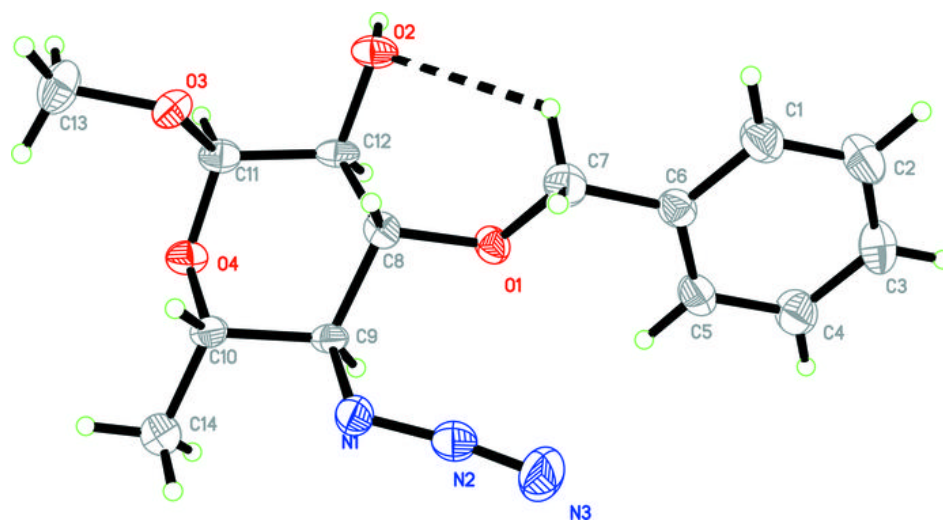


Fig. 2

