Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

Di-*tert*-butyl 2,2′-[(2-hydroxyethyl)-azanediyl]diacetate

Yang Yang, Lin Zhu and Huabei Zhang*

Key Laboratory of Radiopharmaceuticals, Ministry of Education, Department of Chemistry, Beijing Normal University, Xin Jie Kou Wai Street 19, 100875 Beijing, People's Republic of China
Correspondence e-mail: hbzhang@bnu.edu.cn

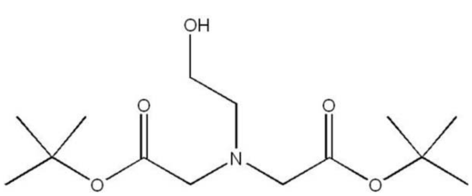
Received 21 October 2009; accepted 17 November 2009

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.002 Å; R factor = 0.046; wR factor = 0.128; data-to-parameter ratio = 20.8.

In the title compound, $C_{14}H_{27}NO_5$, the hydroxy group and one of the acetate carbonyl O atoms are linked by an intramolecular O—H⋯O hydrogen bond, forming an eight-membered ring. This interaction gives rise to an asymmetric molecular conformation.

Related literature

For details of the synthesis, see: Williams & Rapoport (1993); Amedio *et al.* (2000). For possible applications of the title compound, see: Yang *et al.* (2007).



Experimental

Crystal data

$C_{14}H_{27}NO_5$
M_r = 289.37
Orthorhombic, *Pbca*
a = 11.9767 (4) Å
b = 9.7187 (3) Å
c = 29.3476 (7) Å
V = 3416.00 (18) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 296 K
0.36 × 0.21 × 0.08 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)
T_{min} = 0.970, T_{max} = 0.993
12339 measured reflections
3958 independent reflections
2503 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.128
S = 1.02
3958 reflections
190 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O5—H5⋯O2	0.848 (10)	2.128 (17)	2.8658 (18)	145 (2)

Data collection: *SMART* (Bruker, 1998); 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*.

This work was supported by the Natural Science Foundation of China (No. 20671013) and the National Basic Research Program of China (No. 2006CB500705).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2237).

References

Amedio, J. C., Van Wagenen, G., Zavlin, G., Gyorkos, A. & Peterson, S. A. (2000). *Synth. Commun.* **30**, 3755-3763.
Bruker (1998). *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2005). *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2007). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Williams, M. A. & Rapoport, H. (1993). *J. Org. Chem.* **58**, 1151–1158.
Yang, Y., Zhang, J. X., Wang, J. J. & Zhu, L. (2007). *J. Radioanal. Nucl. Chem.* **273**, 31–36.

supplementary materials

Acta Cryst. (2009). E**65**, o3167 [**doi:10.1107/S1600536809049022**]

Di-*tert*-butyl 2,2'-[(2-hydroxyethyl)azanediyl]diacetate

Y. Yang, L. Zhu and H. Zhang

Comment

The aminodiacetate derivatives can find potential application when labeled by the novel $^{99m}Tc(CO)_2(NO)^{2+}$ core to explore the ^{99m}Tc labelled radiopharmaceuticals (Yang *et al.*, 2007). Thus, the development of aminodiacetate derivatives may lead to obtain some new imaging agents labelled by ^{99m}Tc core. Here we report the crystal structure of the title compound which can be used as a precursor in the synthesis of aminodiacetate derivatives.

The molecule of the title compound is shown in Fig. 1. The molecular conformation is to a large extentd determined by the intramolecular hydrogen bond O(5)—H(5)···O(2) (Table 1) which is a part of the eight-membered ring –O(5)—C(14)—C(13)—N(1)—C(6)—C(5)—O(2)······H(5)-. In the above ring, the torsion angles N(1)—C(13)—C(14)—O(5) and N(1)—C(6)—C(5)—O(2) are -57.2 (2)° and -3.5 (2)°.

