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## Structure Reports

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**tert-Butyl N-(3,6-dihydro-1,2-dioxin-4-ylmethyl)carbamate**Daniel Sejer Pedersen,<sup>a</sup> Dennis K. Taylor<sup>a‡</sup> and Edward R. T. Tiekink<sup>b\*</sup><sup>a</sup>Department of Chemistry, The University of Adelaide, 5005 South Australia, Australia, and <sup>b</sup>Department of Chemistry, The University of Texas at San Antonio, One UTSA Circle, San Antonio, Texas 78249-0698, USA  
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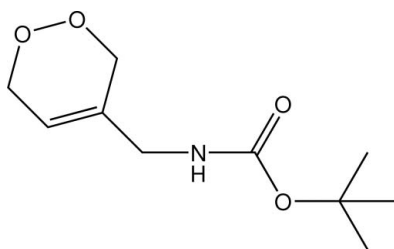
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Key indicators: single-crystal X-ray study;  $T = 120$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å; disorder in main residue;  $R$  factor = 0.038;  $wR$  factor = 0.100; data-to-parameter ratio = 9.4.

The six-membered ring in the title compound,  $\text{C}_{10}\text{H}_{17}\text{NO}_4$ , has a half-chair conformation and lies to one side of the central chromophore. Molecules are connected into a supramolecular chain *via*  $\text{N}-\text{H}\cdots\text{O}$  hydrogen-bonding interactions and these are consolidated into a three-dimensional structure *via*  $\text{C}-\text{H}\cdots\text{O}$  interactions. Two C atoms and two O atoms in the ring are disordered over two positions; the site occupancy factors are *ca* 0.7 and 0.3.

## Related literature

For the synthesis of the *t*-butyl 2-formylallylcarbamate reactant, see Erkkilä & Pihko (2006). For the conversion of 1,2-dioxines to carbohydrates, see Robinson *et al.* (2006) and Pedersen & Rosenbohm (2001) for some experimental techniques. For calculations on ring puckering, see Cremer & Pople (1975).



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## Experimental

## Crystal data

 $\text{C}_{10}\text{H}_{17}\text{NO}_4$   
 $M_r = 215.25$   
Orthorhombic,  $P2_12_12_1$   
 $a = 9.435$  (4) Å  
 $b = 9.628$  (4) Å  
 $c = 12.698$  (6) Å  
 $V = 1153.6$  (9) Å<sup>3</sup>  
 $Z = 4$   
Mo  $K\alpha$  radiation  
 $\mu = 0.10$  mm<sup>-1</sup>  
 $T = 120$  (2) K  
 $0.40 \times 0.40 \times 0.10$  mm

## Data collection

Rigaku AFC12 $\kappa$ /SATURN724 diffractometer  
Absorption correction: none  
11678 measured reflections  
1369 independent reflections  
1347 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.027$ 

## Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.038$   
 $wR(F^2) = 0.100$   
 $S = 1.07$   
1369 reflections  
145 parameters  
H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.14$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.16$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N1}-\text{H1}\cdots\text{O3}^{\text{i}}$	0.88	2.16	2.984 (2)	156
$\text{C3}-\text{H3A}\cdots\text{O3}^{\text{ii}}$	0.99	2.52	3.425 (3)	152
$\text{C4}-\text{H4B}\cdots\text{O4}^{\text{iii}}$	0.99	2.55	3.446 (3)	150

Symmetry codes: (i)  $-x + 1, y + \frac{1}{2}, -z + \frac{3}{2}$ ; (ii)  $-x + \frac{3}{2}, -y + 1, z + \frac{1}{2}$ ; (iii)  $-x + 1, y - \frac{1}{2}, -z + \frac{3}{2}$ .

Data collection: *CrystalClear* (Rigaku Americas Corporation, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *SHELXL97*.

We are grateful to the Australian Research Council for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2332).

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**supplementary materials**

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### ***tert*-Butyl *N*-(3,6-dihydro-1,2-dioxin-4-ylmethyl)carbamate**

**D. Sejer Pedersen, D. K. Taylor and E. R. T. Tiekink**

#### **Comment**

Recently, we reported that 1,2-dioxines can be converted to a wide variety of carbohydrates by dihydroxylation of the olefin, followed by cleavage of the peroxide bond (Robinson *et al.*, 2006). To extend this methodology further, a number of amino dioxines were synthesized, such as the title compound, C<sub>10</sub>H<sub>17</sub>NO<sub>4</sub> (I), that could potentially be converted to amino sugars by the same method.

