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## Structure Reports

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2-(4-Chlorophenyl)-2-oxoethyl  
3,4-dimethoxybenzoateHoong-Kun Fun,<sup>a,\*</sup> Ching Kheng Quah,<sup>a,§</sup> A. M. Vijesh,<sup>b,c</sup>  
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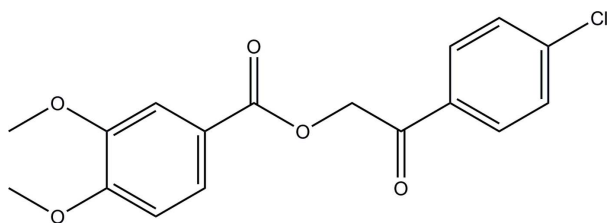
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Key indicators: single-crystal X-ray study;  $T = 296$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.052;  $wR$  factor = 0.234; data-to-parameter ratio = 21.6.

In the title compound,  $\text{C}_{17}\text{H}_{15}\text{ClO}_5$ , the benzene rings forms a dihedral angle of  $74.45$  ( $10$ )°. In the crystal, molecules are linked into  $C(13)$  chains along  $[011]$  via  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bonds. The crystal packing also features short  $\text{Cl}\cdots\text{Cl}$  contacts of  $3.1253$  ( $10$ ) Å.

## Related literature

For a related structure and background to the properties and applications of phenacyl benzoate derivatives, see: Fun *et al.* (2011). For reference bond-length data, see: Allen *et al.* (1987).



## Experimental

## Crystal data

 $\text{C}_{17}\text{H}_{15}\text{ClO}_5$   
 $M_r = 334.74$   
Triclinic,  $P\bar{1}$  $a = 8.2277$  (6) Å  
 $b = 9.3380$  (6) Å  
 $c = 10.5986$  (7) Å $\alpha = 89.062$  (2)°  
 $\beta = 76.752$  (2)°  
 $\gamma = 83.674$  (2)°  
 $V = 787.76$  (9) Å<sup>3</sup>  
 $Z = 2$ Mo  $K\alpha$  radiation  
 $\mu = 0.27$  mm<sup>-1</sup>  
 $T = 296$  K  
 $0.31 \times 0.22 \times 0.13$  mm

## Data collection

Bruker SMART APEXII DUO  
CCD diffractometer  
Absorption correction: multi-scan  
(SADABS; Bruker, 2009)  
 $T_{\min} = 0.922$ ,  $T_{\max} = 0.966$ 12242 measured reflections  
4535 independent reflections  
3056 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.022$ 

## Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.052$   
 $wR(F^2) = 0.234$   
 $S = 1.05$   
4535 reflections210 parameters  
H-atom parameters constrained  
 $\Delta\rho_{\max} = 0.34$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.44$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C2}-\text{H2A}\cdots\text{O5}^i$	0.93	2.58	3.397 (3)	147

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

Data collection: APEX2 (Bruker, 2009); cell refinement: SAINT (Bruker, 2009); 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 and PLATON (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6500).

## References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.  
Bruker (2009). APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.  
Fun, H.-K., Quah, C. K., Garudachari, B., Isloor, A. M. & Satyanarayan, M. N. (2011). *Acta Cryst. E* **67**, o1724.  
Sheldrick, G. M. (2008). *Acta Cryst. A* **64**, 112–122.  
Spek, A. L. (2009). *Acta Cryst. D* **65**, 148–155.

\* Thomson Reuters ResearcherID: A-3561-2009.

§ Thomson Reuters ResearcherID: A-5525-2009.

**supplementary materials**

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## 2-(4-Chlorophenyl)-2-oxoethyl 3,4-dimethoxybenzoate

H.-K. Fun, C. K. Quah, A. M. Vijesh, A. M. Isloor and T. Arulmoli

### Comment

As part of our ongoing studies of phenacyl benzoates (Fun *et al.*, 2011) with possible applications in medicinal chemistry, we hereby report the crystal structure of the title compound, (I).

The molecular structure of the title compound is shown in Fig. 1. The chloro-bound phenyl (C1-C6) and benzene (C10-C15) rings form a dihedral angle of 74.45 (10)°. Bond lengths (Allen *et al.*, 1987) and angles are within normal ranges and are comparable to a related structure (Fun *et al.*, 2011).