Experimental

Tert-butyl 2-bromoacetate (22 g, 114 mmol) and $KHCO_3$ (13 g, 130 mmol) were dissolved in DMF (100 ml) at 0°C. Then 2-aminoethanol (3.2 ml, 50 mmol) was added to the solution in drops within 1 h. After adding 2-aminoethanol, the solution was heated at 55 °C for 20 h. Subsequently, the mixture was washed by the saturated $NaHCO_3$ solution and the crude product was extracted by ethyl acetate. After that, the organic phase was washed by saturated NaCl solution and the new organic phase was then dried by Na_2SO_4 for 48 h. After filtering the solution, the crude product was obtained. The crude product was recrystallized from ethyl acetate giving colorless block crystals of the title compound suitable for the single-crystal X-ray diffraction. IRnfrared Spectrum: 3438.3 cm^{-1}; 2978.5 cm^{-1}; 2933.7 cm^{-1}; 1456.8 cm^{-1}; 1393.6 cm^{-1}; 1732.0 cm^{-1}; 1368.6 cm^{-1}; 1223.5 cm^{-1}; 1070.9 cm^{-1}; 1150.5 cm$^{-1.}$ ^{1}H-NMR ($CDCl_3$, 400 MHz): 3.90 (s, 1H), 3.53 (t, J = 5.1 Hz, 2H), 3.45 (s, 4H), 2.89 (t, J = 5.1 Hz, 2H), 1.47 (s, 18H). ^{13}C-NMR ($CDCl_3$, 400 MHz): δ 28.13, 56.64, 57.07, 59.34, 81.49, 171.46. MS:m/*z* 290.3 [*M* + H].

Refinement

The H atoms bound to C atoms were introduced in idealized positions (C-H = 0.96-0.97 Å) and allowed to ride on their respective parent atoms with U_{iso}(H) =1.2 U_{eq}(C). The H atom from the hydroxy group was located in a difference Fourier synthesis and in the refinement the O-H distance was restrained to 0.86 (1) Å [U_{iso}(H) =1.5 U_{eq}(O)].

Figures

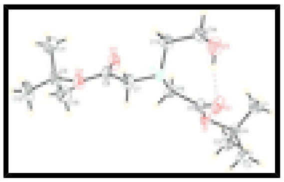


Fig. 1. The molecule the title compound, showing the atomic numbering; the displacement ellipsoids were drawn at the 30% probability level.

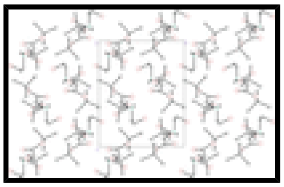


Fig. 2. The packing diagram of title compound (for clarity, all H atoms are not shown).

Di-*tert*-butyl 2,2'-[(2-hydroxyethyl)azanediyl]diacetate

Crystal data

$C_{14}H_{27}NO_5$	F_{000} = 1264
M_r = 289.37	D_x = 1.125 Mg m^{-3}
Orthorhombic, *Pbca*	Mo *K*α radiation, λ = 0.71073 Å
Hall symbol: -P 2ac 2ab	Cell parameters from 3711 reflections
a = 11.9767 (4) Å	θ = 2.2–26.1°
b = 9.7187 (3) Å	μ = 0.08 mm^{-1}
c = 29.3476 (7) Å	*T* = 296 K
V = 3416.00 (18) Å^3	Block, colorless
Z = 8	0.36 × 0.21 × 0.08 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer	3958 independent reflections
Radiation source: fine-focus sealed tube	2503 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.022
T = 296 K	θ_{max} = 27.6°
phi and ω scans	θ_{min} = 2.2°
Absorption correction: multi-scan (SADABS; Bruker, 2007)	$h = -15 \rightarrow 10$
T_{min} = 0.970, T_{max} = 0.993	$k = -12 \rightarrow 7$
12339 measured reflections	$l = -38 \rightarrow 25$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.046	H atoms treated by a mixture of independent and constrained refinement

$wR(F^2) = 0.128$	$w = 1/[\sigma^2(F_o^2) + (0.0551P)^2 + 0.5511P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} = 0.001$
3958 reflections	$\Delta\rho_{max} = 0.13$ e Å^{-3}
190 parameters	$\Delta\rho_{min} = -0.17$ e Å^{-3}
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.52162 (16)	0.1474 (2)	0.74442 (6)	0.0719 (5)
H1A	0.5798	0.0876	0.7338	0.108*
H1B	0.4957	0.1161	0.7736	0.108*
H1C	0.5502	0.2393	0.7473	0.108*
N1	0.63856 (10)	0.38227 (12)	0.59060 (4)	0.0459 (3)
O1	0.47237 (8)	0.16745 (10)	0.66460 (3)	0.0480 (3)
C2	0.37573 (18)	0.00455 (18)	0.70716 (6)	0.0777 (6)
H2A	0.3187	0.0050	0.6842	0.117*
H2B	0.3436	−0.0212	0.7359	0.117*
H2C	0.4327	−0.0603	0.6990	0.117*
O2	0.52896 (11)	0.38533 (11)	0.67575 (4)	0.0689 (4)
C3	0.33760 (16)	0.2516 (2)	0.72143 (7)	0.0753 (5)
H3A	0.3711	0.3411	0.7233	0.113*
H3B	0.3027	0.2295	0.7500	0.113*
H3C	0.2825	0.2511	0.6977	0.113*
O3	0.69461 (11)	0.19726 (12)	0.51907 (4)	0.0714 (4)
C4	0.42619 (13)	0.14629 (15)	0.71095 (5)	0.0473 (4)
O4	0.61496 (10)	0.33760 (11)	0.46780 (3)	0.0586 (3)
C5	0.52554 (12)	0.28259 (14)	0.65316 (5)	0.0459 (3)
O5	0.73364 (13)	0.53528 (15)	0.66616 (5)	0.0839 (4)
H5	0.6636 (9)	0.524 (3)	0.6645 (9)	0.126*
C6	0.58168 (13)	0.26158 (15)	0.60781 (5)	0.0515 (4)
H6A	0.6354	0.1873	0.6107	0.062*
H6B	0.5259	0.2333	0.5858	0.062*