The molecular structure of (I), Fig. 1, features a six-membered ring with a half-chair conformation so that the O atoms lie on either side of the plane defined by the four C atoms. The ring is characterized by a puckering amplitude = 0.618 (2) Å, and values of  $\theta = 52.10$  (19)° and  $\varphi = 28.0$  (3)° (Cremer & Pople, 1975). The overall conformation of the molecule is such that the ring lies to one side of the central chromophore. Molecules are connected into a supramolecular chain aligned along the *b* axis by N—H⋯O hydrogen bonding interactions (Fig. 2 & Table 1). Chains are consolidated into a 3-D structure via C—H⋯O interactions (Fig. 3).

#### **Experimental**

Diphenylmethylphosphonium iodide (41.8 g, 103 mmol) was suspended in anhydrous THF (350 ml) and cooled to 0 °C. Potassium *tert*-butoxide (11.56 g, 103 mmol) was added in one portion to give a yellow slurry that was allowed to warm to room temperature. *tert*-butyl 2-formylallylcarbamate (Erkkilä & Pihko, 2006) (17.4 g, 94 mmol) was dissolved in anhydrous THF (150 ml) and added to the above suspension by cannula. The resulting mixture was stirred vigorously overnight under an atmosphere of nitrogen. Half saturated aqueous ammonium chloride (50 ml) was added and the THF removed *in vacuo*. The aqueous phase was transferred to a separatory funnel with water (200 ml) and extracted with 20% ethyl acetate in hexanes, *v/v* (200 ml + 3 x 100 ml). The combined organic phases were dried (Na<sub>2</sub>SO<sub>4</sub>), filtered and concentrated *in vacuo* to give a yellow gum. Purification by Dry Column Vacuum Chromatography (Pedersen & Rosenbohm, 2001) (id 6 cm; 20 ml fractions; 10 x hexanes; 2–20% EtOAc in hexanes, *v/v* – 2% increments; 10 x 20% EtOAc in hexanes, *v/v*) gave *tert*-butyl 2-methylenebut-3-enylcarbamate (3.0 g, 17%) as a tan liquid. This diene (3.0 g, 16.4 mmol) was dissolved in dichloromethane (90 ml), Rose Bengal bis-triethylammonium salt (100 mg) was added and the solution photolysed for 24 h whilst a stream of oxygen was bubbled through the solution. The solvent was removed *in vacuo* to give a pink gum that was purified by Dry Column Vacuum Chromatography (id 4 cm; 20 ml fractions; 8 x hexanes; 5 – 60% EtOAc in hexanes, *v/v* – 5% increments) to give dioxine (I) (0.90 g, 26%) as needles.

#### **Refinement**

All H atoms were included in the riding-model approximation, with C—H = 0.95 to 0.99 Å and N—H = 0.88 Å, and with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C, N})$  and  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{methyl-C})$ . Disorder was noted in the C4—O1—O2—C4 residue of the six-membered ring and two positions were discerned for the O atoms. The major component (anisotropic) had a site

# supplementary materials

occupancy factor = 0.706 (5). The absolute structure could not be determined with confidence so the Friedel pairs were averaged.

## Figures

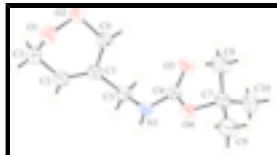


Fig. 1. Molecular structure of (I) showing atom-labelling scheme and displacement ellipsoids at the 50% probability level. The minor component of the disorder was omitted for clarity.

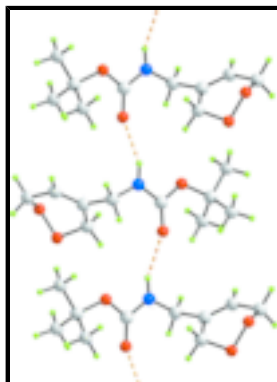


Fig. 2. View of the supramolecular chain in (I) mediated by N—H...O hydrogen bonds, shown as orange-dashed lines. Colour code: red (oxygen), blue (nitrogen), grey (carbon) and green (hydrogen).

