

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

3-(4-Chlorophenyl)-2-methylacrylic acid

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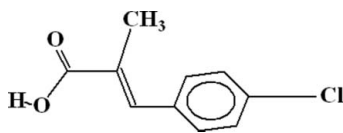
Received 5 July 2008; accepted 15 July 2008

Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.060; wR factor = 0.218; data-to-parameter ratio = 22.1.

In the crystal structure of the title compound, $\text{C}_{10}\text{H}_9\text{ClO}_2$, dimers form as a result of intermolecular $\text{O}-\text{H}\cdots\text{O}$ bonding. These dimers are linked to each other *via* $\text{C}-\text{H}\cdots\text{O}$ bonds, where the CH group belongs to the benzene ring and the O atom is from the carbonyl group of an adjacent molecule. There exist two intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds, which individually form five-membered rings. There also exists a $\pi-\pi$ interaction between the aromatic ring and its symmetry counterpart, with a centroid-centroid distance of 4.0202 (17) Å, and a $\text{C}-\text{H}\cdots\pi$ interaction between a methyl CH group and the aromatic ring.

Related literature

For related literature, see: Bernstein *et al.* (1995); Bravo (1998); Burt (2004); Hertog *et al.* (1995); Muhammad *et al.* (2007*a,b*, 2008*a,b*); Muhammad, Ali *et al.* (2008); Niaz *et al.* (2008).



Experimental

Crystal data

$\text{C}_{10}\text{H}_9\text{ClO}_2$
 $M_r = 196.62$
 Triclinic, $P\bar{1}$
 $a = 7.2164$ (6) Å
 $b = 8.2746$ (7) Å
 $c = 9.1762$ (8) Å
 $\alpha = 115.182$ (4)°
 $\beta = 108.022$ (4)°

$\gamma = 90.052$ (5)°
 $V = 465.91$ (7) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.37$ mm⁻¹
 $T = 296$ (2) K
 $0.28 \times 0.20 \times 0.18$ mm

Data collection

Bruker Kappa APEXII CCD
 diffractometer

Absorption correction: multi-scan
 (SADABS; Bruker, 2005)
 $T_{\min} = 0.910$, $T_{\max} = 0.930$

7513 measured reflections
 2692 independent reflections

1782 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.029$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.059$
 $wR(F^2) = 0.217$
 $S = 1.10$
 2692 reflections
 122 parameters

H atoms treated by a mixture of
 independent and constrained
 refinement
 $\Delta\rho_{\text{max}} = 0.53$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.27$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O1}-\text{H1}\cdots\text{O2}^{\text{i}}$	0.88 (4)	1.76 (4)	2.643 (3)	176.4 (14)
$\text{C3}-\text{H3A}\cdots\text{O2}$	0.96	2.41	2.765 (4)	101
$\text{C4}-\text{H4}\cdots\text{O1}$	0.93	2.32	2.720 (3)	106
$\text{C9}-\text{H9}\cdots\text{O2}^{\text{ii}}$	0.93	2.57	3.458 (3)	159
$\text{C3}-\text{H3a}\cdots\text{Cg}^{\text{iii}}$	0.96	2.84	3.638 (3)	141

Symmetry codes: (i) $-x - 1, -y + 1, -z$; (ii) $x + 1, y, z + 1$; (iii) $-x, -y, -z$. Cg is the centroid of the C5-C10 ring.

Data collection: APEX2 (Bruker, 2007); cell refinement: APEX2; data reduction: SAINT (Bruker, 2007); program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997) and PLATON (Spek, 2003); software used to prepare material for publication: WinGX (Farrugia, 1999) and PLATON.

The authors acknowledge the Higher Education Commission, Islamabad, Pakistan, for funding the purchase of the diffractometer at GCU, Lahore, and for financial support to NM for a PhD under the Indigenous Scholarship Scheme.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BQ2090).