C7	0.61304 (14)	0.41188 (16)	0.54343 (5)	0.0545 (4)
H7A	0.6497	0.4972	0.5351	0.065*
H7B	0.5332	0.4266	0.5407	0.065*
C8	0.64704 (13)	0.30191 (16)	0.50949 (5)	0.0508 (4)
C9	0.63142 (15)	0.24561 (18)	0.42820 (5)	0.0618 (4)
C10	0.56917 (19)	0.1123 (2)	0.43595 (8)	0.0933 (7)
H10A	0.6062	0.0599	0.4592	0.140*
H10B	0.5677	0.0601	0.4082	0.140*
H10C	0.4941	0.1320	0.4454	0.140*
C11	0.75478 (18)	0.2232 (3)	0.42041 (7)	0.0882 (6)
H11A	0.7921	0.3105	0.4192	0.132*
H11B	0.7656	0.1754	0.3921	0.132*
H11C	0.7850	0.1694	0.4449	0.132*
C12	0.5793 (2)	0.3278 (2)	0.38978 (6)	0.0994 (8)
H12A	0.5028	0.3477	0.3971	0.149*
H12B	0.5824	0.2753	0.3621	0.149*
H12C	0.6195	0.4123	0.3858	0.149*
C13	0.75774 (14)	0.38686 (19)	0.60054 (5)	0.0614 (4)
H13A	0.7921	0.4587	0.5824	0.074*
H13B	0.7913	0.2999	0.5919	0.074*
C14	0.78138 (16)	0.4137 (2)	0.65010 (6)	0.0750 (5)
H14A	0.7532	0.3373	0.6680	0.090*
H14B	0.8616	0.4180	0.6545	0.090*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0772 (12)	0.0836 (12)	0.0549 (10)	−0.0102 (10)	−0.0087 (9)	0.0146 (9)
N1	0.0527 (7)	0.0491 (7)	0.0360 (6)	−0.0043 (6)	0.0032 (5)	0.0030 (5)
O1	0.0588 (6)	0.0447 (5)	0.0406 (6)	−0.0079 (5)	0.0082 (4)	−0.0007 (4)
C2	0.1038 (14)	0.0652 (11)	0.0641 (11)	−0.0327 (11)	0.0156 (10)	0.0029 (9)
O2	0.1028 (10)	0.0468 (6)	0.0571 (7)	−0.0129 (6)	0.0304 (6)	−0.0092 (5)
C3	0.0693 (11)	0.0845 (13)	0.0722 (12)	0.0094 (10)	0.0238 (10)	0.0024 (10)
O3	0.0984 (9)	0.0681 (7)	0.0478 (7)	0.0330 (7)	−0.0009 (6)	−0.0002 (5)
C4	0.0546 (8)	0.0479 (8)	0.0393 (8)	−0.0062 (7)	0.0076 (7)	0.0034 (6)
O4	0.0760 (7)	0.0625 (7)	0.0374 (6)	0.0156 (6)	−0.0023 (5)	−0.0022 (5)
C5	0.0531 (8)	0.0434 (8)	0.0411 (8)	−0.0006 (7)	0.0041 (7)	0.0015 (6)
O5	0.1030 (10)	0.0812 (9)	0.0674 (8)	−0.0272 (9)	0.0050 (8)	−0.0216 (7)
C6	0.0611 (9)	0.0503 (8)	0.0430 (8)	−0.0069 (7)	0.0096 (7)	−0.0046 (7)
C7	0.0708 (10)	0.0530 (8)	0.0396 (8)	0.0118 (8)	0.0043 (7)	0.0023 (7)
C8	0.0577 (9)	0.0556 (9)	0.0391 (8)	0.0082 (8)	0.0040 (7)	0.0024 (7)
C9	0.0757 (11)	0.0699 (11)	0.0397 (9)	0.0100 (9)	−0.0004 (8)	−0.0105 (8)
C10	0.1030 (16)	0.0898 (15)	0.0872 (16)	−0.0163 (13)	−0.0079 (13)	−0.0199 (12)
C11	0.0809 (13)	0.1181 (17)	0.0658 (12)	0.0111 (13)	0.0199 (11)	−0.0130 (12)
C12	0.146 (2)	0.1065 (17)	0.0456 (11)	0.0351 (15)	−0.0202 (12)	−0.0077 (11)
C13	0.0567 (10)	0.0692 (11)	0.0584 (10)	−0.0081 (8)	0.0033 (8)	−0.0044 (8)
C14	0.0693 (12)	0.0905 (14)	0.0653 (12)	−0.0131 (10)	−0.0139 (9)	−0.0023 (11)

