Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Di-*tert*-butyl 2-benzoylhydrazine-1,1-dicarboxylate

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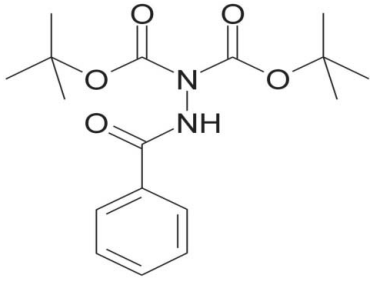
Received 22 November 2007; accepted 27 November 2007

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.038; wR factor = 0.109; data-to-parameter ratio = 8.6.

The crystal structure of the title compound, $C_{17}H_{24}N_2O_5$, was determined in the course of our studies on the preparation of two families of pseudopeptides, *viz*. hydrazino- and *N*-amino-peptides. The most significant interaction in the crystal structure is a bifurcated intermolecular N—H···O hydrogen bond.

Related literature

For the synthesis, see: Brosse *et al.* (2003). For geometry, see: Allen (2002); Kauffmann *et al.* (2004); Fong *et al.* (1996).



Experimental

Crystal data

$C_{17}H_{24}N_2O_5$
M_r = 336.38
Orthorhombic, $P2_12_12_1$
a = 9.9794 (2) Å
b = 11.5763 (3) Å
c = 16.0720 (4) Å
V = 1856.71 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 (2) K
0.3 × 0.05 × 0.05 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Absorption correction: none
9649 measured reflections
1944 independent reflections
1559 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.109
S = 1.03
1944 reflections
226 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.16 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—H2···O2^{i}	0.82 (3)	2.53 (3)	3.233 (3)	145 (3)
N2—H2···O4^{i}	0.82 (3)	2.32 (3)	3.062 (3)	150 (3)

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

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *WebLab ViewerPro 3.5* (MSI, 1999); software used to prepare material for publication: *WinGX* publication routines (Farrugia, 1999).

The authors thank the National Research Agency (ANR) for financial support (No. NT05_4_42848) and the Service Commun de Diffraction X (Nancy Université) for providing access to experimental crystallographic facilities.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HJ2004).

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supplementary materials

Acta Cryst. (2008). E**64**, o205 [**doi:10.1107/S160053680706391X**]

Di-*tert*-butyl 2-benzoylhydrazine-1,1-dicarboxylate

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Comment

As part of our continuing studies on the synthesis and structure of hydrazino- and *N*-amino-peptides, we have described the crystal structure of *N*-(*tert*-Butyloxycarbonylamino)phthalimide (Kauffmann *et al.*, 2004). Here we report the crystal structure of the title compound, *N*-benzoyl-N^{β}, N^{α}- bis (*tert*-butoxycarbonyl) hydrazine (Fig. 1).

Although the title compound is not chiral, it crystallizes in the non-centrosymmetric space group $P2_12_12_1$. The angle between the amide plane and the mean plane of the imidodicarbonate group is 78.43 (18)°, showing that these two groups are nearly perpendicular. The angle between the best-fit phenyl plane and the amide plane of 27.34 (7)° is similar to that reported for the benzamide group (Fong *et al.*, 1996).

In the crystal structure of the title compound, molecules are linked into infinite chains parallel to a *via* bifurcated N—H···O hydrogen bonds involving both carbonate of the aminoimidodicarbonate group (Fig. 2). All other intermolecular interactions are due to van der Waals forces.

Experimental

The title compound was prepared from *N*-(*tert*-Butyloxycarbonylamino)phthalimide (Brosse *et al.*, 2003), and was crystallized by slow evaporation of an ethanol solution.

Refinement

All H atoms were located in difference maps. The C-bonded H atoms were placed at calculated positions and refined using a riding model, with C—H distances of 0.93–0.96 Å. The N-bonded H atom was refined with free positional parameters. The H-atom U_{iso} parameters were fixed at 1.3Ueq(C) for aromatic C—H groups, at 1.3Ueq(N) for the N—H group and at 1.5Ueq(C) for methyl C—H.

