

## Ethyl 2-(3-methoxyphenyl)acetate

Ghulam Qadeer,<sup>a</sup> Nasim Hasan Rama,<sup>a\*</sup> Murat Taşb and Okan Zafer Yeşilel<sup>c</sup>

<sup>a</sup>Department of Chemistry, Quaid-i-Azam University, Islamabad 45320, Pakistan,

<sup>b</sup>Department of Chemistry, Faculty of Arts and Sciences, Giresun University, Giresun,

Turkey, and <sup>c</sup>Department of Chemistry, Faculty of Arts and Sciences, Eskişehir

Osmangazi University, Eskişehir, Turkey

Correspondence e-mail: nasimhrama@yahoo.com

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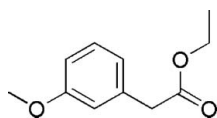
Key indicators: single-crystal X-ray study;  $T = 294$  K; mean  $\sigma(\text{C}-\text{C}) = 0.012$  Å;

$R$  factor = 0.145;  $wR$  factor = 0.345; data-to-parameter ratio = 19.9.

The title compound,  $\text{C}_{11}\text{H}_{14}\text{O}_3$ , is an important intermediate in the synthesis of biologically active heterocyclic compounds. The ester group is oriented with respect to the benzene ring at a dihedral angle of  $5.38$  ( $2$ )°. An intramolecular  $\text{C}-\text{H}\cdots\text{O}$  hydrogen bond results in the formation of a nearly planar six-membered ring, which is oriented with respect to the adjacent benzene ring at a dihedral angle of  $3.92$  ( $2$ )°. Thus, the two rings are nearly coplanar.

### Related literature

For related literature, see: Zheng *et al.* (2003); Al-Talib *et al.* (1990); Yousif *et al.* (1986); Ahmad *et al.* (2001); Al-Soud *et al.* (2004); El-Emam *et al.* (2004); Weinstock *et al.* (1991). For bond-length data, see: Bruno *et al.* (2004).



### Experimental

#### Crystal data

$\text{C}_{11}\text{H}_{14}\text{O}_3$

$M_r = 194.22$

Monoclinic,  $P2_1/n$

$a = 14.868$  ( $2$ ) Å

$b = 4.851$  ( $2$ ) Å

$c = 15.428$  ( $4$ ) Å

$\beta = 107.70$  ( $3$ )°

$V = 1060.1$  ( $6$ ) Å<sup>3</sup>

$Z = 4$

Mo  $K\alpha$  radiation

$\mu = 0.09$  mm<sup>-1</sup>

$T = 294$  ( $2$ ) K

$0.40 \times 0.30 \times 0.30$  mm

#### Data collection

Bruker SMART CCD area-detector diffractometer

Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.960$ ,  $T_{\max} = 0.970$

7468 measured reflections

2564 independent reflections

1976 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.021$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.145$

$wR(F^2) = 0.345$

$S = 1.08$

2564 reflections

129 parameters

1 restraint

H-atom parameters constrained

$\Delta\rho_{\text{max}} = 0.60$  e Å<sup>-3</sup>

$\Delta\rho_{\text{min}} = -0.56$  e Å<sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C7}-\text{H7}\cdots\text{O2}$	0.93	2.17	2.76 (1)	121

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); 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, 1999); 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: HK2373).

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**supplementary materials**

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## Ethyl 2-(3-methoxyphenyl)acetate

G. Qadeer, N. H. Rama, M. Tas and O. Z. Yesilel

### Comment

The title compound, (I), is a lachrymator and a drug intermediate. It is widely employed in synthetic organic chemistry for example, 2,5-disubstituted-1,3,4-oxadiazoles (Zheng *et al.*, 2003; Al-Talib *et al.*, 1990) and 5-substituted-2-mercapto-1,3,4-oxadiazoles (Yousif *et al.*, 1986; Ahmad *et al.*, 2001; Al-Soud *et al.*, 2004; El-Emam *et al.*, 2004). In addition, methyl 4-(bromomethyl)-benzoate has been used in the synthesis of 1-(carboxybenzyl)imidazole-5-acrylic acids, which are potent and selective angiotensin II receptor antagonists (Weinstock *et al.*, 1991). In view of the versatility of these compounds, we have synthesized the title compound, (I), and reported herein its crystal structure.

