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Ethyl 2-[2-acetyl-5-(3-methylbut-2-enyloxy)phenoxy]acetate

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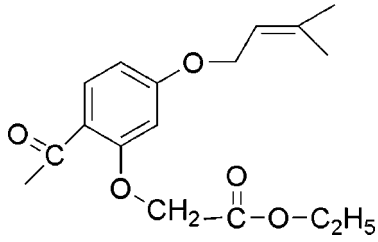
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.003 Å; R factor = 0.039; wR factor = 0.109; data-to-parameter ratio = 13.6.

In the title compound, $C_{17}H_{22}O_5$, an intermediate in the synthesis of the antiulcer agent sofalcone, centrosymmetric dimers are formed in the crystal structure due to a strong C—H···O interaction (H···O = 2.41 Å and C—H···O = 170°).

Related literature

For related literature, see: Kazuaki & Katsuo (1979).



Experimental

Crystal data

$C_{17}H_{22}O_5$
M_r = 306.35
Triclinic, $P\overline{1}$
a = 8.271 (3) Å
b = 9.663 (3) Å
c = 10.514 (4) Å
α = 108.837 (6)°
β = 90.689 (6)°
γ = 94.101 (6)°
V = 792.7 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 294 (2) K
0.34 × 0.30 × 0.26 mm

Data collection

Bruker SMART 1000 CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.954, T_{max} = 0.976
4147 measured reflections
2779 independent reflections
2119 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.109
S = 1.03
2779 reflections
204 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C9—H9B···O2^{i}	0.97	2.41	3.373 (2)	170

Symmetry code: (i) $-x, -y, -z+1$.

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

The authors thank Mr Haibin Song of Nankai University for the X-ray crystallographic determination and for helpful discussions and theory analysis.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2518).

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supplementary materials

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Ethyl 2-[2-acetyl-5-(3-methylbut-2-enyloxy)phenoxy]acetate

C. Zhang, H. Li, D. Liu and M. Liu

Comment

Sofalcone is an antiulcer agent which is effective for the treatment of gastric ulcer(Kazuaki & Katsuo, 1979). The structure of the title compound, (I), an intermediate in the synthesis of sofalcone, is reported here.

As shown in Fig. 1, atoms C13—C17 are almost coplanar, with an r.m.s. deviation from the mean plane of 0.017 (1) Å. This plane and the C1—C6 benzene ring plane form a dihedral angle of 61.86 (7) °. Another plane defined by atoms C9/C10/O2/O3 is almost coplanar with the C1—C6 plane with a dihedral angle of 5.22 (9)°. The packing is consolidated by a C—H···O interaction (Table 1) leading to centrosymmetric dimers (Fig. 2).

Experimental

The title compound was prepared according to the method of Kazuaki & Katsuo (1979). 2,4-Dihydroxyacetophenone (10 g, 0.066 mo1) and anhydrous potassium carbonate (11 g, 0.080 mo1) was dissolved in 120 ml acetone and the mixture was stirred for 0.5 h at room temperature. Then 1-bromo-3-methyl-2-butene (13 g, 0.088 mo1) was added dropwise and the mixture was stirred for 3 h at 298–303 K. The solvent was removed in vacuuo and the residue was cooled to obtain solid 2-hydroxy-4-(3-methylbut-2-enyloxy)acetophenone.

2-hydroxy-4-(3-methylbut-2-enyloxy)acetophenone (10 g, 0.045 mo1) and potassium hydroxide (3.05 g, 0.055 mo1) were dissolved in 67 ml dried acetone. The mixture was stirred for 20 min. Then, ethyl bromoacetate (8 g, 0.048 mo1) was added dropwise. The mixture was stirred for 3 h. The solid was removed by filtration, and the solvent was evaporated *in vacuo*. The residue was cooled to obtain the title compound and colourless blocks of (I) (m.p. 336 K) were obtained by recrystallization from actone.

