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(*Z*)-2,5-Dimethyl-3,4-diphenylhex-3-ene-2,5-diol

Shu-Hong Li[a] and Cai-Hong Xu[b]*

[a]School of Chemical and Environmental Engineering, Beijing Technology and Business University, Beijing 100037, People's Republic of China, and [b]Laboratory of Advanced Polymer Materials, Institute of Chemistry, Chinese Academy of Sciences, Beijing 100080, People's Republic of China
Correspondence e-mail: caihong@iccas.ac.cn

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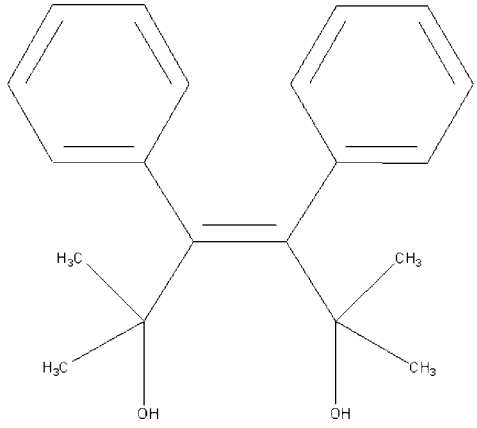
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.003 Å; R factor = 0.043; wR factor = 0.111; data-to-parameter ratio = 14.4.

The title molecule, $C_{20}H_{24}O_2$, shows a Z configuration with respect to the C=C double bond. The crystal structure is stabilized by intra- and intermolecular O—H···O hydrogen bonds.

Related literature

For related literature, see: Yamaguchi *et al.* (2003).



Experimental

Crystal data

$C_{20}H_{24}O_2$
M_r = 296.39
Monoclinic, $P2_1/c$
a = 12.364 (3) Å
b = 16.826 (4) Å
c = 8.770 (2) Å
β = 108.682 (5)°
V = 1728.4 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.07 mm^{-1}
T = 294 (2) K
0.30 × 0.26 × 0.20 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.979, T_{max} = 0.986
8635 measured reflections
3053 independent reflections
1914 reflections with $I > 2\sigma(I)$
R_{int} = 0.042

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.111
S = 1.01
3053 reflections
212 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···O2	0.863 (10)	1.756 (14)	2.562 (2)	155 (2)
O2—H2A···O1^{i}	0.856 (10)	1.944 (10)	2.7998 (19)	177 (2)

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BV2060).

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supplementary materials

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(*Z*)-2,5-Dimethyl-3,4-diphenylhex-3-ene-2,5-diol

S.-H. Li and C.-H. Xu

Comment

As part of our investigations of new intramolecular reductive cyclization (Yamaguchi *et al.* 2003), the title molecule (I), $C_{20}H_{24}O_2$, has been synthesized and structurally characterized (Fig.1. The molecule has a *Z* configuration with respect with the C=C bond; the phenyl groups protrude on the same side of the C=C bond. In the crystal structure (Fig. 2), the molecules are linked by intra- and inter-molecular O—H···O hydrogen bonds.

Experimental

To prove the intermediate species in the reductive reaction of diphenylacetylene under experimental condition, the title molecule was obtained according to the following procedure. A mixture of granular lithium (27.3 mg, 3.9 mmol) and naphthalene (504.2 mg, 3.9 mmol) in THF was stirred at room temperature for 4 h. To the resulting solution of lithium naphthalenide was added a solution of diphenylacetylene (300.0 mg, 1.7 mmol) in THF. After stirring for 5 min, to the reaction mixture was added dried acetone (excess) and stirred for another 30 min. Then the reaction was quenched with a saturated NH_4Cl aqueous solution. The mixture was extracted with diethyl ether, and the organic layer was washed with brine, dried over $MgSO_4$, filtered, and concentrated under reduced pressure. To the resulted mixture was added hexane, the white precipitate was collected and recrystallized from ethanol to give 260.0 mg of product in 52% yield as a colorless solid.

