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tert-Butyl 2-(4-chlorobenzoyl)-2-methylpropanoate

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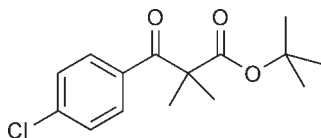
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Key indicators: single-crystal X-ray study; $T = 291$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.047; wR factor = 0.125; data-to-parameter ratio = 15.6.

The title compound, $\text{C}_{15}\text{H}_{19}\text{ClO}_3$, is bent with a dihedral angle of 72.02 (9)° between the mean planes of the benzene ring and a group encompassing the ester functionality ($\text{O}=\text{C}-\text{O}-\text{C}$). In the crystal, molecules related by inversion symmetry are connected by weak $\text{C}-\text{H}\cdots\text{O}$ interactions into infinite chains. These interactions involve H atoms from a methyl group of the dimethyl residue and the O atoms of the ketone on one side of a molecule; on the other side there are interactions between H atoms of the benzene ring and the carbonyl O atoms of the ester functionality. There are no directional interactions between the chains.

Related literature

For the synthesis, spectroscopic characterization and reactivity of the title compound, see: Logue (1974); Logue *et al.* (1975). For related structures, see: Crosse *et al.* (2010); Gould *et al.* (2010); Logue *et al.* (2010). For the syntheses and characterization of structurally similar indanone-derived β -keto ester derivatives, see: Mouri *et al.* (2009); Noritake *et al.* (2008); Rigby & Dixon (2008). For weak hydrogen-bonded interactions, see: Karle *et al.* (2009).



Experimental

Crystal data

$\text{C}_{15}\text{H}_{19}\text{ClO}_3$	$b = 9.214$ (2) Å
$M_r = 282.75$	$c = 11.033$ (2) Å
Triclinic, $P\bar{1}$	$\alpha = 72.67$ (2)°
$a = 8.601$ (3) Å	$\beta = 74.62$ (2)°

$\gamma = 74.02$ (3)°
 $V = 786.3$ (4) Å³
 $Z = 2$
 Mo $K\alpha$ radiation

$\mu = 0.24$ mm⁻¹
 $T = 291$ K
 $0.30 \times 0.30 \times 0.30$ mm

Data collection

Enraf–Nonius TurboCAD-4 diffractometer
 Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.905$, $T_{\max} = 0.929$
 2961 measured reflections

2759 independent reflections
 1589 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.020$
 3 standard reflections every 166 min
 intensity decay: 9%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.047$
 $wR(F^2) = 0.125$
 $S = 1.01$
 2759 reflections

177 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.16$ e Å⁻³
 $\Delta\rho_{\min} = -0.17$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C10}-\text{H10A}\cdots\text{O1}^i$	0.96	2.58	3.476 (4)	155
$\text{C5}-\text{H5}\cdots\text{O2}^{ii}$	0.93	2.7	3.316 (3)	125

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

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *pubCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZL2267).

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supplementary materials

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***tert*-Butyl 2-(4-chlorobenzoyl)-2-methylpropanoate**

C. M. Crosse, E. C. Kelly, M. W. Logue, R. L. Luck, J. S. Maass, K. C. Mehne and L. R. Pignotti

Comment

Treatment of 2,2-disubstituted *t*-butyl β -keto esters with trifluoroacetic acid at room temperature quantitatively generates the corresponding 2,2-disubstituted β -keto acids, which were used to probe the nature of the transition state for the thermal decarboxylation of β -keto acids (Logue *et al.*, 1975). Structurally similar indanone-derived β -keto ester derivatives have been prepared recently (Mouri *et al.*, 2009; Noritake *et al.*, 2008; Rigby & Dixon, 2008). The directing nature of weak C—H \cdots O H-bonds has been noted to be of importance to afford the three dimensional structure observed in these kinds of molecules (Karle *et al.*, 2009).

In this contribution we present the solid state structure of one such 2,2-disubstituted β -keto acid, i.e. the title compound being the *para*-chlorobenzene derivative. This is the third paper in a series of four dealing with substituted derivatives (H-, CH₃-, Cl- (this paper) and NO₂- on the *para*-position of the phenyl ring) of the title compound. A more detailed comparison of all four substitution compounds will be given in the fourth paper of this series (Crosse *et al.*, 2010).