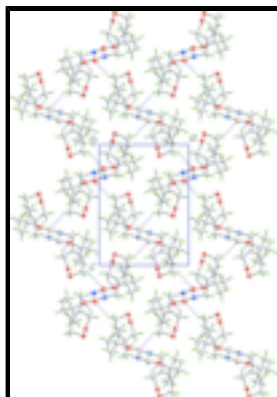


Fig. 3. View of the unit-cell contents of (I) down the *b* axis. Colour code for atoms as for Fig. 2. Hydrogen bonding contacts of the type N—H...O and C—H...O are shown as orange- and blue-dashed lines, respectively.

## *tert*-Butyl *N*-(3,6-dihydro-1,2-dioxin-4-ylmethyl)carbamate

### Crystal data

C<sub>10</sub>H<sub>17</sub>NO<sub>4</sub>

*M<sub>r</sub>* = 215.25

Orthorhombic, *P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>

Hall symbol: *P* 2ac 2ab

*a* = 9.435 (4) Å

*b* = 9.628 (4) Å

*c* = 12.698 (6) Å

*V* = 1153.6 (9) Å<sup>3</sup>

*Z* = 4

*F*<sub>000</sub> = 464

*D<sub>x</sub>* = 1.239 Mg m<sup>-3</sup>

Mo *K*α radiation

λ = 0.71070 Å

Cell parameters from 4236 reflections

θ = 2.2–30.4°

μ = 0.10 mm<sup>-1</sup>

*T* = 120 (2) K

Prism, colorless

0.40 × 0.40 × 0.10 mm

*Data collection*

Rigaku AFC12κ/SATURN724 diffractometer	1347 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{\text{int}} = 0.027$
Monochromator: graphite	$\theta_{\text{max}} = 26.5^\circ$
$T = 120(2)$ K	$\theta_{\text{min}} = 2.7^\circ$
$\omega$ scans	$h = -10 \rightarrow 11$
Absorption correction: none	$k = -10 \rightarrow 12$
11678 measured reflections	$l = -15 \rightarrow 15$
1369 independent reflections	

*Refinement*

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.038$	H-atom parameters constrained
$wR(F^2) = 0.100$	$w = 1/[\sigma^2(F_o^2) + (0.0581P)^2 + 0.2125P]$
$S = 1.07$	where $P = (F_o^2 + 2F_c^2)/3$
1369 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
145 parameters	$\Delta\rho_{\text{max}} = 0.14 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.16 \text{ e } \text{\AA}^{-3}$
	Extinction correction: none

*Special details*

**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  $F^2 > 2\sigma(F^2)$  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 ( $\text{\AA}^2$ )*

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
O3	0.45338 (14)	0.52172 (13)	0.69574 (12)	0.0403 (3)	
O4	0.33495 (14)	0.72768 (13)	0.68622 (11)	0.0370 (3)	
C3	0.8147 (3)	0.6760 (3)	1.05230 (18)	0.0568 (6)	0.706 (5)
H3A	0.8892	0.6070	1.0690	0.068*	0.706 (5)
H3B	0.8385	0.7636	1.0891	0.068*	0.706 (5)
C4	0.6062 (2)	0.5477 (2)	0.92708 (15)	0.0393 (4)	0.706 (5)