Refinement

All the H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

Figures

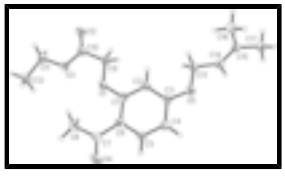

Fig. 1. The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

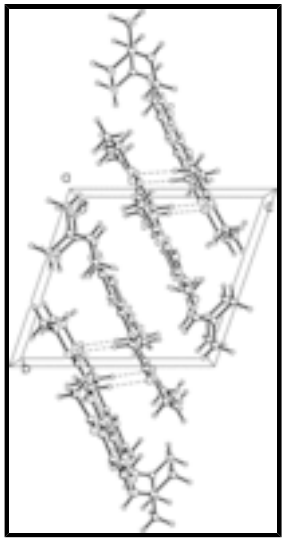

Fig. 2. The crystal packing for (I), with C—H···O interactions shown as dashed lines.

Ethyl 2-[2-acetyl-5-(3-methylbut-2-enyloxy)phenoxy]acetate

Crystal data

$C_{17}H_{22}O_5$	$Z = 2$
$M_r = 306.35$	$F_{000} = 328$
Triclinic, $P\bar{1}$	$D_x = 1.284$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 8.271$ (3) Å	Cell parameters from 2006 reflections
$b = 9.663$ (3) Å	$\theta = 3.2$–26.2º
$c = 10.514$ (4) Å	$\mu = 0.09$ mm^{-1}
$\alpha = 108.837$ (6)º	$T = 294$ (2) K
$\beta = 90.689$ (6)º	Block, colourless
$\gamma = 94.101$ (6)º	0.34 × 0.30 × 0.26 mm
$V = 792.7$ (5) Å^3	

Data collection

Bruker SMART 1000 CCD diffractometer	2779 independent reflections
Radiation source: fine-focus sealed tube	2119 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.026$
$T = 294$(2) K	$\theta_{max} = 25.0$º
ω scans	$\theta_{min} = 2.1$º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -9 \rightarrow 9$
$T_{min} = 0.954$, $T_{max} = 0.976$	$k = -11 \rightarrow 10$
4147 measured reflections	$l = -12 \rightarrow 12$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.039$

$wR(F^2) = 0.109$

$S = 1.03$

2779 reflections

204 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0467P)^2 + 0.1875P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.17$ e Å^{-3}

$\Delta\rho_{min} = -0.15$ e Å^{-3}

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

Extinction coefficient: 0.034 (5)

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 $F^2 > 2\text{sigma}(F^2)$ 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}
O1	0.34331 (13)	0.16237 (13)	0.45657 (12)	0.0523 (3)
O2	0.01594 (14)	−0.08029 (15)	0.29291 (13)	0.0662 (4)
O3	0.28162 (13)	−0.08099 (13)	0.27446 (12)	0.0517 (3)
O4	0.82619 (14)	0.29474 (17)	0.52084 (15)	0.0751 (4)
O5	0.23306 (13)	0.61679 (13)	0.78407 (12)	0.0554 (4)
C1	0.39009 (18)	0.29473 (17)	0.54707 (15)	0.0401 (4)
C2	0.27944 (18)	0.38430 (17)	0.62136 (15)	0.0427 (4)
H2	0.1696	0.3534	0.6117	0.051*
C3	0.33193 (19)	0.51963 (18)	0.70987 (15)	0.0436 (4)
C4	0.4951 (2)	0.56354 (19)	0.72555 (17)	0.0490 (4)
H4	0.5314	0.6547	0.7858	0.059*
C5	0.60211 (19)	0.47321 (19)	0.65274 (17)	0.0475 (4)
H5	0.7119	0.5044	0.6646	0.057*
C6	0.55584 (18)	0.33658 (18)	0.56134 (15)	0.0413 (4)
C7	0.6866 (2)	0.2509 (2)	0.48763 (17)	0.0487 (4)
C8	0.6515 (2)	0.1141 (2)	0.3735 (2)	0.0629 (5)