The molecule, Fig. 1, displays a bent geometry with a dihedral angle between the mean planes of the phenyl ring and a plane composed of the ester functionality of 72.02 (9)°. Molecules are linked by C—H \cdots O weak hydrogen bonds generating infinite chains as shown in Fig. 2. The phenyl rings are not involved in intercalation or stacking interactions either within or between the chains. Instead, neighbouring *t*-butyl groups on adjacent chains exhibit hydrophobic stacking.

Experimental

Crystals of the material were synthesized as reported earlier and were grown by evaporation of a solution in hexane (Logue, 1974). *M.p.* 600–606 K. IR (neat, cm⁻¹) 2982 (m, ring), 1726 (s, C=O), 1682 (s, O—C=O), 1588 (*s*), 1488 (*m*), 1388 (*m*), 1368 (*w*), 1273 (*m*), 1133 (*m*), 1091.6 (*m*), 987 (*w*), 920 (*w*), 842 (*m*), 739 (*m*). ¹H NMR (CDCl₃) δ : 1.27 (*s*, 9H), 1.47 (*s*, 6H), 7.36 (*d*, 2H, *J*=8.8 Hz), 7.80 (*d*, 2H, *J*=9.2 Hz). ¹³C NMR (CDCl₃) δ : 23.8, 27.6, 54.0, 81.9, 130.2, 133.6, 139.0, 173.8, 196.8.

Refinement

All H atoms were placed at calculated positions, with C—H = 0.93 Å (aromatic) or 0.96 Å (methyl) and refined using a riding model with *U*_{iso}(H) constrained to be 1.5 *U*_{eq}(C) for methyl groups and 1.2 *U*_{eq}(C) for all other C atoms. The quality of the data as reflected by only 58% of the reflections observed, large ADP's and inaccurate C—C bond lengths is low. The data had been collected on a 30 year old single point detector instrument not equipped with a low temperature device as part of a class project with undergraduate students. Due to the time constraints imposed by the class schedule a maximum exposure time of 60 s had to be allotted for measuring each reflection.

Figures

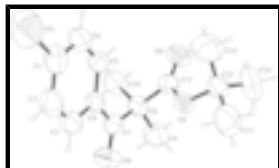


Fig. 1. ORTEP-3 (Farrugia, 1997) drawing of the title compound with displacement ellipsoids drawn at the 50% probability level.

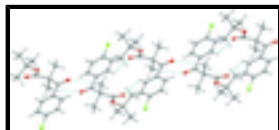


Fig. 2. A Mercury (Macrae *et al.*, 2008) illustration of the packing in the title compound depicting the H-bonded linkages using dashed lines which result in infinite chains.

tert-Butyl 2-(4-chlorobenzoyl)-2-methylpropanoate

Crystal data

$C_{15}H_{19}ClO_3$	$Z = 2$
$M_r = 282.75$	$F(000) = 300$
Triclinic, $P\bar{1}$	$D_x = 1.194 \text{ Mg m}^{-3}$
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
$a = 8.601 (3) \text{ \AA}$	Cell parameters from 25 reflections
$b = 9.214 (2) \text{ \AA}$	$\theta = 10\text{--}15^\circ$
$c = 11.033 (2) \text{ \AA}$	$\mu = 0.24 \text{ mm}^{-1}$
$\alpha = 72.67 (2)^\circ$	$T = 291 \text{ K}$
$\beta = 74.62 (2)^\circ$	Prism, colourless
$\gamma = 74.02 (3)^\circ$	$0.30 \times 0.30 \times 0.30 \text{ mm}$
$V = 786.3 (4) \text{ \AA}^3$	

Data collection

Enraf–Nonius TurboCAD-4 diffractometer	1589 reflections with $I > 2\sigma(I)$
Radiation source: Enraf Nonius FR590 graphite	$R_{\text{int}} = 0.020$
non-profiled $\omega/2\tau$ scans	$\theta_{\text{max}} = 25.0^\circ$, $\theta_{\text{min}} = 2.0^\circ$
Absorption correction: ψ scan (North <i>et al.</i> , 1968)	$h = 0 \rightarrow 10$
$T_{\text{min}} = 0.905$, $T_{\text{max}} = 0.929$	$k = -10 \rightarrow 10$
2961 measured reflections	$l = -12 \rightarrow 13$
2759 independent reflections	3 standard reflections every 166 min
	intensity decay: 9%