## supplementary materials

H4A	0.5147	0.5971	0.9347	0.047*	0.706 (5)
H4B	0.5905	0.4671	0.8801	0.047*	0.706 (5)
O1	0.6742 (3)	0.6237 (2)	1.09082 (16)	0.0500 (8)	0.706 (5)
O2	0.6513 (2)	0.4980 (2)	1.02975 (15)	0.0429 (7)	0.706 (5)
C3A	0.8147 (3)	0.6760 (3)	1.05230 (18)	0.0568 (6)	0.294 (5)
H3C	0.9137	0.6753	1.0782	0.068*	0.294 (5)
H3D	0.7621	0.7501	1.0896	0.068*	0.294 (5)
C4A	0.6062 (2)	0.5477 (2)	0.92708 (15)	0.0393 (4)	0.294 (5)
H4C	0.5113	0.5591	0.8949	0.047*	0.294 (5)
H4D	0.6361	0.4497	0.9199	0.047*	0.294 (5)
O1A	0.7525 (6)	0.5504 (6)	1.0696 (4)	0.0480 (18)*	0.294 (5)
O2A	0.6048 (6)	0.5901 (6)	1.0373 (4)	0.0443 (16)*	0.294 (5)
N1	0.56110 (16)	0.72726 (15)	0.73168 (12)	0.0330 (3)	
H1	0.5514	0.8181	0.7329	0.040*	
C1	0.71146 (19)	0.64320 (18)	0.87711 (14)	0.0317 (4)	
C2	0.8118 (2)	0.7010 (2)	0.93588 (16)	0.0402 (5)	
H2	0.8817	0.7582	0.9041	0.048*	
C5	0.69754 (18)	0.6658 (2)	0.76017 (14)	0.0336 (4)	
H5A	0.7748	0.7278	0.7360	0.040*	
H5B	0.7083	0.5757	0.7235	0.040*	
C6	0.45012 (19)	0.64797 (18)	0.70358 (13)	0.0314 (4)	
C7	0.2026 (2)	0.6673 (2)	0.64337 (17)	0.0412 (5)	
C8	0.1393 (2)	0.5642 (2)	0.7209 (2)	0.0512 (6)	
H8A	0.2016	0.4832	0.7268	0.077*	
H8B	0.0458	0.5347	0.6960	0.077*	
H8C	0.1298	0.6084	0.7901	0.077*	
C9	0.1067 (2)	0.7929 (3)	0.6341 (3)	0.0623 (7)	
H9A	0.0893	0.8317	0.7042	0.093*	
H9B	0.0164	0.7651	0.6022	0.093*	
H9C	0.1523	0.8632	0.5897	0.093*	
C10	0.2326 (3)	0.6033 (3)	0.53635 (19)	0.0593 (7)	
H10A	0.2947	0.5225	0.5450	0.089*	
H10B	0.2792	0.6721	0.4912	0.089*	
H10C	0.1433	0.5740	0.5038	0.089*	

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O3	0.0397 (7)	0.0282 (7)	0.0530 (8)	-0.0002 (5)	-0.0028 (6)	-0.0033 (6)
O4	0.0308 (6)	0.0281 (6)	0.0521 (7)	-0.0014 (5)	-0.0070 (6)	-0.0002 (6)
C3	0.0668 (15)	0.0525 (13)	0.0513 (12)	-0.0174 (13)	-0.0236 (12)	0.0077 (10)
C4	0.0340 (9)	0.0463 (11)	0.0376 (9)	-0.0043 (8)	-0.0034 (8)	0.0026 (8)
O1	0.0606 (15)	0.0528 (14)	0.0366 (10)	-0.0119 (12)	0.0015 (11)	-0.0054 (9)
O2	0.0519 (12)	0.0383 (12)	0.0384 (10)	-0.0098 (10)	-0.0043 (9)	0.0065 (8)
C3A	0.0668 (15)	0.0525 (13)	0.0513 (12)	-0.0174 (13)	-0.0236 (12)	0.0077 (10)
C4A	0.0340 (9)	0.0463 (11)	0.0376 (9)	-0.0043 (8)	-0.0034 (8)	0.0026 (8)
N1	0.0326 (8)	0.0274 (7)	0.0390 (8)	-0.0008 (6)	-0.0047 (6)	0.0008 (6)
C1	0.0297 (8)	0.0268 (8)	0.0386 (9)	0.0035 (7)	-0.0015 (7)	-0.0013 (7)

C2	0.0384 (10)	0.0337 (9)	0.0484 (10)	-0.0047 (9)	-0.0084 (9)	0.0022 (8)
C5	0.0274 (8)	0.0344 (9)	0.0391 (9)	-0.0002 (7)	0.0009 (7)	0.0005 (7)
C6	0.0330 (9)	0.0282 (8)	0.0329 (8)	-0.0008 (7)	-0.0008 (7)	0.0002 (7)
C7	0.0292 (9)	0.0395 (10)	0.0551 (11)	-0.0065 (8)	-0.0074 (9)	0.0022 (9)
C8	0.0383 (10)	0.0475 (12)	0.0677 (14)	-0.0083 (9)	0.0038 (10)	0.0065 (11)
C9	0.0337 (10)	0.0512 (13)	0.102 (2)	-0.0017 (11)	-0.0148 (12)	0.0145 (14)
C10	0.0469 (12)	0.0782 (18)	0.0527 (12)	-0.0201 (13)	-0.0100 (10)	-0.0062 (12)