Figures

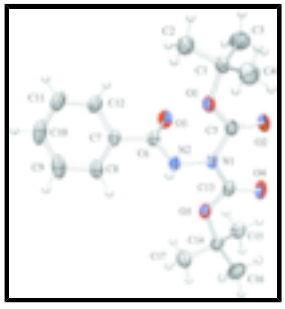

Fig. 1. The molecular structure of title compound showing the atom-numbering scheme. All non-H atoms are represented by 25% probability displacement ellipsoids.

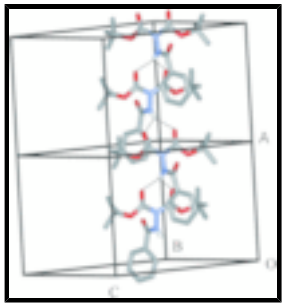

Fig. 2. Part of the crystal structure of the title compound showing the chains along [100]. The intermolecular hydrogen bonds are shown as dashed lines.

Di-*tert*-butyl 2-benzoylhydrazine-1,1-dicarboxylate

Crystal data

$C_{17}H_{24}N_2O_5$	F_{000} = 720
M_r = 336.38	D_x = 1.203 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.7107 Å
Hall symbol: P 2ac 2ab	Cell parameters from 9649 reflections
a = 9.9794 (2) Å	θ = 2.5–25.5º
b = 11.5763 (3) Å	μ = 0.09 mm^{-1}
c = 16.0720 (4) Å	T = 293 (2) K
V = 1856.71 (8) Å^3	Prism, colorless
Z = 4	0.3 × 0.05 × 0.05 mm

Data collection

Nonius KappaCCD area-detector diffractometer	1559 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.032
Monochromator: graphite	θ_{max} = 25.5º
ω and φ scans	θ_{min} = 2.5º
Absorption correction: none	h = −11→11
9649 measured reflections	k = −14→14
1944 independent reflections	l = −19→19

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0717P)^2 + 0.077P]$ where $P = (F_o^2 + 2F_c^2)/3$
Least-squares matrix: full	$(\Delta/\sigma)_{max}$ = 0.044
$R[F^2 > 2\sigma(F^2)]$ = 0.038	$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$wR(F^2)$ = 0.109	$\Delta\rho_{min}$ = −0.15 e Å^{-3}
S = 1.03	Extinction correction: none
1944 reflections	
226 parameters	
H atoms treated by a mixture of independent and constrained refinement	