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supporting information

Acta Cryst. (2008). E64, o1542 [doi:10.1107/S1600536808022198]

3-(4-Chlorophenyl)-2-methylacrylic acid

Niaz Muhammad, Muhammad Nawaz Tahir, Zia-ur-Rehman, Saqib Ali and Farkhanda Shaheen

S1. Comment

Cinnamic acids compose a relatively large family of organic acid isomers (Bravo, 1998). In nature, cinnamic acid derivatives are important metabolic building blocks in the production of lignins for higher plants. Cinnamic acid possesses antibacterial, antifungal and parasite fighting abilities (Burt, 2004). A derivative of cinnamic acid is an important pharmaceutical for high blood pressure, stroke prevention and possess antitumour activity (Hertog *et al.*, 1995). In continuation of our efforts to synthesize various derivatives of cinnamic acids (Niaz *et al.*, 2008, Muhammad, Ali *et al.*, 2008) and their tin complexes (Muhammad *et al.*, 2008a, 2008b), we herein report the structure of the title compound (I).

The crystal structure of 3-(4-Bromophenyl)-2-methylacrylic acid (II) (Muhammad *et al.*, 2007a) and 3-(4-Bromophenyl)-2-ethylacrylic acid (Muhammad *et al.*, 2007b) has been previously reported. The title compound (I) have a replacement of Br-atom with Cl-atom. Thus the reported compound (II) is the best example for the comparison of bond geometry *etc.*

In the crystal structure of the title compound, the C—C bonds are in the range [1.467 (3)–1.503 (4) Å], and C=C have a value of 1.341 (3) Å. The resonant C—O bonds have values of 1.231 (3) and 1.310 (3) Å. In the asymmetric unit, there are two intermolecular H-bonds of C—H \cdots O type (Table 2, Fig 1). Due to these H-bonds two five membered rings (O1/C1/C2/C4/H4 \cdots O1) and (O2/C1/C2/C3/H3A \cdots O2) are formed. Centrosymmetric dimers, $R_2^2(8)$ (Bernstein *et al.* 1995) are formed due to the intermolecular O1—H1 \cdots O2ⁱ [symmetry code: $i = -x - 1, -y + 1, -z$] hydrogen bonding. These dimers are linked to each other by intermolecular H-bonding, C9—H9 \cdots O2ⁱⁱ [symmetry code: $ii = x + 1, y, z + 1$] as shown in Fig 2. There exist an interaction, C3—H3A \cdots Cgⁱⁱⁱ [symmetry code: $iii = -x, -y, -z$] with a distance of 3.638 (3) Å between C3 and Cgⁱⁱⁱ [Cg is the center of the (C5-C10) benzene ring]. There also exist a $\pi\cdots\pi$ -interaction between the benzene rings of adjacent molecules. The distance between the centroids of Cg and Cg^{iv} [symmetry code: $iv = -x + 1, -y + 1, -z + 1$], is 4.0202 (17) Å.

S2. Experimental

Compound (I) was prepared according to the reported procedure (Muhammad *et al.*, 2007a). A mixture of 4-chlorobenzaldehyde (1.40 g, 10 mmol), methylmalonic acid (2.36 g, 20 mmol) and piperidine (1.98 ml, 20 mmol) in a pyridine (12.5 ml) solution was heated on a steam-bath for 24 h. The reaction mixture was cooled and added to a mixture of 25 ml of concentrated HCl and 50 g of ice. The precipitate formed in the acidified mixture was filtered off and washed with ice-cold water. The product was recrystallized from ethanol. The yield was 89%.

S3. Refinement

The coordinates of H atom attached to O1 were refined freely. All other H atoms were positioned geometrically, C—H = 0.93, and 0.96 Å for aromatic and methyl H, and constrained to ride on their parent atoms and were treated as isotropic

with $U_{\text{iso}}(\text{H}) = xU_{\text{eq}}(\text{C}, \text{O})$, where $x = 1.5$ for methyl H, and $x = 1.2$ for all other H atoms.