In the crystal (Fig. 2), the molecules are linked into one-dimensional chains along [011] via intermolecular C2–H2A...O1<sup>i</sup> hydrogen bonds (Table 1). There is a short Cl1...Cl1 contact (symmetry code: 1-x, -y, -z) with distance = 3.1253 (10) Å which is shorter than the sum of van der Waals radii of the chlorine atoms.

### Experimental

The mixture of 3,4-dimethoxybenzoic acid (1.0 g, 0.0055 mol) potassium carbonate (0.95 g, 0.0069 mol) and 2-bromo-1-(4-chlorophenyl)ethanone (1.28 g, 0.0055 mol) in dimethyl formamide (10 ml) was stirred at room temperature for 2 h. On cooling, colorless needles of (I) begins to separate. They were collected by filtration and recrystallized from ethanol. Yield: 1.59 g, 86.9 %, *m.p.*: 415–416 K.

### Refinement

All H atoms were positioned geometrically and refined using a riding model with C–H = 0.93–0.98 Å and  $U_{\text{iso}}(\text{H}) = 1.2$  or  $1.5 U_{\text{eq}}(\text{C})$ . A rotating-group model was applied for the methyl groups.

### Figures

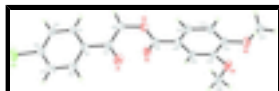


Fig. 1. The molecular structure of the title compound showing 30% probability displacement ellipsoids for non-H atoms.

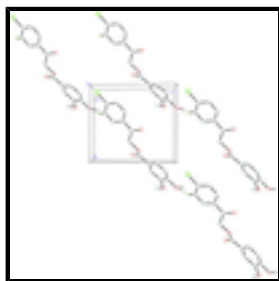


Fig. 2. The crystal structure of the title compound, viewed along the *a* axis. H atoms not involved in hydrogen bonds (dashed lines) have been omitted for clarity.

## 2-(4-Chlorophenyl)-2-oxoethyl 3,4-dimethoxybenzoate

### Crystal data

$C_{17}H_{15}ClO_5$	$Z = 2$
$M_r = 334.74$	$F(000) = 348$
Triclinic, $PT$	$D_x = 1.411 \text{ Mg m}^{-3}$
Hall symbol: $-P 1$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
$a = 8.2277 (6) \text{ \AA}$	Cell parameters from 3650 reflections
$b = 9.3380 (6) \text{ \AA}$	$\theta = 2.6\text{--}29.9^\circ$
$c = 10.5986 (7) \text{ \AA}$	$\mu = 0.27 \text{ mm}^{-1}$
$\alpha = 89.062 (2)^\circ$	$T = 296 \text{ K}$
$\beta = 76.752 (2)^\circ$	Needle, colourless
$\gamma = 83.674 (2)^\circ$	$0.31 \times 0.22 \times 0.13 \text{ mm}$
$V = 787.76 (9) \text{ \AA}^3$	

### Data collection

Bruker SMART APEXII DUO CCD diffractometer	4535 independent reflections
Radiation source: fine-focus sealed tube graphite	3056 reflections with $I > 2\sigma(I)$
$\varphi$ and $\omega$ scans	$R_{\text{int}} = 0.022$
Absorption correction: multi-scan (SADABS; Bruker, 2009)	$\theta_{\text{max}} = 30.1^\circ$ , $\theta_{\text{min}} = 2.0^\circ$
$T_{\text{min}} = 0.922$ , $T_{\text{max}} = 0.966$	$h = -11 \rightarrow 11$
12242 measured reflections	$k = -13 \rightarrow 11$
	$l = -14 \rightarrow 14$

### Refinement

Refinement on $F^2$	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.052$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.234$	H-atom parameters constrained
$S = 1.05$	$w = 1/[\sigma^2(F_o^2) + (0.1525P)^2 + 0.0576P]$
4535 reflections	where $P = (F_o^2 + 2F_c^2)/3$
210 parameters	$(\Delta/\sigma)_{\text{max}} = 0.001$
0 restraints	$\Delta\rho_{\text{max}} = 0.34 \text{ e \AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.44 \text{ e \AA}^{-3}$