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.5100 (2)	−0.0209 (2)	−0.17416 (18)	0.0659 (7)
C2	0.6320 (3)	0.0439 (4)	−0.2024 (2)	0.0978 (11)
H2A	0.6566	0.0997	−0.1609	0.147*
H2B	0.6128	0.0829	−0.2538	0.147*
H2C	0.7046	−0.0092	−0.2106	0.147*
C3	0.3937 (3)	0.0597 (3)	−0.1641 (3)	0.1131 (13)
H3A	0.3137	0.0158	−0.154	0.17*
H3B	0.3829	0.1045	−0.2139	0.17*
H3C	0.4099	0.1104	−0.1179	0.17*
C4	0.4826 (5)	−0.1216 (4)	−0.2302 (2)	0.1084 (12)
H4A	0.5609	−0.1695	−0.2334	0.163*
H4B	0.4602	−0.094	−0.2848	0.163*
H4C	0.4092	−0.1657	−0.2084	0.163*
O1	0.55435 (15)	−0.06767 (16)	−0.09296 (11)	0.0615 (5)
C5	0.4705 (2)	−0.1231 (2)	−0.04390 (17)	0.0556 (6)
O2	0.35449 (18)	−0.14394 (19)	−0.05764 (15)	0.0834 (6)
N1	0.53802 (19)	−0.15842 (17)	0.02859 (13)	0.0533 (5)
N2	0.67171 (18)	−0.12688 (16)	0.03912 (13)	0.0493 (5)
H2	0.723 (3)	−0.177 (2)	0.0211 (18)	0.064*
C6	0.7009 (2)	−0.0142 (2)	0.05343 (15)	0.0501 (6)
O3	0.61438 (18)	0.05698 (16)	0.06796 (13)	0.0722 (5)
C7	0.8465 (2)	0.0151 (2)	0.04796 (15)	0.0530 (6)
C8	0.9465 (2)	−0.0644 (2)	0.06302 (17)	0.0629 (7)
H8	0.9244	−0.139	0.0795	0.082*
C9	1.0804 (3)	−0.0334 (3)	0.0536 (2)	0.0855 (10)
H9	1.1477	−0.0872	0.0637	0.111*
C10	1.1127 (3)	0.0761 (4)	0.0296 (2)	0.0952 (11)
H10	1.2022	0.0965	0.0232	0.124*
C11	1.0149 (4)	0.1561 (3)	0.0149 (2)	0.0937 (11)
H11	1.0378	0.2304	−0.0019	0.122*
C12	0.8811 (3)	0.1263 (3)	0.02503 (19)	0.0735 (8)
H12	0.8146	0.1812	0.0164	0.096*
C13	0.4706 (2)	−0.2161 (2)	0.09331 (16)	0.0542 (6)
O4	0.35904 (18)	−0.25409 (17)	0.08502 (12)	0.0743 (6)
O5	0.54681 (15)	−0.22196 (15)	0.15998 (11)	0.0584 (4)
C14	0.5017 (3)	−0.2927 (2)	0.23280 (17)	0.0595 (6)
C15	0.3708 (3)	−0.2462 (2)	0.26688 (19)	0.0715 (8)
H15A	0.3791	−0.1646	0.2764	0.107*

H15B	0.3502	−0.2843	0.3184	0.107*
H15C	0.3003	−0.2601	0.2275	0.107*
C16	0.4944 (3)	−0.4176 (3)	0.2060 (2)	0.0883 (10)
H16A	0.423	−0.427	0.1665	0.132*
H16B	0.4778	−0.4655	0.2536	0.132*
H16C	0.5777	−0.4396	0.1807	0.132*