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.047$	Hydrogen site location: inferred from neighbouring sites

$wR(F^2) = 0.125$	H-atom parameters constrained
$S = 1.00$	$w = 1/[\sigma^2(F_o^2) + (0.0499P)^2 + 0.1997P]$
2759 reflections	where $P = (F_o^2 + 2F_c^2)/3$
177 parameters	$(\Delta/\sigma)_{\max} < 0.001$
0 restraints	$\Delta\rho_{\max} = 0.16 \text{ e } \text{\AA}^{-3}$
	$\Delta\rho_{\min} = -0.17 \text{ e } \text{\AA}^{-3}$

Special details

Experimental. Number of psi-scan sets used was 3. Theta correction was applied. Averaged transmission function was used. No Fourier smoothing was applied.

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	0.16763 (12)	0.07087 (11)	0.34258 (10)	0.1148 (4)
C1	0.0088 (3)	0.2115 (3)	0.2795 (3)	0.0715 (8)
C2	0.0200 (4)	0.2567 (3)	0.1470 (3)	0.0732 (8)
H2	0.112	0.2139	0.0922	0.088*
C3	-0.1066 (3)	0.3656 (3)	0.0975 (3)	0.0652 (7)
H3	-0.0996	0.3952	0.0083	0.078*
C4	-0.2453 (3)	0.4331 (3)	0.1768 (2)	0.0539 (6)
C5	-0.2524 (3)	0.3851 (3)	0.3101 (2)	0.0616 (7)
H5	-0.3433	0.4288	0.3654	0.074*
C6	-0.1270 (4)	0.2740 (3)	0.3615 (3)	0.0733 (8)
H6	-0.1341	0.2417	0.4507	0.088*
C7	-0.3731 (3)	0.5568 (3)	0.1130 (2)	0.0567 (6)
O1	-0.3574 (2)	0.5870 (2)	-0.00407 (18)	0.0818 (6)
C8	-0.5234 (3)	0.6450 (3)	0.1932 (2)	0.0580 (7)
C9	-0.6140 (4)	0.7842 (4)	0.1003 (3)	0.0858 (9)
H9A	-0.6522	0.7464	0.0436	0.129*
H9B	-0.5396	0.8509	0.0499	0.129*
H9C	-0.7065	0.8416	0.1499	0.129*
C10	-0.6413 (3)	0.5359 (4)	0.2725 (3)	0.0815 (9)
H10A	-0.6754	0.4959	0.215	0.122*
H10B	-0.7364	0.5926	0.3205	0.122*
H10C	-0.5855	0.451	0.3316	0.122*
C11	-0.4693 (3)	0.7139 (3)	0.2811 (3)	0.0557 (6)