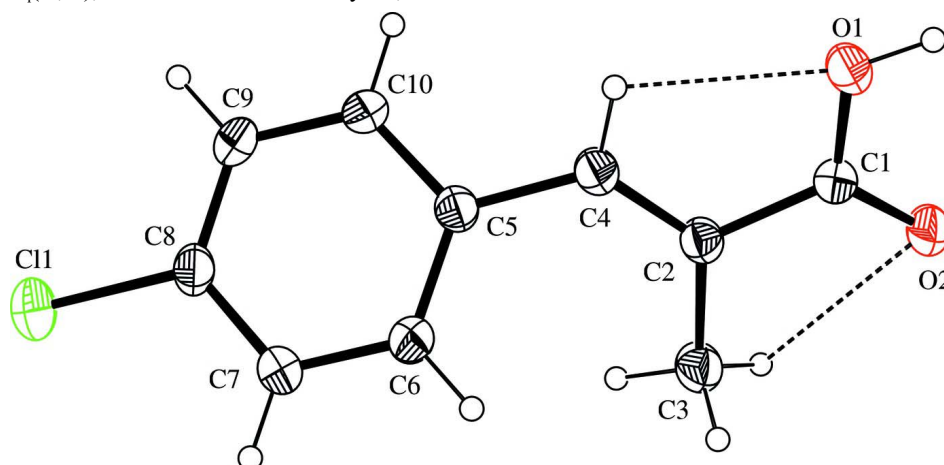


Figure 1

ORTEP drawing of (I) with the atom numbering scheme. The thermal ellipsoids are drawn at the 50% probability level. The intramolecular H-bonds are shown by dotted lines.

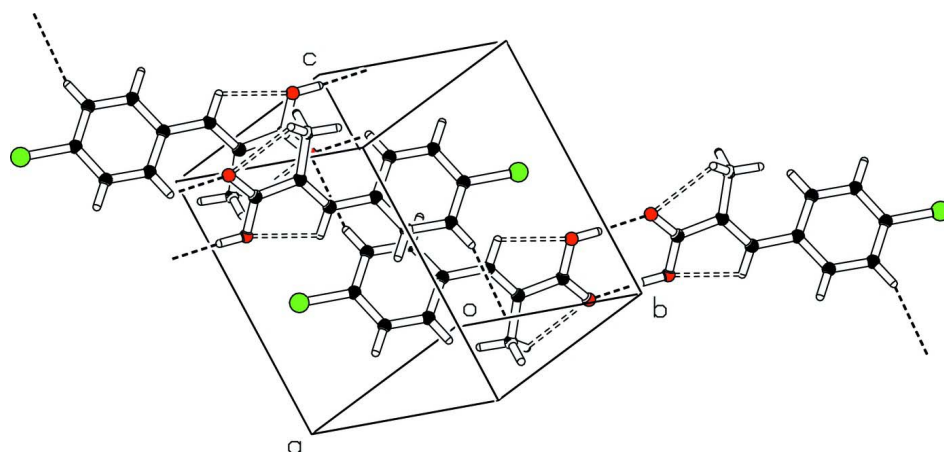


Figure 2

The packing figure (*PLATON*: Spek, 2003) which shows the dimeric nature of the compound and the interlinkages of the dimers.

3-(4-Chlorophenyl)-2-methylacrylic acid

Crystal data

$\text{C}_{10}\text{H}_9\text{ClO}_2$

$M_r = 196.62$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 7.2164$ (6) Å

$b = 8.2746$ (7) Å

$c = 9.1762$ (8) Å

$\alpha = 115.182$ (4)°

$\beta = 108.022$ (4)°

$\gamma = 90.052$ (5)°

$V = 465.91$ (7) Å³

$Z = 2$

$F(000) = 204$

$D_x = 1.402$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2692 reflections

$\theta = 2.6\text{--}30.3^\circ$

$\mu = 0.37$ mm⁻¹

$T = 296$ K

Prism, colourless

$0.28 \times 0.20 \times 0.18$ mm

Data collection

Bruker Kappa APEXII CCD
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 Detector resolution: 7.2 pixels mm⁻¹
 ω scans
 Absorption correction: multi-scan
 (SADABS; Bruker, 2005)
 $T_{\min} = 0.910$, $T_{\max} = 0.930$