The title molecule (Fig. 1) shows no unusual features when compared with the Mogul norms (Bruno *et al.*, 2004). The dihedral angle between benzene ring A (C5—C10) and the planar ester group is  $5.38(2)^\circ$ . The intramolecular C—H $\cdots$ O hydrogen bond (Table 1) results in the formation of a nearly planar six-membered ring; B (O2/C1/C2/C7/C8/H7), which is oriented with respect to the adjacent ring A, at a dihedral angle of A/B =  $3.92(2)^\circ$ . So, rings A and B are nearly co-planar.

As can be seen from the packing diagram (Fig. 2), the molecules are stacked along the *a* axis and enlotted along the *b* axis.

### Experimental

For the preparation of the title compound, a mixture of 2-(3-methoxyphenyl)-acetic acid (1.66 g, 10 mmol) and absolute ethanol (50 ml) in the presence of a few drops of sulphuric acid was refluxed for 5 h. The excess solvent was removed by distillation. The solid residue was filtered off, washed with water and recrystallized from ethanol (30%) to give the title compound (yield; 83%, m.p. 398–399 K). Colorless single crystals of (I) were obtained by slow evaporation of an ethanol solution at room temperature.

### Refinement

H atoms were positioned geometrically, with C—H = 0.93, 0.97 and 0.96 Å, for aromatic, methylene and methyl H atoms, respectively, and constrained to ride on their parent atoms, with  $U_{\text{iso}}(\text{H}) = xU_{\text{eq}}(\text{C})$ , where  $x = 1.5$  for methyl H and  $x = 1.2$  for all other H atoms.

## Figures

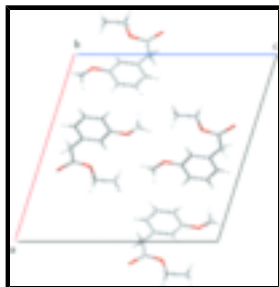
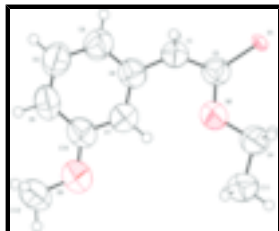


Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

Fig. 2. A packing diagram of (I).

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### Crystal data

$C_{11}H_{14}O_3$

$M_r = 194.22$

Monoclinic,  $P2_1/n$

Hall symbol:  $-P\ 2_1/n$

$a = 14.868\ (2)\ \text{\AA}$

$b = 4.851\ (2)\ \text{\AA}$

$c = 15.428\ (4)\ \text{\AA}$

$\beta = 107.70\ (3)^\circ$

$V = 1060.1\ (6)\ \text{\AA}^3$

$Z = 4$

$F_{000} = 416$

$D_x = 1.217\ \text{Mg m}^{-3}$

Melting point:  $398(1)\ \text{K}$

Mo  $K\alpha$  radiation

$\lambda = 0.71073\ \text{\AA}$

Cell parameters from 1609 reflections

$\theta = 2.9\text{--}23.6^\circ$

$\mu = 0.09\ \text{mm}^{-1}$

$T = 294\ (2)\ \text{K}$

Block, colorless

$0.40 \times 0.30 \times 0.30\ \text{mm}$

### Data collection

Bruker SMART CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 294(2)\ \text{K}$

$\varphi$  and  $\omega$  scans

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

$T_{\min} = 0.960$ ,  $T_{\max} = 0.970$

2564 independent reflections

1976 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.021$

$\theta_{\text{max}} = 28.3^\circ$

$\theta_{\text{min}} = 2.3^\circ$

$h = -19 \rightarrow 19$

$k = -6 \rightarrow 6$

7468 measured reflections

$l = -20 \rightarrow 20$

*Refinement*

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.145$	H-atom parameters constrained
$wR(F^2) = 0.345$	$w = 1/[\sigma^2(F_o^2) + (0.5P)^2]$
$S = 1.08$	where $P = (F_o^2 + 2F_c^2)/3$
2564 reflections	$(\Delta/\sigma)_{\max} = 0.090$
129 parameters	$\Delta\rho_{\max} = 0.60 \text{ e } \text{\AA}^{-3}$
1 restraint	$\Delta\rho_{\min} = -0.56 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: SHELXL, $F_c^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
	Extinction coefficient: 0.8 (3)

*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  $F^2 > 2\sigma(F^2)$  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 ( $\text{\AA}^2$ )*