Geometric parameters (Å, °)

C1—C4	1.507 (2)	O5—H5	0.848 (10)
C1—H1A	0.9600	C6—H6A	0.9700
C1—H1B	0.9600	C6—H6B	0.9700
C1—H1C	0.9600	C7—C8	1.517 (2)
N1—C7	1.4465 (18)	C7—H7A	0.9700
N1—C6	1.4475 (18)	C7—H7B	0.9700
N1—C13	1.458 (2)	C9—C11	1.511 (3)
O1—C5	1.3306 (17)	C9—C10	1.512 (3)
O1—C4	1.4830 (16)	C9—C12	1.516 (2)
C2—C4	1.508 (2)	C10—H10A	0.9600
C2—H2A	0.9600	C10—H10B	0.9600
C2—H2B	0.9600	C10—H10C	0.9600
C2—H2C	0.9600	C11—H11A	0.9600
O2—C5	1.1992 (17)	C11—H11B	0.9600
C3—C4	1.506 (2)	C11—H11C	0.9600
C3—H3A	0.9600	C12—H12A	0.9600
C3—H3B	0.9600	C12—H12B	0.9600
C3—H3C	0.9600	C12—H12C	0.9600
O3—C8	1.1992 (17)	C13—C14	1.504 (2)
O4—C8	1.3285 (17)	C13—H13A	0.9700
O4—C9	1.4794 (18)	C13—H13B	0.9700
C5—C6	1.505 (2)	C14—H14A	0.9700
O5—C14	1.394 (2)	C14—H14B	0.9700
C4—C1—H1A	109.5	N1—C7—H7B	108.4
C4—C1—H1B	109.5	C8—C7—H7B	108.4
H1A—C1—H1B	109.5	H7A—C7—H7B	107.4
C4—C1—H1C	109.5	O3—C8—O4	125.07 (14)
H1A—C1—H1C	109.5	O3—C8—C7	124.84 (14)
H1B—C1—H1C	109.5	O4—C8—C7	110.08 (13)
C7—N1—C6	113.32 (12)	O4—C9—C11	109.66 (14)
C7—N1—C13	113.11 (12)	O4—C9—C10	109.51 (14)
C6—N1—C13	114.57 (13)	C11—C9—C10	112.41 (17)
C5—O1—C4	121.77 (11)	O4—C9—C12	102.18 (14)
C4—C2—H2A	109.5	C11—C9—C12	111.49 (17)
C4—C2—H2B	109.5	C10—C9—C12	111.10 (17)
H2A—C2—H2B	109.5	C9—C10—H10A	109.5
C4—C2—H2C	109.5	C9—C10—H10B	109.5
H2A—C2—H2C	109.5	H10A—C10—H10B	109.5
H2B—C2—H2C	109.5	C9—C10—H10C	109.5
C4—C3—H3A	109.5	H10A—C10—H10C	109.5
C4—C3—H3B	109.5	H10B—C10—H10C	109.5
H3A—C3—H3B	109.5	C9—C11—H11A	109.5
C4—C3—H3C	109.5	C9—C11—H11B	109.5
H3A—C3—H3C	109.5	H11A—C11—H11B	109.5
H3B—C3—H3C	109.5	C9—C11—H11C	109.5
O1—C4—C3	110.86 (12)	H11A—C11—H11C	109.5