supplementary materials

O2	-0.5376 (2)	0.7174 (2)	0.39021 (18)	0.0768 (6)
O3	-0.3370 (2)	0.77357 (18)	0.21611 (15)	0.0573 (5)
C12	-0.2559 (3)	0.8503 (3)	0.2752 (3)	0.0652 (7)
C13	-0.1158 (4)	0.8952 (4)	0.1653 (3)	0.0982 (11)
H13A	-0.1601	0.9655	0.0928	0.147*
H13B	-0.0465	0.8035	0.14	0.147*
H13C	-0.052	0.945	0.1934	0.147*
C14	-0.3741 (4)	0.9938 (4)	0.3095 (4)	0.1056 (12)
H14A	-0.4215	1.0563	0.2361	0.158*
H14B	-0.3158	1.053	0.3331	0.158*
H14C	-0.4602	0.9636	0.381	0.158*
C15	-0.1904 (5)	0.7350 (4)	0.3885 (3)	0.1139 (13)
H15A	-0.2809	0.7124	0.4587	0.171*
H15B	-0.1196	0.7781	0.4159	0.171*
H15C	-0.129	0.6409	0.3633	0.171*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0975 (7)	0.1088 (7)	0.1345 (8)	0.0243 (5)	-0.0444 (6)	-0.0478 (6)
C1	0.0677 (18)	0.0628 (18)	0.088 (2)	-0.0045 (14)	-0.0202 (16)	-0.0288 (16)
C2	0.0654 (18)	0.0720 (19)	0.080 (2)	-0.0162 (16)	0.0064 (16)	-0.0313 (16)
C3	0.0745 (19)	0.0649 (17)	0.0575 (16)	-0.0239 (15)	0.0015 (15)	-0.0214 (14)
C4	0.0614 (15)	0.0556 (15)	0.0498 (14)	-0.0241 (13)	-0.0005 (12)	-0.0191 (12)
C5	0.0671 (17)	0.0592 (16)	0.0559 (16)	-0.0098 (14)	-0.0034 (13)	-0.0213 (13)
C6	0.084 (2)	0.0683 (18)	0.0657 (17)	-0.0017 (16)	-0.0181 (16)	-0.0243 (15)
C7	0.0618 (17)	0.0631 (16)	0.0541 (16)	-0.0303 (14)	-0.0073 (13)	-0.0156 (13)
O1	0.0877 (14)	0.1077 (16)	0.0519 (12)	-0.0260 (12)	-0.0120 (10)	-0.0194 (11)
C8	0.0535 (15)	0.0655 (16)	0.0594 (15)	-0.0163 (13)	-0.0123 (12)	-0.0171 (13)
C9	0.081 (2)	0.093 (2)	0.090 (2)	-0.0075 (17)	-0.0387 (18)	-0.0230 (18)
C10	0.0607 (18)	0.104 (2)	0.090 (2)	-0.0384 (17)	0.0024 (15)	-0.0343 (18)
C11	0.0538 (15)	0.0572 (16)	0.0549 (16)	-0.0106 (13)	-0.0100 (13)	-0.0139 (12)
O2	0.0796 (13)	0.0971 (15)	0.0575 (12)	-0.0311 (11)	0.0045 (10)	-0.0289 (10)
O3	0.0587 (10)	0.0672 (11)	0.0543 (10)	-0.0232 (9)	-0.0074 (8)	-0.0217 (8)
C12	0.0684 (17)	0.0764 (18)	0.0660 (17)	-0.0271 (15)	-0.0107 (14)	-0.0314 (15)
C13	0.090 (2)	0.122 (3)	0.105 (3)	-0.058 (2)	0.0055 (19)	-0.049 (2)
C14	0.096 (3)	0.102 (3)	0.145 (3)	-0.026 (2)	-0.008 (2)	-0.077 (2)
C15	0.131 (3)	0.137 (3)	0.100 (3)	-0.054 (3)	-0.063 (2)	-0.009 (2)

Geometric parameters (\AA , $^\circ$)

C11—C1	1.739 (3)	C9—H9C	0.96
C1—C6	1.376 (4)	C10—H10A	0.96
C1—C2	1.380 (4)	C10—H10B	0.96
C2—C3	1.370 (4)	C10—H10C	0.96
C2—H2	0.93	C11—O2	1.197 (3)
C3—C4	1.392 (3)	C11—O3	1.337 (3)
C3—H3	0.93	O3—C12	1.483 (3)
C4—C5	1.394 (3)	C12—C15	1.504 (4)