C17	0.6120 (3)	−0.2720 (4)	0.2951 (2)	0.0920 (10)
H17A	0.6962	−0.2953	0.2717	0.138*
H17B	0.5947	−0.3162	0.3445	0.138*
H17C	0.6153	−0.1914	0.3089	0.138*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0554 (13)	0.0720 (17)	0.0704 (16)	−0.0016 (12)	−0.0099 (13)	0.0087 (14)
C2	0.077 (2)	0.125 (3)	0.091 (2)	−0.022 (2)	−0.0004 (18)	0.034 (2)
C3	0.080 (2)	0.096 (2)	0.163 (4)	0.026 (2)	0.012 (3)	0.041 (3)
C4	0.137 (3)	0.108 (3)	0.080 (2)	−0.020 (3)	−0.023 (2)	−0.010 (2)
O1	0.0458 (8)	0.0760 (11)	0.0626 (10)	−0.0073 (9)	−0.0035 (8)	0.0080 (9)
C5	0.0431 (13)	0.0549 (13)	0.0689 (16)	−0.0016 (11)	0.0006 (12)	0.0000 (12)
O2	0.0423 (10)	0.1008 (14)	0.1071 (16)	−0.0138 (9)	−0.0132 (10)	0.0264 (13)
N1	0.0362 (10)	0.0588 (11)	0.0648 (12)	−0.0080 (8)	0.0018 (9)	0.0017 (10)
N2	0.0359 (10)	0.0495 (11)	0.0626 (12)	−0.0023 (8)	0.0041 (9)	−0.0032 (9)
C6	0.0476 (13)	0.0537 (13)	0.0490 (13)	−0.0005 (10)	0.0015 (11)	−0.0047 (11)
O3	0.0571 (10)	0.0634 (10)	0.0960 (13)	0.0068 (9)	0.0060 (9)	−0.0205 (11)
C7	0.0470 (12)	0.0615 (14)	0.0503 (13)	−0.0099 (11)	0.0009 (11)	−0.0090 (11)
C8	0.0499 (13)	0.0681 (15)	0.0707 (16)	−0.0058 (13)	−0.0006 (12)	−0.0143 (14)
C9	0.0511 (15)	0.106 (3)	0.099 (2)	−0.0035 (16)	0.0008 (16)	−0.032 (2)
C10	0.0616 (18)	0.125 (3)	0.099 (2)	−0.039 (2)	0.0132 (18)	−0.022 (2)
C11	0.088 (2)	0.096 (2)	0.098 (3)	−0.042 (2)	0.0036 (19)	0.0067 (19)
C12	0.0696 (17)	0.0702 (17)	0.0806 (19)	−0.0189 (14)	−0.0040 (15)	0.0049 (16)
C13	0.0439 (13)	0.0551 (13)	0.0637 (14)	−0.0060 (11)	0.0075 (12)	−0.0056 (12)
O4	0.0537 (10)	0.0951 (14)	0.0740 (12)	−0.0262 (10)	0.0028 (9)	−0.0005 (10)
O5	0.0451 (8)	0.0669 (10)	0.0632 (10)	−0.0113 (8)	0.0039 (8)	0.0058 (9)
C14	0.0508 (13)	0.0568 (15)	0.0711 (17)	−0.0038 (11)	0.0058 (12)	0.0078 (12)
C15	0.0632 (16)	0.0717 (18)	0.0796 (18)	0.0039 (14)	0.0190 (14)	0.0056 (14)
C16	0.092 (2)	0.0553 (17)	0.118 (3)	0.0054 (16)	0.023 (2)	0.0053 (16)
C17	0.0674 (17)	0.123 (3)	0.086 (2)	−0.0129 (19)	−0.0080 (17)	0.027 (2)