O1—C4—C1	108.31 (12)	H11B—C11—H11C	109.5
C3—C4—C1	113.35 (15)	C9—C12—H12A	109.5
O1—C4—C2	102.02 (12)	C9—C12—H12B	109.5
C3—C4—C2	110.68 (15)	H12A—C12—H12B	109.5
C1—C4—C2	111.02 (14)	C9—C12—H12C	109.5
C8—O4—C9	121.82 (12)	H12A—C12—H12C	109.5
O2—C5—O1	125.25 (13)	H12B—C12—H12C	109.5
O2—C5—C6	125.91 (13)	N1—C13—C14	112.53 (14)
O1—C5—C6	108.83 (12)	N1—C13—H13A	109.1
C14—O5—H5	106.1 (19)	C14—C13—H13A	109.1
N1—C6—C5	114.14 (12)	N1—C13—H13B	109.1
N1—C6—H6A	108.7	C14—C13—H13B	109.1
C5—C6—H6A	108.7	H13A—C13—H13B	107.8
N1—C6—H6B	108.7	O5—C14—C13	113.37 (16)
C5—C6—H6B	108.7	O5—C14—H14A	108.9
H6A—C6—H6B	107.6	C13—C14—H14A	108.9
N1—C7—C8	115.57 (13)	O5—C14—H14B	108.9
N1—C7—H7A	108.4	C13—C14—H14B	108.9
C8—C7—H7A	108.4	H14A—C14—H14B	107.7

C5—O1—C4—C3	62.24 (18)	C9—O4—C8—O3	2.7 (2)
C5—O1—C4—C1	−62.70 (17)	C9—O4—C8—C7	−176.42 (14)
C5—O1—C4—C2	−179.88 (14)	N1—C7—C8—O3	−1.9 (2)
C4—O1—C5—O2	−10.4 (2)	N1—C7—C8—O4	177.19 (13)
C4—O1—C5—C6	168.74 (12)	C8—O4—C9—C11	−63.4 (2)
C7—N1—C6—C5	−132.21 (13)	C8—O4—C9—C10	60.4 (2)
C13—N1—C6—C5	95.96 (16)	C8—O4—C9—C12	178.25 (16)
O2—C5—C6—N1	−3.5 (2)	C7—N1—C13—C14	156.64 (14)
O1—C5—C6—N1	177.39 (12)	C6—N1—C13—C14	−71.43 (18)
C6—N1—C7—C8	−62.21 (18)	N1—C13—C14—O5	−57.2 (2)
C13—N1—C7—C8	70.33 (18)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5···O2	0.848 (10)	2.128 (17)	2.8658 (18)	145 (2)

Fig. 1

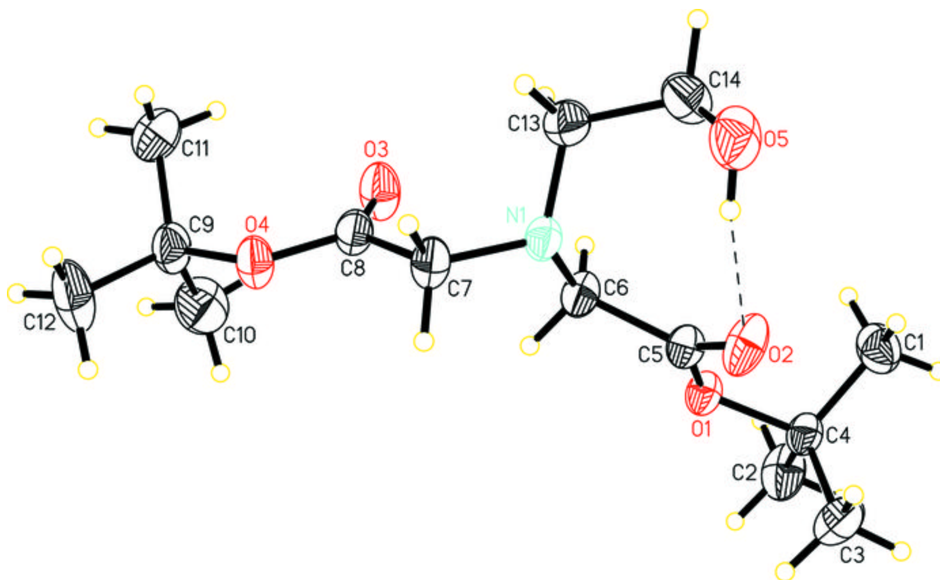


Fig. 2