*Geometric parameters (Å, °)*

O3—C6	1.220 (2)	N1—C6	1.344 (2)
O4—C6	1.349 (2)	N1—C5	1.462 (2)
O4—C7	1.481 (2)	N1—H1	0.8800
C3—O1	1.500 (4)	C1—C2	1.327 (3)
C3—C2	1.498 (3)	C1—C5	1.507 (3)
C3—H3A	0.9900	C2—H2	0.9500
C3—H3B	0.9900	C5—H5A	0.9900
C4—O2	1.453 (3)	C5—H5B	0.9900
C4—C1	1.495 (3)	C7—C9	1.515 (3)
C4—H4A	0.9900	C7—C10	1.519 (3)
C4—H4B	0.9900	C7—C8	1.520 (3)
O1—O2	1.454 (3)	C8—H8A	0.9800
C3A—O1A	1.362 (6)	C8—H8B	0.9800
C3A—C2	1.498 (3)	C8—H8C	0.9800
C3A—H3C	0.9900	C9—H9A	0.9800
C3A—H3D	0.9900	C9—H9B	0.9800
C4A—O2A	1.458 (6)	C9—H9C	0.9800
C4A—C1	1.495 (3)	C10—H10A	0.9800
C4A—H4C	0.9900	C10—H10B	0.9800
C4A—H4D	0.9900	C10—H10C	0.9800
O1A—O2A	1.502 (8)		
C6—O4—C7	121.07 (14)	C1—C2—C3A	120.0 (2)
O1—C3—C2	111.0 (2)	C1—C2—H2	120.0
O1—C3—H3A	109.4	C3—C2—H2	120.0
C2—C3—H3A	109.4	C3A—C2—H2	120.0
O1—C3—H3B	109.4	N1—C5—C1	112.28 (15)
C2—C3—H3B	109.4	N1—C5—H5A	109.1
H3A—C3—H3B	108.0	C1—C5—H5A	109.1
O2—C4—C1	112.89 (17)	N1—C5—H5B	109.1
O2—C4—H4A	109.0	C1—C5—H5B	109.1
C1—C4—H4A	109.0	H5A—C5—H5B	107.9
O2—C4—H4B	109.0	O3—C6—N1	124.61 (17)
C1—C4—H4B	109.0	O3—C6—O4	125.03 (17)
H4A—C4—H4B	107.8	N1—C6—O4	110.36 (14)
O2—O1—C3	103.7 (2)	O4—C7—C9	102.64 (17)
C4—O2—O1	104.33 (18)	O4—C7—C10	109.30 (17)
O1A—C3A—C2	107.1 (3)	C9—C7—C10	111.4 (2)
O1A—C3A—H3C	110.3	O4—C7—C8	110.44 (17)
C2—C3A—H3C	110.3	C9—C7—C8	109.71 (19)