Geometric parameters (Å, °)

C1—O1	1.481 (3)	C8—C9	1.393 (4)
C1—C3	1.498 (4)	C8—H8	0.93
C1—C4	1.498 (5)	C9—C10	1.363 (5)
C1—C2	1.500 (4)	C9—H9	0.93
C2—H2A	0.96	C10—C11	1.367 (5)
C2—H2B	0.96	C10—H10	0.93
C2—H2C	0.96	C11—C12	1.388 (4)
C3—H3A	0.96	C11—H11	0.93

C3—H3B	0.96	C12—H12	0.93
C3—H3C	0.96	C13—O4	1.204 (3)
C4—H4A	0.96	C13—O5	1.316 (3)
C4—H4B	0.96	O5—C14	1.498 (3)
C4—H4C	0.96	C14—C17	1.508 (4)
O1—C5	1.317 (3)	C14—C16	1.510 (4)
C5—O2	1.203 (3)	C14—C15	1.515 (4)
C5—N1	1.407 (3)	C15—H15A	0.96
N1—N2	1.393 (3)	C15—H15B	0.96
N1—C13	1.407 (3)	C15—H15C	0.96
N2—C6	1.356 (3)	C16—H16A	0.96
N2—H2	0.82 (3)	C16—H16B	0.96
C6—O3	1.216 (3)	C16—H16C	0.96
C6—C7	1.495 (3)	C17—H17A	0.96
C7—C8	1.378 (4)	C17—H17B	0.96
C7—C12	1.383 (4)	C17—H17C	0.96
O1—C1—C3	111.4 (3)	C9—C8—H8	119.9
O1—C1—C4	107.5 (2)	C10—C9—C8	119.8 (3)
C3—C1—C4	114.0 (3)	C10—C9—H9	120.1
O1—C1—C2	102.0 (2)	C8—C9—H9	120.1
C3—C1—C2	110.5 (3)	C9—C10—C11	120.7 (3)
C4—C1—C2	110.8 (3)	C9—C10—H10	119.6
C1—C2—H2A	109.5	C11—C10—H10	119.6
C1—C2—H2B	109.5	C10—C11—C12	119.8 (3)
H2A—C2—H2B	109.5	C10—C11—H11	120.1
C1—C2—H2C	109.5	C12—C11—H11	120.1
H2A—C2—H2C	109.5	C7—C12—C11	120.2 (3)
H2B—C2—H2C	109.5	C7—C12—H12	119.9
C1—C3—H3A	109.5	C11—C12—H12	119.9
C1—C3—H3B	109.5	O4—C13—O5	127.3 (2)
H3A—C3—H3B	109.5	O4—C13—N1	122.3 (2)
C1—C3—H3C	109.5	O5—C13—N1	110.47 (18)
H3A—C3—H3C	109.5	C13—O5—C14	119.39 (17)
H3B—C3—H3C	109.5	O5—C14—C17	102.3 (2)
C1—C4—H4A	109.5	O5—C14—C16	108.3 (2)
C1—C4—H4B	109.5	C17—C14—C16	112.2 (3)
H4A—C4—H4B	109.5	O5—C14—C15	110.3 (2)
C1—C4—H4C	109.5	C17—C14—C15	109.4 (2)
H4A—C4—H4C	109.5	C16—C14—C15	113.7 (2)
H4B—C4—H4C	109.5	C14—C15—H15A	109.5
C5—O1—C1	121.07 (18)	C14—C15—H15B	109.5
O2—C5—O1	126.8 (3)	H15A—C15—H15B	109.5
O2—C5—N1	123.7 (2)	C14—C15—H15C	109.5
O1—C5—N1	109.43 (19)	H15A—C15—H15C	109.5
N2—N1—C5	118.9 (2)	H15B—C15—H15C	109.5
N2—N1—C13	119.5 (2)	C14—C16—H16A	109.5
C5—N1—C13	121.37 (19)	C14—C16—H16B	109.5
C6—N2—N1	118.55 (19)	H16A—C16—H16B	109.5
C6—N2—H2	127.0 (19)	C14—C16—H16C	109.5

N1—N2—H2	111.5 (19)	H16A—C16—H16C	109.5
O3—C6—N2	122.2 (2)	H16B—C16—H16C	109.5
O3—C6—C7	123.2 (2)	C14—C17—H17A	109.5
N2—C6—C7	114.6 (2)	C14—C17—H17B	109.5
C8—C7—C12	119.1 (2)	H17A—C17—H17B	109.5
C8—C7—C6	122.8 (2)	C14—C17—H17C	109.5
C12—C7—C6	118.0 (2)	H17A—C17—H17C	109.5
C7—C8—C9	120.2 (3)	H17B—C17—H17C	109.5
C7—C8—H8	119.9		
C3—C1—O1—C5	56.4 (3)	C12—C7—C8—C9	−1.3 (4)
C4—C1—O1—C5	−69.2 (3)	C6—C7—C8—C9	177.3 (3)
C2—C1—O1—C5	174.2 (3)	C7—C8—C9—C10	0.2 (5)
C1—O1—C5—O2	1.2 (4)	C8—C9—C10—C11	0.2 (5)
C1—O1—C5—N1	179.81 (19)	C9—C10—C11—C12	0.5 (6)
O2—C5—N1—N2	−178.3 (2)	C8—C7—C12—C11	2.0 (4)
O1—C5—N1—N2	3.1 (3)	C6—C7—C12—C11	−176.7 (3)
O2—C5—N1—C13	−3.7 (4)	C10—C11—C12—C7	−1.6 (5)
O1—C5—N1—C13	177.6 (2)	N2—N1—C13—O4	−173.4 (2)
C5—N1—N2—C6	69.7 (3)	C5—N1—C13—O4	12.0 (4)
C13—N1—N2—C6	−105.0 (3)	N2—N1—C13—O5	5.8 (3)
N1—N2—C6—O3	9.7 (4)	C5—N1—C13—O5	−168.72 (19)
N1—N2—C6—C7	−169.0 (2)	O4—C13—O5—C14	6.9 (4)
O3—C6—C7—C8	154.6 (3)	N1—C13—O5—C14	−172.37 (19)
N2—C6—C7—C8	−26.7 (4)	C13—O5—C14—C17	−177.6 (2)
O3—C6—C7—C12	−26.7 (4)	C13—O5—C14—C16	63.8 (3)
N2—C6—C7—C12	151.9 (2)	C13—O5—C14—C15	−61.3 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O2^{i}	0.82 (3)	2.53 (3)	3.233 (3)	145 (3)
N2—H2···O4^{i}	0.82 (3)	2.32 (3)	3.062 (3)	150 (3)