Refinement

H atoms bonded to O atoms were located in a difference map and refined with distance restraints of O—H = 0.86 (2) \%A, with Uĩso~(H)=1.7U~eq~(O). Other H atoms were positioned geometrically and refined using a riding model with C—H bonds 0.93–0.96\%A and with Uĩso~(H)=1.2U~eq~(C) for aromatic C or Uĩso~(H)=1.5U~eq~(C) for methyl groups.

Figures

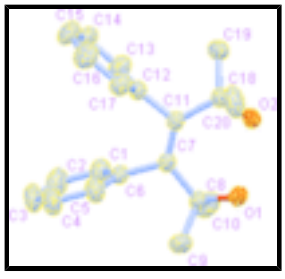

Fig. 1. The molecular structure of (I), with atom labels and 50% probability displacement ellipsoids for non-H atoms.

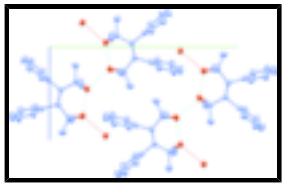

Fig. 2. The packing of (I), viewed down the *a* axis, showing one layer of molecules connected by O—H···O hydrogen bonds (dashed lines). H atoms have been omitted.

(*Z*)-2,5-Dimethyl-3,4-diphenylhex-3-ene-2,5-diol

Crystal data

$C_{20}H_{24}O_2$	F_{000} = 640
M_r = 296.39	D_x = 1.139 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 12.364 (3) Å	Cell parameters from 2045 reflections
b = 16.826 (4) Å	θ = 2.4–23.9º
c = 8.770 (2) Å	μ = 0.07 mm^{-1}
β = 108.682 (5)º	T = 294 (2) K
V = 1728.4 (7) Å^3	Platelet, colourless
Z = 4	0.30 × 0.26 × 0.20 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer	3053 independent reflections
Radiation source: fine-focus sealed tube	1914 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.042
T = 294(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 1.7º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −14→11
T_{min} = 0.979, T_{max} = 0.986	k = −19→20
8635 measured reflections	l = −5→10

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.043	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.111	$w = 1/[\sigma^2(F_o^2) + (0.0409P)^2 + 0.3594P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.01	$(\Delta/\sigma)_{max}$ = 0.001
3053 reflections	$\Delta\rho_{max}$ = 0.14 e Å^{-3}
212 parameters	$\Delta\rho_{min}$ = −0.12 e Å^{-3}

2 restraints

Primary atom site location: structure-invariant direct methods

Extinction correction: SHELXL97 (Sheldrick, 1997a), $Fc^{*}=kFc[1+0.001xFc^{2}\lambda^{3}/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.037 (2)

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 $F^2 > 2\mathrm{sigma}(F^2)$ 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}
O1	0.77809 (13)	0.17589 (8)	0.74092 (16)	0.0544 (4)
O2	0.77989 (13)	0.19942 (8)	0.45310 (15)	0.0517 (4)
C1	0.91048 (17)	−0.07738 (11)	0.6473 (2)	0.0487 (5)
H1	0.9597	−0.0482	0.6079	0.058*
C2	0.9351 (2)	−0.15607 (13)	0.6900 (3)	0.0659 (7)
H2	1.0005	−0.1795	0.6796	0.079*
C3	0.8625 (2)	−0.19964 (13)	0.7481 (3)	0.0716 (7)
H3	0.8794	−0.2524	0.7781	0.086*
C4	0.7652 (2)	−0.16549 (13)	0.7616 (3)	0.0642 (7)
H4	0.7157	−0.1952	0.7995	0.077*
C5	0.74132 (18)	−0.08725 (11)	0.7191 (2)	0.0485 (5)
H5	0.6752	−0.0645	0.7286	0.058*
C6	0.81348 (16)	−0.04124 (10)	0.6621 (2)	0.0356 (5)
C7	0.78709 (15)	0.04481 (10)	0.6162 (2)	0.0321 (4)
C8	0.83953 (16)	0.10249 (10)	0.7584 (2)	0.0379 (5)
C9	0.83320 (19)	0.06978 (12)	0.9172 (2)	0.0544 (6)
H9A	0.8624	0.1086	1.0005	0.082*
H9B	0.8780	0.0221	0.9442	0.082*
H9C	0.7552	0.0580	0.9069	0.082*
C10	0.96388 (18)	0.11861 (13)	0.7733 (3)	0.0605 (6)
H10A	0.9677	0.1421	0.6754	0.091*
H10B	1.0057	0.0695	0.7925	0.091*
H10C	0.9966	0.1543	0.8614	0.091*
C11	0.72791 (15)	0.06071 (10)	0.4617 (2)	0.0321 (4)
C12	0.68450 (16)	−0.00883 (10)	0.3491 (2)	0.0343 (4)
C13	0.74947 (18)	−0.04143 (12)	0.2630 (2)	0.0475 (5)
H13	0.8219	−0.0211	0.2766	0.057*
C14	0.7095 (2)	−0.10337 (13)	0.1572 (2)	0.0582 (6)
H14	0.7546	−0.1241	0.0998	0.070*