H8A	0.7514	0.0783	0.3356	0.094*
H8B	0.5856	0.1333	0.3063	0.094*
H8C	0.5946	0.0417	0.4047	0.094*
C9	0.17736 (18)	0.11706 (19)	0.43778 (17)	0.0489 (4)
H9A	0.1194	0.1908	0.4161	0.059*
H9B	0.1357	0.1068	0.5204	0.059*
C10	0.14942 (19)	−0.02523 (19)	0.32741 (16)	0.0463 (4)
C11	0.2603 (2)	−0.21987 (19)	0.16799 (18)	0.0552 (5)
H11A	0.1845	−0.2142	0.0991	0.066*
H11B	0.2181	−0.2961	0.2030	0.066*
C12	0.4202 (3)	−0.2529 (2)	0.1116 (2)	0.0712 (6)
H12A	0.4613	−0.1761	0.0784	0.107*
H12B	0.4103	−0.3445	0.0392	0.107*
H12C	0.4936	−0.2598	0.1804	0.107*
C13	0.06248 (19)	0.58754 (19)	0.75686 (18)	0.0510 (4)
H13A	0.0214	0.5093	0.7897	0.061*
H13B	0.0388	0.5575	0.6608	0.061*
C14	−0.0144 (2)	0.72352 (19)	0.82578 (17)	0.0478 (4)
H14	0.0133	0.8030	0.7966	0.057*
C15	−0.11704 (19)	0.74632 (17)	0.92300 (16)	0.0437 (4)
C16	−0.1704 (2)	0.6375 (2)	0.98913 (19)	0.0587 (5)
H16A	−0.1138	0.5507	0.9531	0.088*
H16B	−0.2850	0.6129	0.9730	0.088*
H16C	−0.1470	0.6784	1.0842	0.088*
C17	−0.1906 (2)	0.8888 (2)	0.9761 (2)	0.0615 (5)
H17A	−0.1407	0.9572	0.9367	0.092*
H17B	−0.1737	0.9265	1.0721	0.092*
H17C	−0.3048	0.8746	0.9539	0.092*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0314 (6)	0.0518 (7)	0.0579 (7)	0.0009 (5)	0.0063 (5)	−0.0039 (6)
O2	0.0364 (7)	0.0719 (9)	0.0689 (9)	−0.0024 (6)	−0.0001 (6)	−0.0051 (7)
O3	0.0376 (6)	0.0504 (7)	0.0534 (7)	0.0022 (5)	0.0063 (5)	−0.0021 (5)
O4	0.0318 (7)	0.0928 (11)	0.0856 (10)	0.0073 (7)	0.0039 (6)	0.0078 (8)
O5	0.0402 (6)	0.0567 (7)	0.0521 (7)	0.0063 (5)	−0.0015 (5)	−0.0064 (6)
C1	0.0356 (8)	0.0455 (9)	0.0364 (8)	0.0019 (7)	0.0019 (6)	0.0096 (7)
C2	0.0301 (8)	0.0513 (10)	0.0415 (9)	0.0020 (7)	0.0028 (7)	0.0080 (7)
C3	0.0400 (9)	0.0489 (9)	0.0375 (9)	0.0071 (7)	0.0017 (7)	0.0070 (7)
C4	0.0414 (9)	0.0497 (10)	0.0479 (10)	−0.0004 (8)	−0.0056 (8)	0.0060 (8)
C5	0.0316 (8)	0.0582 (10)	0.0500 (10)	−0.0016 (8)	−0.0039 (7)	0.0151 (8)
C6	0.0320 (8)	0.0514 (10)	0.0410 (9)	0.0042 (7)	0.0013 (7)	0.0154 (7)
C7	0.0345 (9)	0.0623 (11)	0.0522 (10)	0.0080 (8)	0.0066 (7)	0.0214 (9)
C8	0.0434 (10)	0.0638 (12)	0.0734 (13)	0.0123 (9)	0.0167 (9)	0.0086 (10)
C9	0.0323 (8)	0.0542 (10)	0.0499 (10)	0.0029 (7)	0.0029 (7)	0.0030 (8)
C10	0.0351 (9)	0.0534 (10)	0.0457 (9)	0.0035 (8)	0.0034 (7)	0.0094 (8)
C11	0.0524 (10)	0.0487 (10)	0.0510 (10)	0.0029 (8)	0.0034 (8)	−0.0024 (8)

C12	0.0651 (13)	0.0719 (13)	0.0625 (13)	0.0082 (10)	0.0206 (10)	0.0013 (10)
C13	0.0373 (9)	0.0543 (10)	0.0504 (10)	0.0044 (8)	0.0034 (7)	0.0017 (8)
C14	0.0431 (9)	0.0459 (9)	0.0500 (10)	0.0028 (7)	0.0020 (8)	0.0093 (8)
C15	0.0350 (8)	0.0425 (9)	0.0459 (9)	0.0017 (7)	−0.0030 (7)	0.0042 (7)
C16	0.0515 (11)	0.0604 (12)	0.0627 (12)	0.0071 (9)	0.0078 (9)	0.0173 (9)
C17	0.0509 (11)	0.0504 (11)	0.0746 (13)	0.0097 (9)	0.0103 (9)	0.0069 (9)