## supplementary materials

O1A—C3A—H3D	110.3	C10—C7—C8	112.8 (2)
C2—C3A—H3D	110.3	C7—C8—H8A	109.5
H3C—C3A—H3D	108.6	C7—C8—H8B	109.5
O2A—C4A—C1	104.0 (3)	H8A—C8—H8B	109.5
O2A—C4A—H4C	111.0	C7—C8—H8C	109.5
C1—C4A—H4C	111.0	H8A—C8—H8C	109.5
O2A—C4A—H4D	111.0	H8B—C8—H8C	109.5
C1—C4A—H4D	111.0	C7—C9—H9A	109.5
H4C—C4A—H4D	109.0	C7—C9—H9B	109.5
C3A—O1A—O2A	97.5 (4)	H9A—C9—H9B	109.5
C4A—O2A—O1A	100.5 (4)	C7—C9—H9C	109.5
C6—N1—C5	121.46 (14)	H9A—C9—H9C	109.5
C6—N1—H1	119.3	H9B—C9—H9C	109.5
C5—N1—H1	119.3	C7—C10—H10A	109.5
C2—C1—C4	119.53 (17)	C7—C10—H10B	109.5
C2—C1—C4A	119.53 (17)	H10A—C10—H10B	109.5
C2—C1—C5	123.74 (18)	C7—C10—H10C	109.5
C4—C1—C5	116.71 (16)	H10A—C10—H10C	109.5
C4A—C1—C5	116.71 (16)	H10B—C10—H10C	109.5
C1—C2—C3	120.0 (2)		
C2—C3—O1—O2	58.0 (2)	C5—C1—C2—C3A	178.1 (2)
C1—C4—O2—O1	57.4 (2)	O1—C3—C2—C1	-18.2 (3)
C3—O1—O2—C4	-77.6 (2)	O1—C3—C2—C3A	0(100)
C2—C3A—O1A—O2A	-67.3 (4)	O1A—C3A—C2—C1	27.2 (4)
C1—C4A—O2A—O1A	-66.0 (4)	O1A—C3A—C2—C3	0(100)
C3A—O1A—O2A—C4A	93.9 (4)	C6—N1—C5—C1	-93.6 (2)
O2—C4—C1—C2	-16.9 (3)	C2—C1—C5—N1	-120.2 (2)
O2—C4—C1—C4A	0(11)	C4—C1—C5—N1	60.9 (2)
O2—C4—C1—C5	162.06 (17)	C4A—C1—C5—N1	60.9 (2)
O2A—C4A—C1—C2	24.3 (3)	C5—N1—C6—O3	-2.6 (3)
O2A—C4A—C1—C4	0(38)	C5—N1—C6—O4	176.90 (14)
O2A—C4A—C1—C5	-156.7 (3)	C7—O4—C6—O3	-7.0 (3)
C4—C1—C2—C3	-3.0 (3)	C7—O4—C6—N1	173.47 (15)
C4A—C1—C2—C3	-3.0 (3)	C6—O4—C7—C9	-178.27 (18)
C5—C1—C2—C3	178.1 (2)	C6—O4—C7—C10	-59.9 (2)
C4—C1—C2—C3A	-3.0 (3)	C6—O4—C7—C8	64.8 (2)
C4A—C1—C2—C3A	-3.0 (3)		

### Hydrogen-bond geometry ( $\text{\AA}$ , $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1 $\cdots$ O3 <sup>i</sup>	0.88	2.16	2.984 (2)	156
C3—H3A $\cdots$ O3 <sup>ii</sup>	0.99	2.52	3.425 (3)	152
C4—H4B $\cdots$ O4 <sup>iii</sup>	0.99	2.55	3.446 (3)	150

Symmetry codes: (i)  $-x+1, y+1/2, -z+3/2$ ; (ii)  $-x+3/2, -y+1, z+1/2$ ; (iii)  $-x+1, y-1/2, -z+3/2$ .