C15	0.6033 (2)	−0.13453 (13)	0.1366 (3)	0.0629 (7)
H15	0.5763	−0.1765	0.0656	0.076*
C16	0.5369 (2)	−0.10355 (13)	0.2211 (3)	0.0603 (6)
H16	0.4649	−0.1246	0.2075	0.072*
C17	0.57722 (17)	−0.04096 (11)	0.3267 (2)	0.0454 (5)
H17	0.5317	−0.0202	0.3834	0.054*
C18	0.69315 (17)	0.14181 (10)	0.3770 (2)	0.0394 (5)
C19	0.6856 (2)	0.13876 (12)	0.2005 (2)	0.0632 (7)
H19A	0.6655	0.1903	0.1531	0.095*
H19B	0.6284	0.1009	0.1450	0.095*
H19C	0.7581	0.1231	0.1920	0.095*
C20	0.5791 (2)	0.16815 (13)	0.3915 (3)	0.0691 (7)
H20A	0.5850	0.1716	0.5031	0.104*
H20B	0.5212	0.1302	0.3387	0.104*
H20C	0.5591	0.2193	0.3418	0.104*
H1A	0.781 (2)	0.1982 (14)	0.654 (2)	0.095 (10)*
H2A	0.778 (2)	0.2382 (11)	0.389 (2)	0.089 (9)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0885 (12)	0.0351 (8)	0.0381 (8)	0.0161 (7)	0.0179 (8)	−0.0040 (6)
O2	0.0783 (11)	0.0328 (8)	0.0376 (8)	−0.0157 (7)	0.0097 (7)	0.0031 (6)
C1	0.0482 (13)	0.0397 (12)	0.0561 (13)	0.0041 (10)	0.0140 (10)	−0.0054 (9)
C2	0.0624 (16)	0.0436 (14)	0.0833 (18)	0.0183 (12)	0.0119 (13)	−0.0084 (12)
C3	0.092 (2)	0.0308 (12)	0.0811 (18)	0.0125 (14)	0.0120 (15)	0.0068 (11)
C4	0.0833 (19)	0.0372 (13)	0.0721 (16)	−0.0039 (13)	0.0250 (13)	0.0076 (11)
C5	0.0523 (14)	0.0354 (12)	0.0585 (13)	0.0007 (10)	0.0187 (10)	0.0048 (10)
C6	0.0421 (12)	0.0284 (10)	0.0335 (10)	0.0031 (9)	0.0080 (8)	−0.0027 (8)
C7	0.0363 (11)	0.0285 (10)	0.0343 (10)	0.0006 (8)	0.0153 (8)	−0.0025 (8)
C8	0.0503 (13)	0.0295 (10)	0.0339 (10)	0.0023 (9)	0.0134 (9)	−0.0025 (8)
C9	0.0783 (16)	0.0498 (13)	0.0338 (11)	0.0005 (11)	0.0160 (10)	−0.0031 (9)
C10	0.0582 (15)	0.0595 (15)	0.0620 (15)	−0.0184 (12)	0.0168 (11)	−0.0193 (11)
C11	0.0353 (11)	0.0281 (10)	0.0353 (10)	−0.0003 (8)	0.0149 (8)	−0.0021 (8)
C12	0.0417 (12)	0.0290 (10)	0.0319 (10)	0.0003 (9)	0.0113 (8)	0.0004 (8)
C13	0.0461 (13)	0.0484 (12)	0.0486 (12)	−0.0020 (10)	0.0158 (10)	−0.0111 (10)
C14	0.0676 (16)	0.0549 (14)	0.0514 (13)	0.0043 (13)	0.0183 (11)	−0.0187 (11)
C15	0.0778 (18)	0.0470 (14)	0.0538 (14)	−0.0081 (13)	0.0068 (12)	−0.0203 (11)
C16	0.0568 (15)	0.0542 (14)	0.0644 (15)	−0.0190 (12)	0.0116 (12)	−0.0104 (12)
C17	0.0457 (13)	0.0425 (12)	0.0490 (12)	−0.0054 (10)	0.0166 (10)	−0.0055 (9)
C18	0.0509 (13)	0.0288 (10)	0.0355 (10)	−0.0050 (9)	0.0096 (9)	0.0014 (8)
C19	0.1003 (19)	0.0448 (13)	0.0359 (12)	−0.0130 (13)	0.0096 (12)	0.0031 (9)
C20	0.0644 (17)	0.0423 (13)	0.1009 (19)	0.0165 (12)	0.0270 (14)	0.0149 (12)