### 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  $F^2 > 2\sigma(F^2)$  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	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	0.43412 (9)	0.12795 (7)	0.09809 (7)	0.0826 (3)
O1	0.2269 (3)	0.5869 (2)	0.59869 (14)	0.0726 (5)
O2	0.05337 (18)	0.83733 (16)	0.56180 (15)	0.0570 (4)
O3	0.31494 (19)	0.90247 (18)	0.50295 (14)	0.0618 (4)
O4	0.2833 (2)	1.30026 (19)	0.84644 (15)	0.0648 (5)
O5	-0.0100 (2)	1.30829 (18)	0.99126 (14)	0.0638 (4)
C1	0.1905 (3)	0.4855 (2)	0.27869 (18)	0.0484 (4)
H1A	0.1148	0.5615	0.2632	0.058*
C2	0.2470 (3)	0.3768 (2)	0.1871 (2)	0.0543 (5)
H2A	0.2108	0.3791	0.1101	0.065*
C3	0.3586 (3)	0.2648 (2)	0.2128 (2)	0.0530 (5)
C4	0.4143 (3)	0.2581 (3)	0.3249 (3)	0.0674 (6)
H4A	0.4896	0.1814	0.3397	0.081*
C5	0.3572 (3)	0.3669 (2)	0.4157 (2)	0.0593 (5)
H5A	0.3938	0.3631	0.4926	0.071*
C6	0.2449 (2)	0.48282 (19)	0.39351 (16)	0.0418 (4)
C7	0.1915 (2)	0.5993 (2)	0.49354 (17)	0.0467 (4)
C8	0.0964 (3)	0.7348 (2)	0.4583 (2)	0.0510 (5)
H8A	0.1644	0.7770	0.3828	0.061*
H8B	-0.0054	0.7113	0.4358	0.061*
C9	0.1789 (2)	0.9134 (2)	0.57600 (18)	0.0467 (4)
C10	0.1251 (2)	1.01115 (19)	0.69027 (17)	0.0443 (4)
C11	-0.0310 (3)	1.0126 (2)	0.7725 (2)	0.0510 (5)
H11A	-0.1037	0.9479	0.7590	0.061*
C12	-0.0813 (3)	1.1103 (2)	0.87599 (19)	0.0508 (5)
H12A	-0.1872	1.1106	0.9310	0.061*
C13	0.0261 (2)	1.2065 (2)	0.89671 (17)	0.0457 (4)
C14	0.1885 (2)	1.2027 (2)	0.81470 (17)	0.0449 (4)
C15	0.2367 (2)	1.1067 (2)	0.71224 (17)	0.0433 (4)
H15A	0.3430	1.1050	0.6576	0.052*
C16	0.4543 (3)	1.2941 (4)	0.7765 (2)	0.0756 (8)
H16A	0.5112	1.3597	0.8151	0.113*
H16B	0.4584	1.3204	0.6880	0.113*
H16C	0.5082	1.1979	0.7794	0.113*
C17	-0.1706 (3)	1.3195 (3)	1.0768 (2)	0.0704 (7)
H17A	-0.1783	1.3946	1.1394	0.106*
H17B	-0.1871	1.2296	1.1207	0.106*
H17C	-0.2554	1.3418	1.0284	0.106*

