Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# Ethyl (*Z*)-2-(4-chlorobenzylidene)-3-oxobutanoate

**Shaaban K. Mohamed,[a] Antar A. Abdelhamid,[b] Atash V. Gurbanov,[b] A. M. Maharramov[b] and Seik Weng Ng[c]***

[a]School of Biology, Chemistry and Material Science, Manchester Metropolitan University, Manchester, England, [b]Department of Organic Chemistry, Baku State University, Baku, Azerbaijan, and [c]Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
Correspondence e-mail: seikweng@um.edu.my

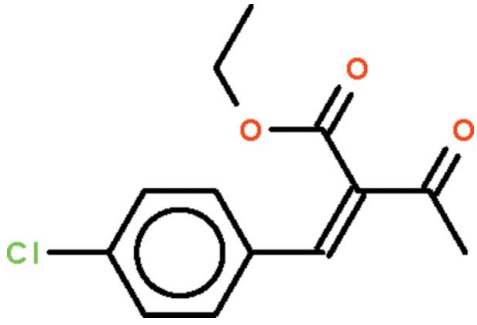
Received 6 January 2011; accepted 6 January 2011

Key indicators: single-crystal X-ray study; $T$ = 295 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.065; $wR$ factor = 0.201; data-to-parameter ratio = 17.9.

The C=C double-bond in the title compound, $C_{13}H_{13}ClO_3$, has a $Z$ configuration. The aliphatic substituents at one end of the double bond, *i.e.* the $CH_3CO$– and $C_2H_5O_2C$– groups, are aligned at 82.1 (3)° with respect to each other.

## Related literature

For related structures, see: Deng *et al.* (2007); Shi (2008).




## Experimental

### Crystal data

$C_{13}H_{13}ClO_3$
$M_r$ = 252.68
Monoclinic, $P2_1/n$
$a$ = 9.9956 (6) Å
$b$ = 7.7487 (5) Å
$c$ = 16.2709 (10) Å
$\beta$ = 99.624 (1)°
$V$ = 1242.49 (13) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.30 mm$^{-1}$
$T$ = 295 K
0.20 × 0.20 × 0.20 mm

### Data collection

Bruker APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.942, $T_{max}$ = 0.942
11255 measured reflections
2790 independent reflections
1968 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.069

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.201
$S$ = 1.02
2790 reflections
156 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.40 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.52 e Å$^{-3}$

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

We thank Manchester Metropolitan University, Baku State University and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2789).

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# supporting information

*Acta Cryst.* (2011). E**67**, o365 [doi:10.1107/S1600536811000717]

## Ethyl (*Z*)-2-(4-chlorobenzylidene)-3-oxobutanoate

**Shaaban K. Mohamed, Antar A. Abdelhamid, Atash V. Gurbanov, A. M. Maharramov and Seik Weng Ng**

### S1. Comment

Trizma is a mildly-basic primary aminoalcohol that catalyzes Knoevenagel condensation reactions. The yield can be high under microwave irradiation; the title compound has been synthesized albeit by a conventional route. The carbon-carbon double-bond in $C_{13}H_{13}ClO_3$ has a *Z*-configuration. The aliphatic substituents at one end of the double-bond, *i.e.*, the $CH_3CO$– and planar $C_2H_5O_2C$– groups, are aligned at 82.1 (3) ° with respect to each other. Bond dimensions in the molecule compare favorably with those found in similar molecules (Deng *et al.*, 2007; Shi, 2008).

### S2. Experimental

Trizma (0.01 mol), *p*-chlorobenzaldehyde (0.01 mol) and ethyl acetoacetate (0.02 mol) were heated in ethanol (50 ml) for 3 h. The reaction was monitored with TLC. The solid that separated was collected and recrystallized from ethanol to give a colorless crystals, m.p. 373 K (60% yield).

### S3. Refinement

Carbon-bound H-atoms were placed in calculated positions [C–H 0.93 to 0.97 Å; *U*(H) 1.2 to 1.5*U*(C)] and were included in the refinement in the riding model approximation, with *U*(H) set to 1.2 to 1.5*U*(C).