Geometric parameters (Å, °)

O1—C8	1.432 (2)	C10—H10B	0.9600
O1—H1A	0.863 (10)	C10—H10C	0.9600
O2—C18	1.441 (2)	C11—C12	1.514 (2)

O2—H2A	0.856 (10)	C11—C18	1.547 (2)
C1—C2	1.383 (3)	C12—C13	1.381 (3)
C1—C6	1.388 (3)	C12—C17	1.387 (3)
C1—H1	0.9300	C13—C14	1.377 (3)
C2—C3	1.377 (3)	C13—H13	0.9300
C2—H2	0.9300	C14—C15	1.371 (3)
C3—C4	1.372 (3)	C14—H14	0.9300
C3—H3	0.9300	C15—C16	1.373 (3)
C4—C5	1.375 (3)	C15—H15	0.9300
C4—H4	0.9300	C16—C17	1.386 (3)
C5—C6	1.389 (3)	C16—H16	0.9300
C5—H5	0.9300	C17—H17	0.9300
C6—C7	1.510 (2)	C18—C19	1.521 (3)
C7—C11	1.345 (2)	C18—C20	1.522 (3)
C7—C8	1.549 (2)	C19—H19A	0.9600
C8—C9	1.522 (3)	C19—H19B	0.9600
C8—C10	1.525 (3)	C19—H19C	0.9600
C9—H9A	0.9600	C20—H20A	0.9600
C9—H9B	0.9600	C20—H20B	0.9600
C9—H9C	0.9600	C20—H20C	0.9600
C10—H10A	0.9600		
C8—O1—H1A	107.3 (18)	H10B—C10—H10C	109.5
C18—O2—H2A	110.1 (17)	C7—C11—C12	117.92 (15)
C2—C1—C6	121.1 (2)	C7—C11—C18	129.59 (15)
C2—C1—H1	119.5	C12—C11—C18	112.48 (14)
C6—C1—H1	119.5	C13—C12—C17	117.73 (17)
C3—C2—C1	119.8 (2)	C13—C12—C11	121.14 (17)
C3—C2—H2	120.1	C17—C12—C11	121.12 (16)
C1—C2—H2	120.1	C14—C13—C12	121.5 (2)
C4—C3—C2	120.2 (2)	C14—C13—H13	119.2
C4—C3—H3	119.9	C12—C13—H13	119.2
C2—C3—H3	119.9	C15—C14—C13	120.0 (2)
C3—C4—C5	119.7 (2)	C15—C14—H14	120.0
C3—C4—H4	120.1	C13—C14—H14	120.0
C5—C4—H4	120.1	C14—C15—C16	119.8 (2)
C4—C5—C6	121.6 (2)	C14—C15—H15	120.1
C4—C5—H5	119.2	C16—C15—H15	120.1
C6—C5—H5	119.2	C15—C16—C17	120.0 (2)
C1—C6—C5	117.60 (18)	C15—C16—H16	120.0
C1—C6—C7	120.97 (18)	C17—C16—H16	120.0
C5—C6—C7	121.42 (17)	C16—C17—C12	120.9 (2)
C11—C7—C6	117.43 (15)	C16—C17—H17	119.5
C11—C7—C8	129.71 (15)	C12—C17—H17	119.5
C6—C7—C8	112.79 (14)	O2—C18—C19	106.75 (15)
O1—C8—C9	103.22 (14)	O2—C18—C20	109.71 (16)
O1—C8—C10	109.96 (16)	C19—C18—C20	109.76 (17)
C9—C8—C10	109.36 (16)	O2—C18—C11	108.64 (14)
O1—C8—C7	112.54 (14)	C19—C18—C11	112.09 (15)
C9—C8—C7	112.26 (15)	C20—C18—C11	109.81 (16)