Fig. 1

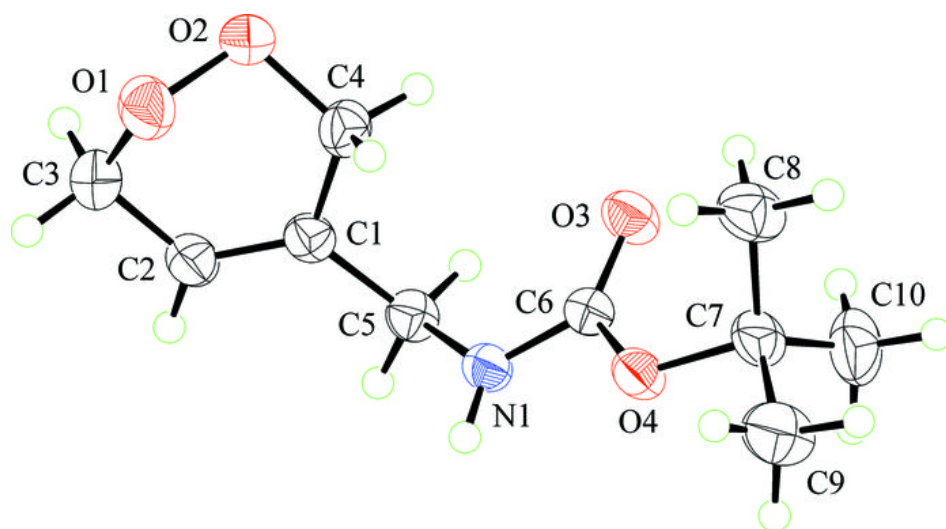


Fig. 2

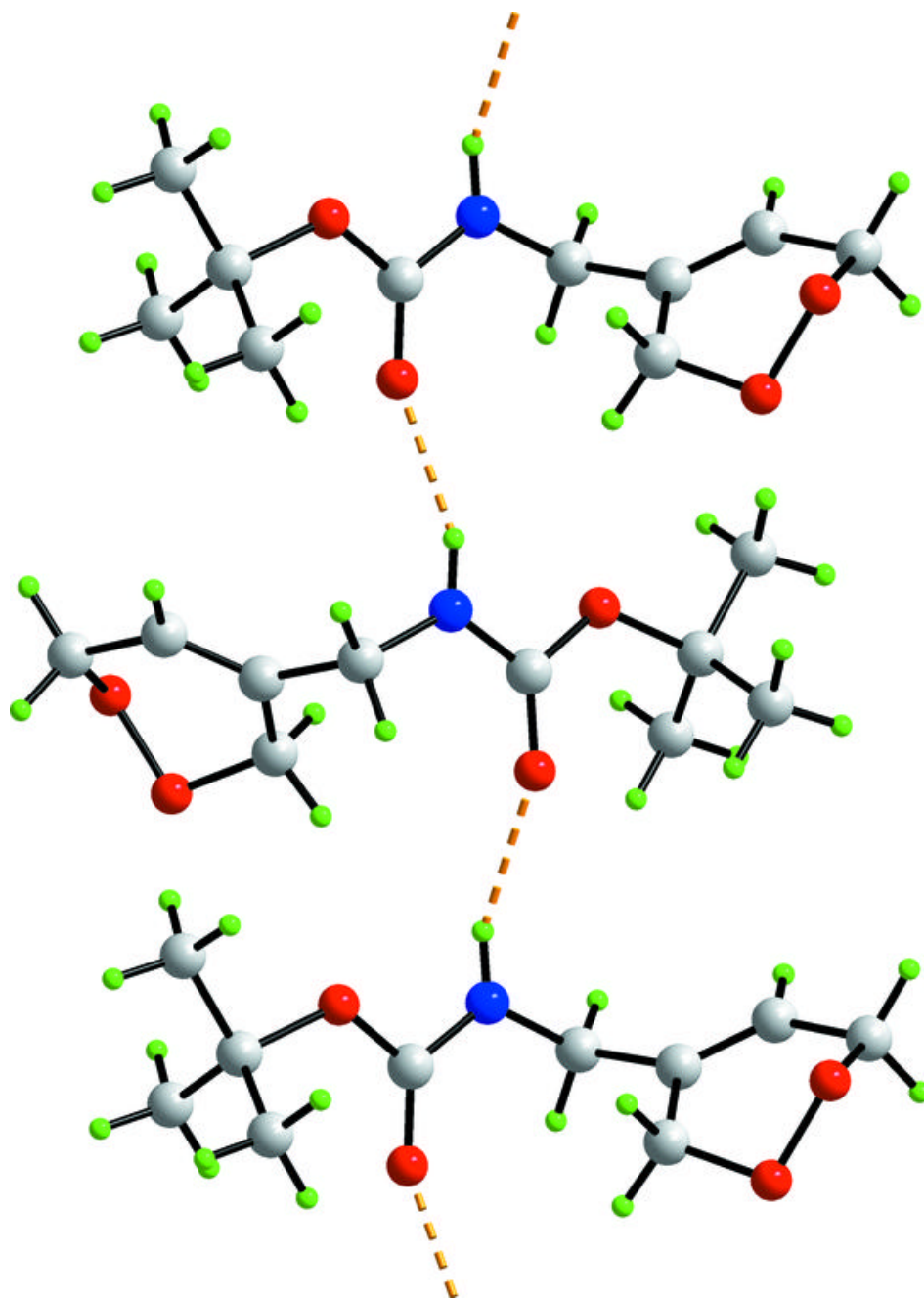


Fig. 3