## supplementary materials

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
C11	0.0809 (5)	0.0657 (4)	0.0905 (5)	-0.0085 (3)	0.0055 (3)	-0.0403 (3)
O1	0.0967 (13)	0.0770 (11)	0.0437 (8)	0.0070 (9)	-0.0223 (8)	-0.0125 (7)
O2	0.0492 (8)	0.0534 (8)	0.0659 (9)	-0.0067 (6)	-0.0058 (6)	-0.0238 (7)
O3	0.0507 (8)	0.0716 (10)	0.0580 (8)	-0.0082 (7)	0.0002 (6)	-0.0221 (7)
O4	0.0603 (9)	0.0731 (10)	0.0626 (9)	-0.0264 (8)	-0.0069 (7)	-0.0200 (8)
O5	0.0633 (9)	0.0717 (10)	0.0532 (8)	-0.0114 (7)	-0.0028 (7)	-0.0268 (7)
C1	0.0541 (11)	0.0444 (9)	0.0500 (10)	-0.0020 (7)	-0.0197 (8)	-0.0070 (7)
C2	0.0626 (12)	0.0537 (11)	0.0502 (10)	-0.0094 (9)	-0.0176 (9)	-0.0120 (8)
C3	0.0481 (10)	0.0473 (10)	0.0599 (11)	-0.0090 (8)	-0.0021 (8)	-0.0159 (8)
C4	0.0667 (14)	0.0561 (12)	0.0789 (15)	0.0152 (10)	-0.0252 (12)	-0.0127 (11)
C5	0.0662 (13)	0.0572 (12)	0.0579 (11)	0.0074 (10)	-0.0273 (10)	-0.0083 (9)
C6	0.0432 (9)	0.0416 (9)	0.0418 (8)	-0.0070 (7)	-0.0106 (7)	-0.0033 (7)
C7	0.0491 (10)	0.0497 (10)	0.0411 (8)	-0.0078 (8)	-0.0081 (7)	-0.0074 (7)
C8	0.0501 (10)	0.0487 (10)	0.0545 (10)	-0.0020 (8)	-0.0133 (8)	-0.0156 (8)
C9	0.0438 (9)	0.0447 (9)	0.0496 (9)	-0.0032 (7)	-0.0069 (7)	-0.0080 (7)
C10	0.0456 (9)	0.0411 (9)	0.0446 (9)	-0.0032 (7)	-0.0069 (7)	-0.0079 (7)
C11	0.0482 (10)	0.0498 (10)	0.0532 (10)	-0.0107 (8)	-0.0048 (8)	-0.0099 (8)
C12	0.0463 (10)	0.0543 (11)	0.0474 (9)	-0.0078 (8)	0.0002 (8)	-0.0091 (8)
C13	0.0518 (10)	0.0455 (9)	0.0388 (8)	-0.0035 (7)	-0.0088 (7)	-0.0062 (7)
C14	0.0476 (9)	0.0455 (9)	0.0437 (9)	-0.0094 (7)	-0.0123 (7)	-0.0031 (7)
C15	0.0408 (9)	0.0452 (9)	0.0424 (8)	-0.0048 (7)	-0.0061 (7)	-0.0021 (7)
C16	0.0647 (14)	0.107 (2)	0.0586 (12)	-0.0436 (14)	-0.0054 (10)	-0.0113 (13)
C17	0.0721 (15)	0.0784 (16)	0.0519 (12)	0.0014 (12)	0.0013 (10)	-0.0237 (11)

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

C11—C3	1.739 (2)	C7—C8	1.502 (3)
O1—C7	1.215 (2)	C8—H8A	0.9700
O2—C9	1.353 (2)	C8—H8B	0.9700
O2—C8	1.423 (2)	C9—C10	1.482 (2)
O3—C9	1.201 (2)	C10—C11	1.376 (3)
O4—C14	1.356 (2)	C10—C15	1.407 (2)
O4—C16	1.427 (3)	C11—C12	1.396 (3)
O5—C13	1.352 (2)	C11—H11A	0.9300
O5—C17	1.415 (3)	C12—C13	1.381 (3)
C1—C2	1.382 (3)	C12—H12A	0.9300
C1—C6	1.389 (3)	C13—C14	1.414 (3)
C1—H1A	0.9300	C14—C15	1.377 (2)
C2—C3	1.380 (3)	C15—H15A	0.9300
C2—H2A	0.9300	C16—H16A	0.9600
C3—C4	1.367 (3)	C16—H16B	0.9600
C4—C5	1.378 (3)	C16—H16C	0.9600
C4—H4A	0.9300	C17—H17A	0.9600
C5—C6	1.397 (3)	C17—H17B	0.9600
C5—H5A	0.9300	C17—H17C	0.9600