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.38198 (14)	0.3166 (5)	0.90034 (13)	0.0343 (12)
O2	0.4028 (3)	0.4892 (10)	0.7460 (3)	0.0844 (17)
O3	0.5735 (5)	1.0374 (15)	0.6114 (5)	0.115 (2)
C1	0.5010 (5)	0.6757 (11)	0.8699 (4)	0.0728 (17)
H1A	0.5500	0.5672	0.9119	0.087*
H1B	0.4778	0.7992	0.9078	0.087*
C2	0.4286 (4)	0.4921 (11)	0.8377 (4)	0.0729 (18)
C4	0.3303 (5)	0.2921 (18)	0.6985 (6)	0.087 (2)
H4A	0.2702	0.3382	0.7073	0.104*
H4B	0.3478	0.1070	0.7211	0.104*
C5	0.6666 (6)	1.2032 (17)	0.8505 (8)	0.100 (3)
H5	0.7092	1.3204	0.8902	0.120*
C6	0.6564 (6)	1.2170 (15)	0.7615 (7)	0.090 (2)
H6	0.6929	1.3378	0.7396	0.108*
C7	0.5393 (6)	0.8629 (14)	0.7361 (5)	0.085 (2)

## supplementary materials

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H7	0.4965	0.7449	0.6968	0.102*
C8	0.5514 (5)	0.8513 (12)	0.8280 (5)	0.0758 (19)
C9	0.6160 (6)	1.0213 (16)	0.8843 (6)	0.092 (2)
H9	0.6257	1.0133	0.9467	0.111*
C10	0.5896 (5)	1.0460 (15)	0.7020 (5)	0.083 (2)
C11	0.3237 (7)	0.310 (2)	0.6010 (6)	0.104 (3)
H11A	0.2759	0.1855	0.5666	0.156*
H11B	0.3834	0.2614	0.5932	0.156*
H11C	0.3075	0.4950	0.5798	0.156*
C12	0.6114 (9)	1.247 (2)	0.5697 (7)	0.112 (3)
H12A	0.5923	1.2170	0.5051	0.168*
H12B	0.6791	1.2428	0.5931	0.168*
H12C	0.5888	1.4227	0.5824	0.168*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.0308 (16)	0.0497 (17)	0.0225 (15)	-0.0118 (8)	0.0081 (10)	0.0106 (7)
O2	0.085 (3)	0.093 (3)	0.072 (3)	-0.014 (2)	0.020 (2)	0.0118 (19)
O3	0.127 (5)	0.130 (5)	0.098 (4)	-0.035 (4)	0.047 (4)	-0.002 (3)
C1	0.075 (3)	0.076 (3)	0.064 (3)	-0.005 (2)	0.016 (3)	0.004 (2)
C2	0.070 (3)	0.077 (3)	0.070 (4)	0.004 (2)	0.019 (3)	0.004 (2)
C4	0.071 (4)	0.111 (5)	0.075 (4)	-0.008 (3)	0.018 (3)	0.006 (3)
C5	0.090 (5)	0.092 (5)	0.106 (6)	-0.016 (3)	0.012 (5)	0.009 (4)
C6	0.079 (4)	0.081 (4)	0.111 (6)	-0.008 (3)	0.031 (4)	0.021 (4)
C7	0.090 (4)	0.086 (4)	0.078 (4)	-0.011 (3)	0.023 (3)	-0.005 (3)
C8	0.077 (4)	0.071 (3)	0.079 (4)	0.005 (2)	0.023 (3)	0.008 (2)
C9	0.083 (4)	0.097 (4)	0.086 (5)	-0.010 (3)	0.009 (3)	0.009 (3)
C10	0.080 (4)	0.088 (4)	0.085 (4)	0.006 (3)	0.032 (3)	0.011 (3)
C11	0.094 (5)	0.136 (8)	0.073 (5)	-0.019 (4)	0.011 (4)	0.007 (4)
C12	0.148 (9)	0.108 (6)	0.090 (5)	-0.014 (6)	0.051 (6)	0.012 (5)

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

O1—C2	1.596 (6)	C5—H5	0.9300
O2—C2	1.348 (8)	C6—C10	1.401 (12)
O2—C4	1.460 (9)	C6—H6	0.9300
C1—C2	1.369 (9)	C7—C10	1.365 (10)
C1—C8	1.414 (9)	C7—C8	1.376 (11)
C1—H1A	0.9700	C7—H7	0.9300
C1—H1B	0.9700	C8—C9	1.360 (11)
O3—C10	1.344 (10)	C9—H9	0.9300
O3—C12	1.409 (11)	C11—H11A	0.9600
C4—C11	1.480 (12)	C11—H11B	0.9600
C4—H4A	0.9700	C11—H11C	0.9600
C4—H4B	0.9700	C12—H12A	0.9600
C5—C6	1.336 (14)	C12—H12B	0.9600
C5—C9	1.362 (12)	C12—H12C	0.9600