C10—C8—C7	109.33 (15)	C18—C19—H19A	109.5
C8—C9—H9A	109.5	C18—C19—H19B	109.5
C8—C9—H9B	109.5	H19A—C19—H19B	109.5
H9A—C9—H9B	109.5	C18—C19—H19C	109.5
C8—C9—H9C	109.5	H19A—C19—H19C	109.5
H9A—C9—H9C	109.5	H19B—C19—H19C	109.5
H9B—C9—H9C	109.5	C18—C20—H20A	109.5
C8—C10—H10A	109.5	C18—C20—H20B	109.5
C8—C10—H10B	109.5	H20A—C20—H20B	109.5
H10A—C10—H10B	109.5	C18—C20—H20C	109.5
C8—C10—H10C	109.5	H20A—C20—H20C	109.5
H10A—C10—H10C	109.5	H20B—C20—H20C	109.5
C6—C1—C2—C3	0.1 (3)	C6—C7—C11—C18	−176.79 (17)
C1—C2—C3—C4	0.8 (4)	C8—C7—C11—C18	−0.2 (3)
C2—C3—C4—C5	−0.9 (4)	C7—C11—C12—C13	−90.5 (2)
C3—C4—C5—C6	0.0 (3)	C18—C11—C12—C13	90.8 (2)
C2—C1—C6—C5	−0.9 (3)	C7—C11—C12—C17	90.8 (2)
C2—C1—C6—C7	179.66 (18)	C18—C11—C12—C17	−88.0 (2)
C4—C5—C6—C1	0.9 (3)	C17—C12—C13—C14	0.4 (3)
C4—C5—C6—C7	−179.71 (18)	C11—C12—C13—C14	−178.34 (18)
C1—C6—C7—C11	87.2 (2)	C12—C13—C14—C15	−0.5 (3)
C5—C6—C7—C11	−92.1 (2)	C13—C14—C15—C16	0.3 (3)
C1—C6—C7—C8	−90.0 (2)	C14—C15—C16—C17	0.1 (3)
C5—C6—C7—C8	90.7 (2)	C15—C16—C17—C12	−0.2 (3)
C11—C7—C8—O1	28.7 (3)	C13—C12—C17—C16	−0.1 (3)
C6—C7—C8—O1	−154.52 (16)	C11—C12—C17—C16	178.68 (18)
C11—C7—C8—C9	144.7 (2)	C7—C11—C18—O2	31.8 (3)
C6—C7—C8—C9	−38.6 (2)	C12—C11—C18—O2	−149.64 (15)
C11—C7—C8—C10	−93.8 (2)	C7—C11—C18—C19	149.5 (2)
C6—C7—C8—C10	82.98 (19)	C12—C11—C18—C19	−31.9 (2)
C6—C7—C11—C12	4.7 (2)	C7—C11—C18—C20	−88.2 (2)
C8—C7—C11—C12	−178.68 (17)	C12—C11—C18—C20	90.37 (19)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1A···O2	0.863 (10)	1.756 (14)	2.562 (2)	155 (2)
O2—H2A···O1^{i}	0.856 (10)	1.944 (10)	2.7998 (19)	177 (2)