Geometric parameters (Å, °)

O1—C1	1.3510 (19)	C9—C10	1.486 (2)
O1—C9	1.4023 (19)	C9—H9A	0.9700
O2—C10	1.1896 (19)	C9—H9B	0.9700
O3—C10	1.304 (2)	C11—C12	1.470 (3)
O3—C11	1.441 (2)	C11—H11A	0.9700
O4—C7	1.208 (2)	C11—H11B	0.9700
O5—C3	1.3457 (19)	C12—H12A	0.9600
O5—C13	1.4260 (19)	C12—H12B	0.9600
C1—C2	1.375 (2)	C12—H12C	0.9600
C1—C6	1.394 (2)	C13—C14	1.474 (2)
C2—C3	1.374 (2)	C13—H13A	0.9700
C2—H2	0.9300	C13—H13B	0.9700
C3—C4	1.377 (2)	C14—C15	1.310 (2)
C4—C5	1.351 (2)	C14—H14	0.9300
C4—H4	0.9300	C15—C16	1.482 (2)
C5—C6	1.383 (2)	C15—C17	1.486 (2)
C5—H5	0.9300	C16—H16A	0.9600
C6—C7	1.480 (2)	C16—H16B	0.9600
C7—C8	1.478 (3)	C16—H16C	0.9600
C8—H8A	0.9600	C17—H17A	0.9600
C8—H8B	0.9600	C17—H17B	0.9600
C8—H8C	0.9600	C17—H17C	0.9600
C1—O1—C9	118.66 (13)	O3—C10—C9	114.29 (13)
C10—O3—C11	116.17 (13)	O3—C11—C12	107.18 (14)
C3—O5—C13	118.37 (12)	O3—C11—H11A	110.3
O1—C1—C2	121.63 (14)	C12—C11—H11A	110.3
O1—C1—C6	116.88 (14)	O3—C11—H11B	110.3
C2—C1—C6	121.49 (15)	C12—C11—H11B	110.3
C3—C2—C1	119.71 (14)	H11A—C11—H11B	108.5
C3—C2—H2	120.1	C11—C12—H12A	109.5
C1—C2—H2	120.1	C11—C12—H12B	109.5
O5—C3—C2	124.21 (14)	H12A—C12—H12B	109.5
O5—C3—C4	115.94 (14)	C11—C12—H12C	109.5
C2—C3—C4	119.85 (15)	H12A—C12—H12C	109.5
C5—C4—C3	119.53 (16)	H12B—C12—H12C	109.5
C5—C4—H4	120.2	O5—C13—C14	107.62 (13)
C3—C4—H4	120.2	O5—C13—H13A	110.2
C4—C5—C6	123.02 (15)	C14—C13—H13A	110.2
C4—C5—H5	118.5	O5—C13—H13B	110.2
C6—C5—H5	118.5	C14—C13—H13B	110.2

C5—C6—C1	116.38 (15)	H13A—C13—H13B	108.5
C5—C6—C7	116.88 (14)	C15—C14—C13	128.05 (17)
C1—C6—C7	126.73 (15)	C15—C14—H14	116.0
O4—C7—C8	119.01 (17)	C13—C14—H14	116.0
O4—C7—C6	119.05 (17)	C14—C15—C16	124.75 (16)
C8—C7—C6	121.94 (15)	C14—C15—C17	121.01 (17)
C7—C8—H8A	109.5	C16—C15—C17	114.24 (16)
C7—C8—H8B	109.5	C15—C16—H16A	109.5
H8A—C8—H8B	109.5	C15—C16—H16B	109.5
C7—C8—H8C	109.5	H16A—C16—H16B	109.5
H8A—C8—H8C	109.5	C15—C16—H16C	109.5
H8B—C8—H8C	109.5	H16A—C16—H16C	109.5
O1—C9—C10	110.65 (13)	H16B—C16—H16C	109.5
O1—C9—H9A	109.5	C15—C17—H17A	109.5
C10—C9—H9A	109.5	C15—C17—H17B	109.5
O1—C9—H9B	109.5	H17A—C17—H17B	109.5
C10—C9—H9B	109.5	C15—C17—H17C	109.5
H9A—C9—H9B	108.1	H17A—C17—H17C	109.5
O2—C10—O3	124.59 (16)	H17B—C17—H17C	109.5
O2—C10—C9	121.11 (15)		