C6—C7	1.489 (2)		
C9—O2—C8	115.33 (15)	O2—C9—C10	111.37 (16)
C14—O4—C16	117.59 (17)	C11—C10—C15	119.80 (16)
C13—O5—C17	118.36 (17)	C11—C10—C9	121.82 (16)
C2—C1—C6	121.01 (18)	C15—C10—C9	118.36 (16)
C2—C1—H1A	119.5	C10—C11—C12	120.61 (17)
C6—C1—H1A	119.5	C10—C11—H11A	119.7
C3—C2—C1	118.36 (18)	C12—C11—H11A	119.7
C3—C2—H2A	120.8	C13—C12—C11	119.99 (17)
C1—C2—H2A	120.8	C13—C12—H12A	120.0
C4—C3—C2	122.28 (19)	C11—C12—H12A	120.0
C4—C3—C11	118.43 (18)	O5—C13—C12	125.37 (18)
C2—C3—C11	119.28 (17)	O5—C13—C14	115.02 (16)
C3—C4—C5	119.0 (2)	C12—C13—C14	119.61 (16)
C3—C4—H4A	120.5	O4—C14—C15	125.87 (17)
C5—C4—H4A	120.5	O4—C14—C13	114.09 (16)
C4—C5—C6	120.66 (19)	C15—C14—C13	120.04 (16)
C4—C5—H5A	119.7	C14—C15—C10	119.91 (16)
C6—C5—H5A	119.7	C14—C15—H15A	120.0
C1—C6—C5	118.69 (18)	C10—C15—H15A	120.0
C1—C6—C7	123.08 (17)	O4—C16—H16A	109.5
C5—C6—C7	118.22 (16)	O4—C16—H16B	109.5
O1—C7—C6	121.47 (18)	H16A—C16—H16B	109.5
O1—C7—C8	121.02 (18)	O4—C16—H16C	109.5
C6—C7—C8	117.49 (15)	H16A—C16—H16C	109.5
O2—C8—C7	111.92 (16)	H16B—C16—H16C	109.5
O2—C8—H8A	109.2	O5—C17—H17A	109.5
C7—C8—H8A	109.2	O5—C17—H17B	109.5
O2—C8—H8B	109.2	H17A—C17—H17B	109.5
C7—C8—H8B	109.2	O5—C17—H17C	109.5
H8A—C8—H8B	107.9	H17A—C17—H17C	109.5
O3—C9—O2	123.14 (17)	H17B—C17—H17C	109.5
O3—C9—C10	125.48 (17)		
C6—C1—C2—C3	0.3 (3)	O2—C9—C10—C11	3.8 (3)
C1—C2—C3—C4	0.0 (3)	O3—C9—C10—C15	4.4 (3)
C1—C2—C3—C11	-179.06 (15)	O2—C9—C10—C15	-174.77 (16)
C2—C3—C4—C5	0.0 (4)	C15—C10—C11—C12	1.5 (3)
C11—C3—C4—C5	179.11 (19)	C9—C10—C11—C12	-177.06 (18)
C3—C4—C5—C6	-0.4 (4)	C10—C11—C12—C13	-0.1 (3)
C2—C1—C6—C5	-0.7 (3)	C17—O5—C13—C12	-0.3 (3)
C2—C1—C6—C7	178.23 (17)	C17—O5—C13—C14	179.6 (2)
C4—C5—C6—C1	0.7 (3)	C11—C12—C13—O5	178.19 (19)
C4—C5—C6—C7	-178.2 (2)	C11—C12—C13—C14	-1.7 (3)
C1—C6—C7—O1	172.1 (2)	C16—O4—C14—C15	-6.9 (3)
C5—C6—C7—O1	-8.9 (3)	C16—O4—C14—C13	174.0 (2)
C1—C6—C7—C8	-9.6 (3)	O5—C13—C14—O4	1.4 (3)
C5—C6—C7—C8	169.30 (19)	C12—C13—C14—O4	-178.68 (19)
C9—O2—C8—C7	79.1 (2)	O5—C13—C14—C15	-177.72 (17)

## supplementary materials

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O1—C7—C8—O2	-1.5 (3)	C12—C13—C14—C15	2.2 (3)
C6—C7—C8—O2	-179.80 (15)	O4—C14—C15—C10	-179.87 (18)
C8—O2—C9—O3	4.0 (3)	C13—C14—C15—C10	-0.8 (3)
C8—O2—C9—C10	-176.80 (16)	C11—C10—C15—C14	-1.0 (3)
O3—C9—C10—C11	-177.1 (2)	C9—C10—C15—C14	177.60 (16)

### *Hydrogen-bond geometry (Å, °)*

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
C2—H2A $\cdots$ O5 <sup>i</sup>	0.93	2.58	3.397 (3)	147

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



Fig. 1

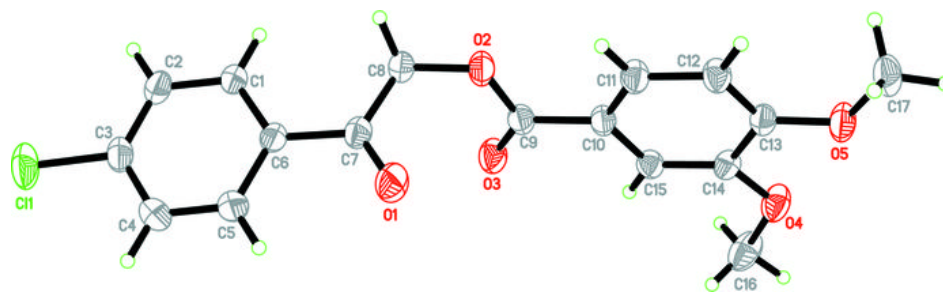


Fig. 2