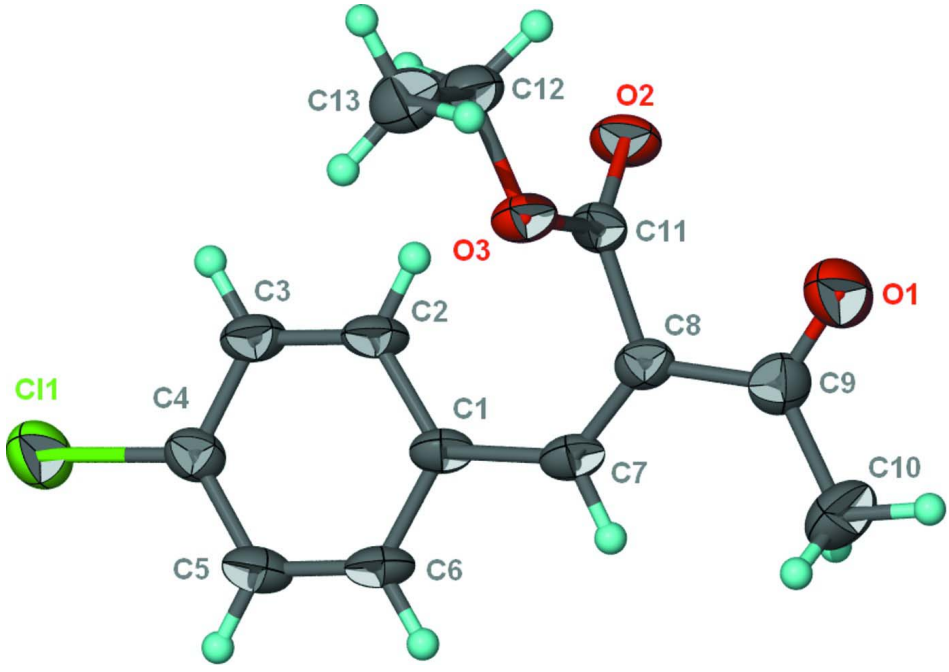


**Figure 1**

Thermal ellipsoid plot (Barbour, 2001) of $C_{13}H_{13}ClO_3$ at the 50% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

### Ethyl (*Z*)-2-(4-chlorobenzylidene)-3-oxobutanoate

*Crystal data*

$C_{13}H_{13}ClO_3$
$M_r = 252.68$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 9.9956$ (6) Å
$b = 7.7487$ (5) Å
$c = 16.2709$ (10) Å
$\beta = 99.624$ (1)°
$V = 1242.49$ (13) Å$^3$
$Z = 4$
$F(000) = 528$
$D_x = 1.351$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3560 reflections
$\theta = 2.2$–$27.8°$
$\mu = 0.30$ mm$^{-1}$
$T = 295$ K
Prism, colorless
0.20 × 0.20 × 0.20 mm

*Data collection*

Bruker APEXII diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.942$, $T_{max} = 0.942$
11255 measured reflections
2790 independent reflections
1968 reflections with $I > 2\sigma(I)$
$R_{int} = 0.069$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.2°$
$h = -12 \rightarrow 12$
$k = -10 \rightarrow 9$
$l = -21 \rightarrow 21$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.065$
$wR(F^2) = 0.201$
$S = 1.02$
2790 reflections
156 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.1314P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.40$ e Å$^{-3}$
$\Delta\rho_{min} = -0.52$ e Å$^{-3}$