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

Fig. 1

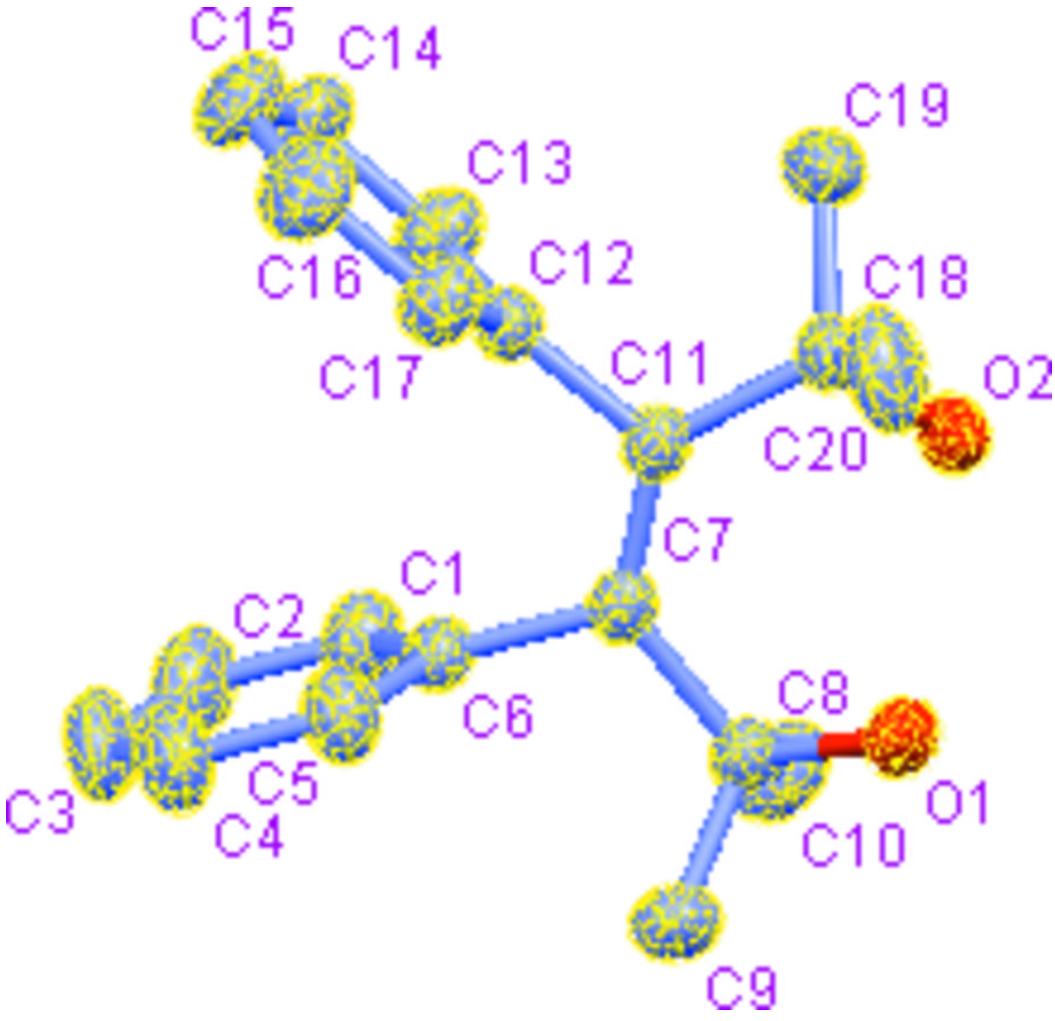
C15
C14
C19
C13
C16
C12
C18
C17
C11
O2
C20
C7
C1
C2
C8
C6
C3
C5
O1
C4
C10
C9

Fig. 2