Symmetry codes: (i) *x*+1/2, −*y*−1/2, −*z*.

Fig. 1

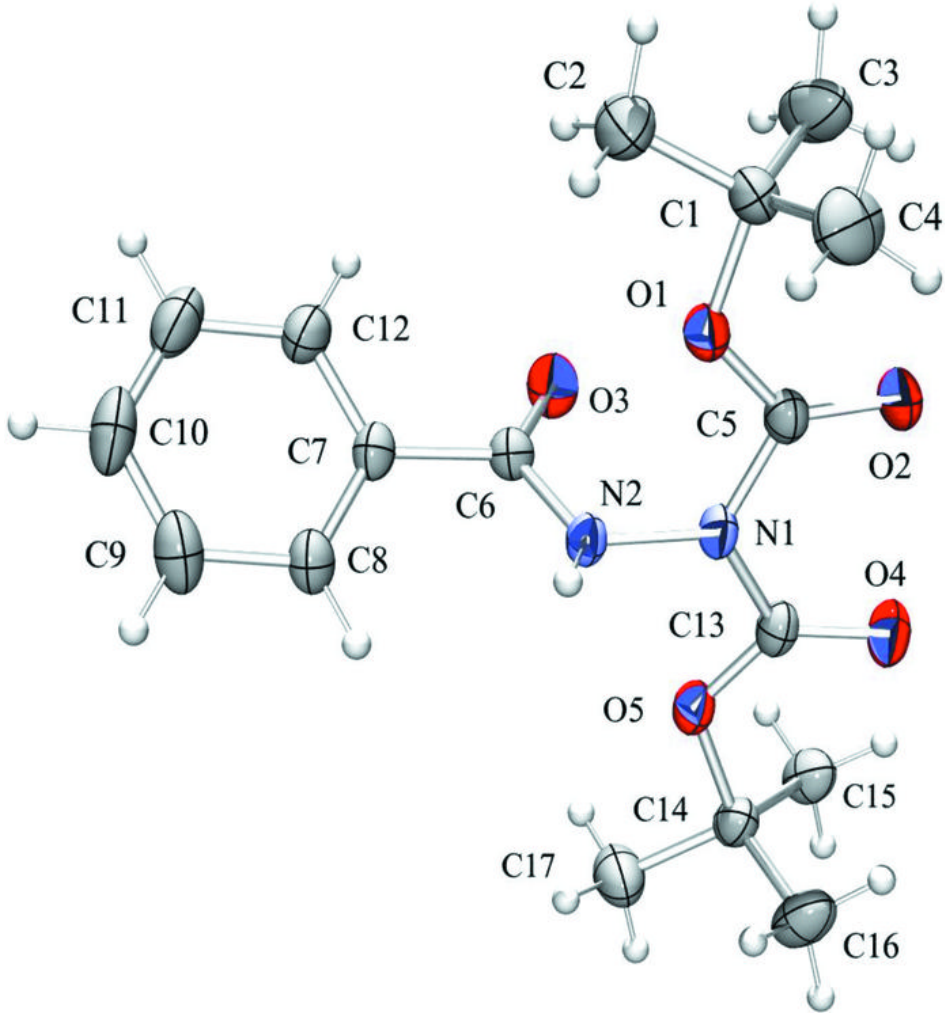
C2
C3
C1
C4
O1
C11
C12
O3
C5
O2
C10
C7
C6
N2
N1
C9
C8
O4
C13
O5
C14
C15
C17
C16

Fig. 2