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Cl1 | 0.17886 (7) | 0.43778 (11) | 0.15024 (4) | 0.0663 (3) |
| O1 | 0.2270 (2) | 0.3753 (3) | 0.72305 (11) | 0.0703 (6) |
| O2 | 0.42363 (15) | 0.4336 (2) | 0.59728 (11) | 0.0555 (5) |
| O3 | 0.27248 (13) | 0.6486 (2) | 0.56982 (10) | 0.0452 (4) |
| C1 | 0.13562 (19) | 0.3488 (3) | 0.41918 (13) | 0.0399 (5) |
| C2 | 0.2540 (2) | 0.4199 (3) | 0.39776 (14) | 0.0453 (6) |
| H2A | 0.3252 | 0.4497 | 0.4397 | 0.054* |
| C3 | 0.2669 (2) | 0.4463 (3) | 0.31626 (14) | 0.0467 (6) |
| H3 | 0.3460 | 0.4940 | 0.3031 | 0.056* |
| C4 | 0.1611 (2) | 0.4015 (3) | 0.25368 (14) | 0.0437 (5) |
| C5 | 0.0440 (2) | 0.3274 (3) | 0.27150 (14) | 0.0490 (6) |
| H5 | −0.0258 | 0.2957 | 0.2290 | 0.059* |
| C6 | 0.03270 (19) | 0.3015 (3) | 0.35374 (14) | 0.0453 (5) |
| H6 | −0.0458 | 0.2509 | 0.3662 | 0.054* |
| C7 | 0.11396 (19) | 0.3175 (3) | 0.50496 (14) | 0.0418 (5) |
| H7 | 0.0387 | 0.2501 | 0.5098 | 0.050* |
| C8 | 0.18613 (19) | 0.3714 (3) | 0.57758 (13) | 0.0405 (5) |
| C9 | 0.1549 (2) | 0.3219 (3) | 0.66085 (15) | 0.0501 (6) |
| C10 | 0.0339 (3) | 0.2111 (4) | 0.66738 (18) | 0.0693 (8) |
| H10A | 0.0333 | 0.1832 | 0.7248 | 0.104* |
| H10B | 0.0385 | 0.1068 | 0.6362 | 0.104* |
| H10C | −0.0476 | 0.2727 | 0.6453 | 0.104* |
| C11 | 0.3088 (2) | 0.4853 (3) | 0.58325 (13) | 0.0395 (5) |
| C12 | 0.3811 (2) | 0.7742 (3) | 0.57098 (17) | 0.0535 (6) |
| H12A | 0.4275 | 0.7563 | 0.5239 | 0.064* |
| H12B | 0.4466 | 0.7634 | 0.6219 | 0.064* |
| C13 | 0.3162 (3) | 0.9486 (3) | 0.56615 (18) | 0.0592 (7) |
| H13A | 0.3836 | 1.0353 | 0.5626 | 0.089* |
| H13B | 0.2764 | 0.9679 | 0.6151 | 0.089* |
| H13C | 0.2470 | 0.9546 | 0.5176 | 0.089* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cl1 | 0.0540 (4) | 0.0967 (7) | 0.0483 (4) | 0.0031 (3) | 0.0085 (3) | 0.0024 (3) |
| O1 | 0.0600 (11) | 0.0996 (17) | 0.0513 (11) | −0.0032 (11) | 0.0095 (9) | 0.0006 (10) |
| O2 | 0.0281 (8) | 0.0621 (12) | 0.0735 (12) | 0.0041 (7) | −0.0001 (7) | 0.0076 (8) |
| O3 | 0.0282 (7) | 0.0479 (10) | 0.0601 (9) | −0.0011 (6) | 0.0091 (6) | −0.0004 (7) |
| C1 | 0.0237 (8) | 0.0445 (12) | 0.0514 (12) | −0.0006 (8) | 0.0061 (8) | −0.0051 (9) |
| C2 | 0.0235 (9) | 0.0614 (15) | 0.0504 (12) | −0.0061 (9) | 0.0049 (8) | −0.0091 (10) |
| C3 | 0.0280 (10) | 0.0594 (15) | 0.0533 (13) | −0.0052 (9) | 0.0086 (9) | −0.0040 (10) |
| C4 | 0.0335 (10) | 0.0524 (13) | 0.0446 (11) | 0.0046 (9) | 0.0050 (8) | −0.0025 (10) |
| C5 | 0.0295 (10) | 0.0605 (14) | 0.0540 (13) | −0.0031 (10) | −0.0013 (9) | −0.0107 (11) |
| C6 | 0.0237 (9) | 0.0528 (13) | 0.0590 (13) | −0.0075 (9) | 0.0060 (8) | −0.0092 (10) |
| C7 | 0.0246 (9) | 0.0464 (12) | 0.0555 (12) | −0.0016 (8) | 0.0099 (8) | −0.0030 (10) |
| C8 | 0.0280 (9) | 0.0422 (12) | 0.0522 (12) | 0.0038 (9) | 0.0092 (8) | 0.0033 (9) |
| C9 | 0.0405 (11) | 0.0575 (15) | 0.0545 (13) | 0.0068 (10) | 0.0147 (10) | 0.0040 (11) |
| C10 | 0.0592 (16) | 0.080 (2) | 0.0759 (18) | −0.0097 (14) | 0.0314 (14) | 0.0065 (15) |
| C11 | 0.0284 (9) | 0.0499 (13) | 0.0400 (10) | 0.0034 (9) | 0.0046 (8) | 0.0021 (9) |
| C12 | 0.0379 (11) | 0.0576 (16) | 0.0653 (14) | −0.0090 (10) | 0.0092 (10) | 0.0009 (12) |
| C13 | 0.0581 (16) | 0.0522 (15) | 0.0686 (17) | −0.0050 (12) | 0.0140 (13) | −0.0020 (12) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Cl1—C4 | 1.745 (2) | C6—H6 | 0.9300 |
| O1—C9 | 1.213 (3) | C7—C8 | 1.344 (3) |
| O2—C11 | 1.201 (2) | C7—H7 | 0.9300 |
| O3—C11 | 1.325 (3) | C8—C9 | 1.491 (3) |
| O3—C12 | 1.456 (3) | C8—C11 | 1.501 (3) |
| C1—C6 | 1.400 (3) | C9—C10 | 1.502 (4) |
| C1—C2 | 1.401 (3) | C10—H10A | 0.9600 |
| C1—C7 | 1.468 (3) | C10—H10B | 0.9600 |
| C2—C3 | 1.369 (3) | C10—H10C | 0.9600 |
| C2—H2A | 0.9300 | C12—C13 | 1.495 (4) |
| C3—C4 | 1.384 (3) | C12—H12A | 0.9700 |
| C3—H3 | 0.9300 | C12—H12B | 0.9700 |
| C4—C5 | 1.377 (3) | C13—H13A | 0.9600 |
| C5—C6 | 1.376 (3) | C13—H13B | 0.9600 |
| C5—H5 | 0.9300 | C13—H13C | 0.9600 |
| | | | |
| C11—O3—C12 | 116.98 (16) | O1—C9—C8 | 119.1 (2) |
| C6—C1—C2 | 117.2 (2) | O1—C9—C10 | 120.6 (2) |
| C6—C1—C7 | 118.26 (18) | C8—C9—C10 | 120.3 (2) |
| C2—C1—C7 | 124.50 (19) | C9—C10—H10A | 109.5 |
| C3—C2—C1 | 121.34 (19) | C9—C10—H10B | 109.5 |
| C3—C2—H2A | 119.3 | H10A—C10—H10B | 109.5 |
| C1—C2—H2A | 119.3 | C9—C10—H10C | 109.5 |
| C2—C3—C4 | 119.4 (2) | H10A—C10—H10C | 109.5 |
| C2—C3—H3 | 120.3 | H10B—C10—H10C | 109.5 |