C2—O2—C4	117.7 (5)	C10—C7—H7	119.7
C2—C1—C8	133.9 (6)	C8—C7—H7	119.7
C2—C1—H1A	103.8	C9—C8—C7	118.6 (7)
C8—C1—H1A	103.7	C9—C8—C1	116.3 (7)
C2—C1—H1B	103.7	C7—C8—C1	125.1 (6)
C8—C1—H1B	103.7	C8—C9—C5	120.9 (8)
H1A—C1—H1B	105.4	C8—C9—H9	119.5
O2—C2—C1	109.2 (5)	C5—C9—H9	119.6
O2—C2—O1	126.3 (5)	O3—C10—C7	115.8 (7)
C1—C2—O1	124.4 (5)	O3—C10—C6	124.5 (7)
C10—O3—C12	118.9 (7)	C7—C10—C6	119.7 (7)
O2—C4—C11	106.4 (6)	C4—C11—H11A	109.4
O2—C4—H4A	110.4	C4—C11—H11B	109.5
C11—C4—H4A	110.5	H11A—C11—H11B	109.5
O2—C4—H4B	110.4	C4—C11—H11C	109.5
C11—C4—H4B	110.4	H11A—C11—H11C	109.5
H4A—C4—H4B	108.6	H11B—C11—H11C	109.5
C6—C5—C9	121.5 (8)	O3—C12—H12A	109.5
C6—C5—H5	119.2	O3—C12—H12B	109.5
C9—C5—H5	119.2	H12A—C12—H12B	109.5
C5—C6—C10	118.7 (7)	O3—C12—H12C	109.5
C5—C6—H6	120.7	H12A—C12—H12C	109.5
C10—C6—H6	120.6	H12B—C12—H12C	109.5
C10—C7—C8	120.6 (7)		
C4—O2—C2—C1	176.1 (5)	C7—C8—C9—C5	1.2 (12)
C4—O2—C2—O1	-5.2 (9)	C1—C8—C9—C5	-178.4 (8)
C8—C1—C2—O2	-0.5 (10)	C6—C5—C9—C8	-1.6 (14)
C8—C1—C2—O1	-179.3 (6)	C12—O3—C10—C7	170.4 (8)
C2—O2—C4—C11	-173.6 (6)	C12—O3—C10—C6	-13.0 (13)
C9—C5—C6—C10	2.2 (13)	C8—C7—C10—O3	179.0 (7)
C10—C7—C8—C9	-1.5 (11)	C8—C7—C10—C6	2.2 (11)
C10—C7—C8—C1	178.0 (7)	C5—C6—C10—O3	-179.0 (8)
C2—C1—C8—C9	175.6 (7)	C5—C6—C10—C7	-2.5 (11)
C2—C1—C8—C7	-4.0 (12)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C7—H7...O2	0.93	2.17	2.76 (1)	121