C9—O1—C1—C2	0.7 (2)	C2—C1—C6—C7	−179.87 (15)
C9—O1—C1—C6	−179.11 (15)	C5—C6—C7—O4	8.6 (2)
O1—C1—C2—C3	−178.37 (15)	C1—C6—C7—O4	−172.29 (17)
C6—C1—C2—C3	1.4 (2)	C5—C6—C7—C8	−170.68 (16)
C13—O5—C3—C2	−9.0 (2)	C1—C6—C7—C8	8.4 (3)
C13—O5—C3—C4	170.27 (15)	C1—O1—C9—C10	175.61 (14)
C1—C2—C3—O5	177.99 (15)	C11—O3—C10—O2	−0.7 (3)
C1—C2—C3—C4	−1.3 (2)	C11—O3—C10—C9	179.33 (15)
O5—C3—C4—C5	−178.82 (15)	O1—C9—C10—O2	−177.66 (16)
C2—C3—C4—C5	0.5 (3)	O1—C9—C10—O3	2.3 (2)
C3—C4—C5—C6	0.2 (3)	C10—O3—C11—C12	172.84 (16)
C4—C5—C6—C1	0.0 (3)	C3—O5—C13—C14	−166.29 (15)
C4—C5—C6—C7	179.18 (16)	O5—C13—C14—C15	−116.58 (19)
O1—C1—C6—C5	179.03 (14)	C13—C14—C15—C16	3.5 (3)
C2—C1—C6—C5	−0.8 (2)	C13—C14—C15—C17	−176.47 (16)
O1—C1—C6—C7	−0.1 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9B···O2^{i}	0.97	2.41	3.373 (2)	170

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

Fig. 1

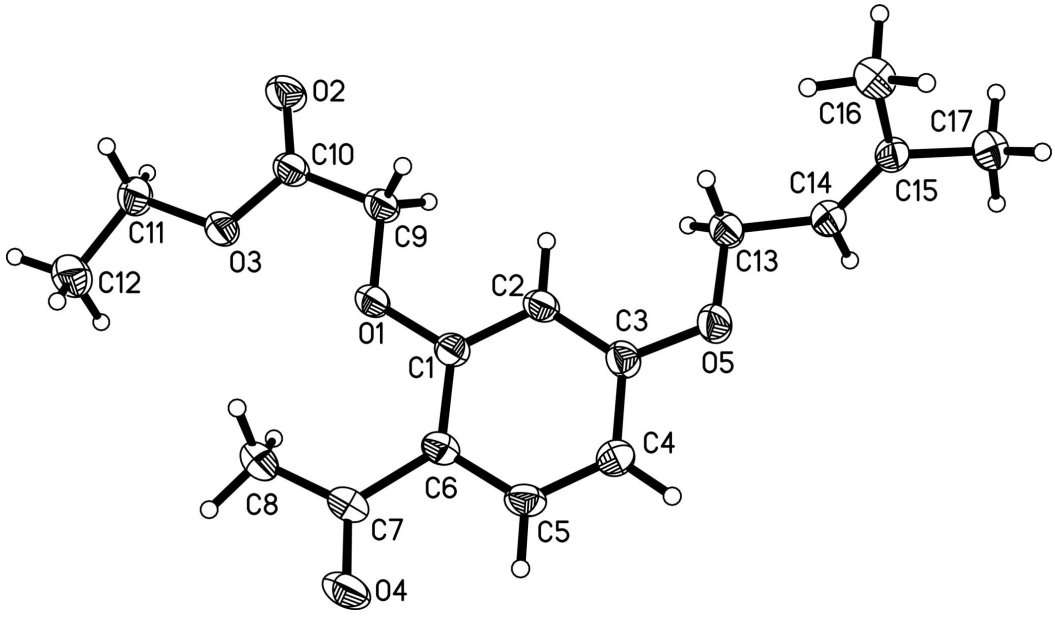
O2
C10
C11
O3
C9
C12
O1
C2
C3
O5
C1
C13
C14
C15
C16
C17
C4
C6
C8
C7
C5
O4

Fig. 2