| | | | |
|---|---|---|---|
| C4—C3—H3 | 120.3 | O2—C11—O3 | 125.2 (2) |
| C5—C4—C3 | 121.4 (2) | O2—C11—C8 | 124.1 (2) |
| C5—C4—Cl1 | 119.85 (17) | O3—C11—C8 | 110.72 (16) |
| C3—C4—Cl1 | 118.72 (18) | O3—C12—C13 | 106.71 (18) |
| C6—C5—C4 | 118.5 (2) | O3—C12—H12A | 110.4 |
| C6—C5—H5 | 120.8 | C13—C12—H12A | 110.4 |
| C4—C5—H5 | 120.8 | O3—C12—H12B | 110.4 |
| C5—C6—C1 | 122.12 (19) | C13—C12—H12B | 110.4 |
| C5—C6—H6 | 118.9 | H12A—C12—H12B | 108.6 |
| C1—C6—H6 | 118.9 | C12—C13—H13A | 109.5 |
| C8—C7—C1 | 130.0 (2) | C12—C13—H13B | 109.5 |
| C8—C7—H7 | 115.0 | H13A—C13—H13B | 109.5 |
| C1—C7—H7 | 115.0 | C12—C13—H13C | 109.5 |
| C7—C8—C9 | 123.8 (2) | H13A—C13—H13C | 109.5 |
| C7—C8—C11 | 123.3 (2) | H13B—C13—H13C | 109.5 |
| C9—C8—C11 | 112.85 (19) | | |

| | | | |
|---|---|---|---|
| C6—C1—C2—C3 | 1.7 (3) | C1—C7—C8—C11 | 1.8 (4) |
| C7—C1—C2—C3 | −179.6 (2) | C7—C8—C9—O1 | 179.1 (2) |
| C1—C2—C3—C4 | −0.2 (4) | C11—C8—C9—O1 | 0.2 (3) |
| C2—C3—C4—C5 | −1.3 (4) | C7—C8—C9—C10 | −2.4 (4) |
| C2—C3—C4—Cl1 | 179.48 (18) | C11—C8—C9—C10 | 178.7 (2) |
| C3—C4—C5—C6 | 1.2 (4) | C12—O3—C11—O2 | 1.0 (3) |
| Cl1—C4—C5—C6 | −179.59 (18) | C12—O3—C11—C8 | −178.29 (18) |
| C4—C5—C6—C1 | 0.4 (4) | C7—C8—C11—O2 | −99.3 (3) |
| C2—C1—C6—C5 | −1.8 (3) | C9—C8—C11—O2 | 79.6 (3) |
| C7—C1—C6—C5 | 179.4 (2) | C7—C8—C11—O3 | 80.1 (3) |
| C6—C1—C7—C8 | −169.6 (2) | C9—C8—C11—O3 | −101.1 (2) |
| C2—C1—C7—C8 | 11.7 (4) | C11—O3—C12—C13 | −172.43 (19) |
| C1—C7—C8—C9 | −177.0 (2) | | |