Fig. 1

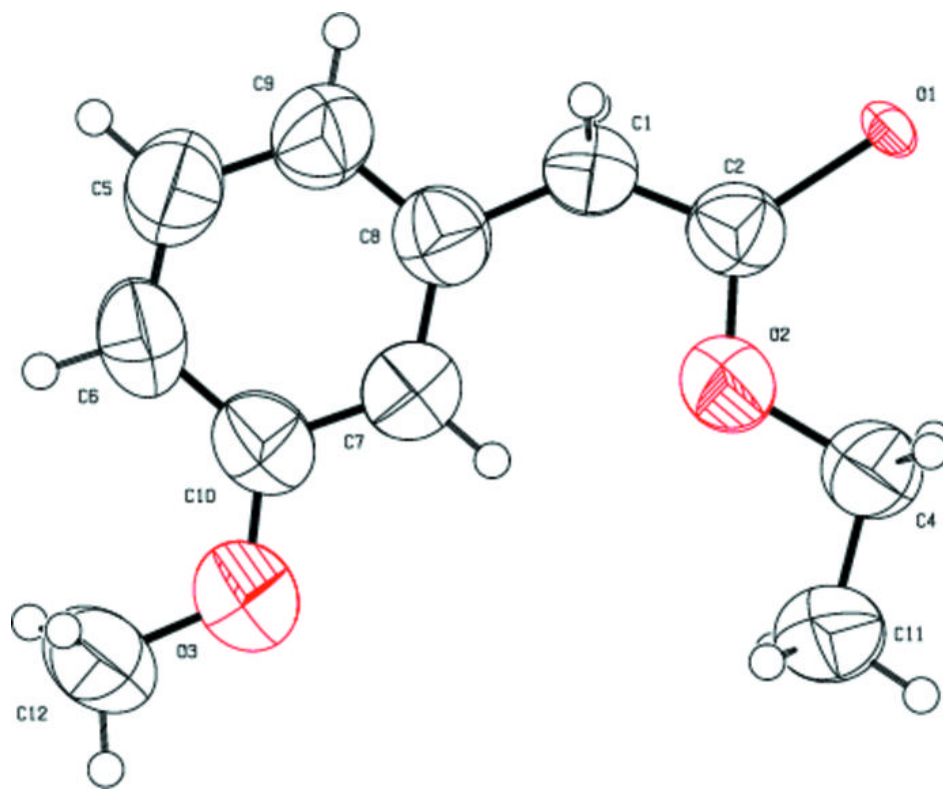




Fig. 2

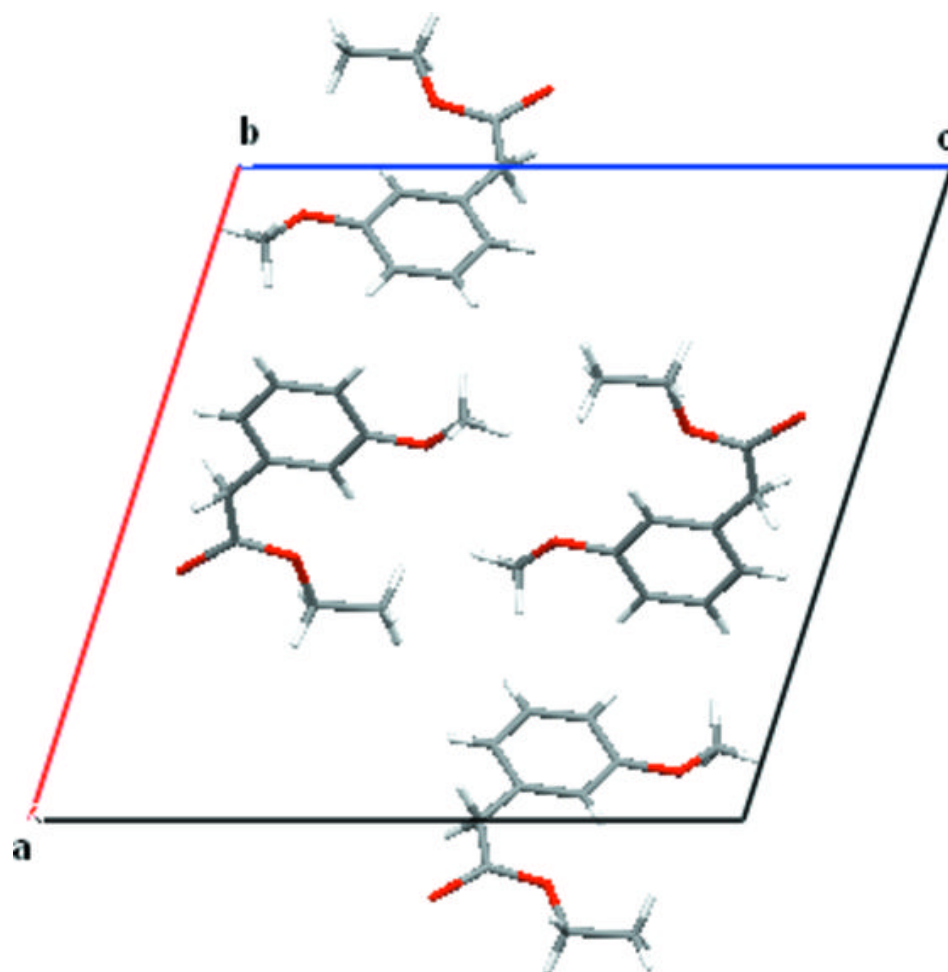


Fig. 3