C4—C7	1.498 (4)	C12—C14	1.509 (4)
C5—C6	1.380 (4)	C12—C13	1.515 (4)
C5—H5	0.93	C13—H13A	0.96
C6—H6	0.93	C13—H13B	0.96
C7—O1	1.216 (3)	C13—H13C	0.96
C7—C8	1.536 (3)	C14—H14A	0.96
C8—C11	1.526 (3)	C14—H14B	0.96
C8—C10	1.540 (4)	C14—H14C	0.96
C8—C9	1.546 (4)	C15—H15A	0.96
C9—H9A	0.96	C15—H15B	0.96
C9—H9B	0.96	C15—H15C	0.96
C6—C1—C2	120.9 (3)	C8—C10—H10B	109.5
C6—C1—C11	120.0 (2)	H10A—C10—H10B	109.5
C2—C1—C11	119.0 (2)	C8—C10—H10C	109.5
C3—C2—C1	119.0 (3)	H10A—C10—H10C	109.5
C3—C2—H2	120.5	H10B—C10—H10C	109.5
C1—C2—H2	120.5	O2—C11—O3	125.4 (2)
C2—C3—C4	122.0 (3)	O2—C11—C8	125.1 (2)
C2—C3—H3	119	O3—C11—C8	109.5 (2)
C4—C3—H3	119	C11—O3—C12	122.39 (19)
C3—C4—C5	117.5 (3)	O3—C12—C15	109.6 (2)
C3—C4—C7	117.9 (2)	O3—C12—C14	109.8 (2)
C5—C4—C7	124.5 (2)	C15—C12—C14	113.7 (3)
C6—C5—C4	121.1 (2)	O3—C12—C13	101.9 (2)
C6—C5—H5	119.4	C15—C12—C13	110.8 (3)
C4—C5—H5	119.4	C14—C12—C13	110.4 (3)
C1—C6—C5	119.4 (3)	C12—C13—H13A	109.5
C1—C6—H6	120.3	C12—C13—H13B	109.5
C5—C6—H6	120.3	H13A—C13—H13B	109.5
O1—C7—C4	119.2 (2)	C12—C13—H13C	109.5
O1—C7—C8	119.7 (2)	H13A—C13—H13C	109.5
C4—C7—C8	121.1 (2)	H13B—C13—H13C	109.5
C11—C8—C7	110.7 (2)	C12—C14—H14A	109.5
C11—C8—C10	111.1 (2)	C12—C14—H14B	109.5
C7—C8—C10	110.0 (2)	H14A—C14—H14B	109.5
C11—C8—C9	106.4 (2)	C12—C14—H14C	109.5
C7—C8—C9	109.0 (2)	H14A—C14—H14C	109.5
C10—C8—C9	109.7 (2)	H14B—C14—H14C	109.5
C8—C9—H9A	109.5	C12—C15—H15A	109.5
C8—C9—H9B	109.5	C12—C15—H15B	109.5
H9A—C9—H9B	109.5	H15A—C15—H15B	109.5
C8—C9—H9C	109.5	C12—C15—H15C	109.5
H9A—C9—H9C	109.5	H15A—C15—H15C	109.5
H9B—C9—H9C	109.5	H15B—C15—H15C	109.5
C8—C10—H10A	109.5		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
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supplementary materials

C10—H10A···O1 ⁱ	0.96	2.58	3.476 (4)	155
C5—H5···O2 ⁱⁱ	0.93	2.7	3.316 (3)	125

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

Fig. 1

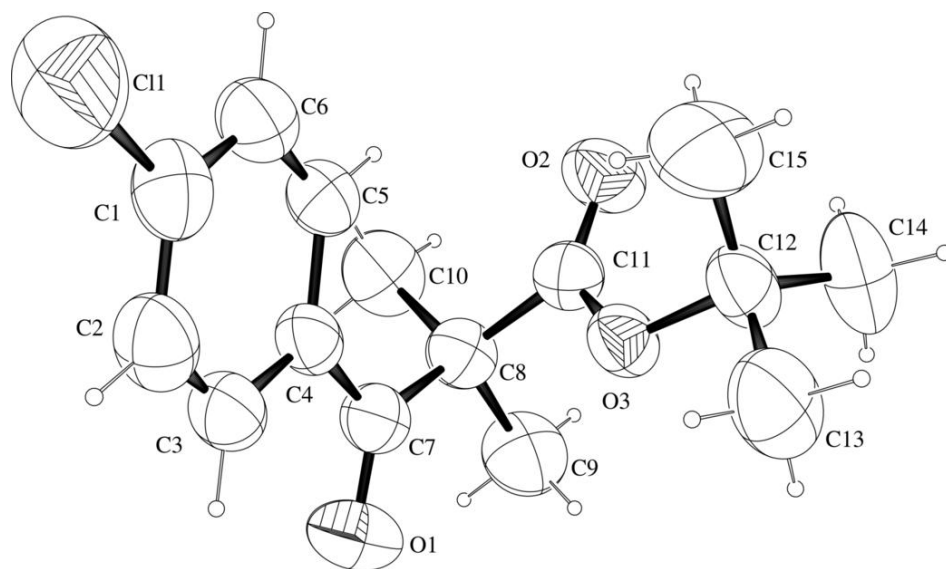


Fig. 2