7513 measured reflections
 2692 independent reflections
 1782 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.029$
 $\theta_{\max} = 30.3^\circ$, $\theta_{\min} = 2.6^\circ$
 $h = -10 \rightarrow 8$
 $k = -10 \rightarrow 11$
 $l = -12 \rightarrow 12$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.059$
 $wR(F^2) = 0.217$
 $S = 1.10$
 2692 reflections
 122 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 atoms treated by a mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.1083P)^2 + 0.1931P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.53 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.27 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. Bond distances, angles *etc.* have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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 $F^2 > \sigma(F^2)$ 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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C11	0.80795 (12)	0.09608 (15)	0.42599 (12)	0.0771 (4)
O1	-0.2602 (3)	0.5461 (3)	0.1669 (2)	0.0510 (6)
O2	-0.3959 (3)	0.3388 (3)	-0.1034 (2)	0.0543 (6)
C1	-0.2554 (3)	0.4027 (3)	0.0322 (3)	0.0390 (7)
C2	-0.0735 (3)	0.3196 (3)	0.0525 (3)	0.0371 (7)
C3	-0.0680 (4)	0.1670 (4)	-0.1102 (3)	0.0484 (8)
C4	0.0606 (3)	0.3764 (3)	0.2098 (3)	0.0403 (7)
C5	0.2464 (3)	0.3096 (3)	0.2602 (3)	0.0364 (6)
C6	0.3638 (4)	0.2508 (4)	0.1573 (3)	0.0432 (8)
C7	0.5363 (4)	0.1864 (4)	0.2086 (3)	0.0461 (8)
C8	0.5923 (4)	0.1793 (4)	0.3641 (3)	0.0438 (7)
C9	0.4803 (4)	0.2379 (4)	0.4686 (3)	0.0493 (8)
C10	0.3099 (4)	0.3051 (4)	0.4171 (3)	0.0451 (7)
H1	-0.377 (5)	0.580 (5)	0.142 (5)	0.0612*
H3A	-0.19861	0.10124	-0.17914	0.0725*
H3B	0.01747	0.08750	-0.08293	0.0725*

H3C	-0.01950	0.21487	-0.17241	0.0725*
H4	0.03229	0.46997	0.29797	0.0484*
H6	0.32524	0.25512	0.05263	0.0518*
H7	0.61382	0.14812	0.13947	0.0554*
H9	0.51873	0.23233	0.57269	0.0592*
H10	0.23624	0.34816	0.48924	0.0541*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0555 (5)	0.1129 (8)	0.0698 (6)	0.0449 (5)	0.0181 (4)	0.0492 (5)
O1	0.0415 (10)	0.0576 (12)	0.0430 (10)	0.0207 (8)	0.0116 (8)	0.0147 (9)
O2	0.0436 (10)	0.0686 (13)	0.0391 (10)	0.0217 (9)	0.0075 (8)	0.0183 (9)
C1	0.0377 (11)	0.0459 (13)	0.0373 (12)	0.0117 (10)	0.0140 (9)	0.0213 (10)
C2	0.0357 (11)	0.0379 (12)	0.0411 (12)	0.0091 (9)	0.0141 (9)	0.0202 (10)
C3	0.0444 (13)	0.0477 (14)	0.0430 (13)	0.0116 (11)	0.0115 (11)	0.0138 (11)
C4	0.0365 (11)	0.0418 (12)	0.0395 (12)	0.0109 (9)	0.0122 (9)	0.0160 (10)
C5	0.0335 (10)	0.0351 (11)	0.0363 (11)	0.0060 (9)	0.0105 (9)	0.0131 (9)
C6	0.0397 (12)	0.0562 (15)	0.0408 (12)	0.0112 (10)	0.0143 (10)	0.0276 (12)
C7	0.0359 (11)	0.0604 (16)	0.0443 (13)	0.0130 (11)	0.0152 (10)	0.0245 (12)
C8	0.0349 (11)	0.0487 (14)	0.0413 (12)	0.0103 (10)	0.0060 (10)	0.0192 (11)
C9	0.0461 (13)	0.0627 (17)	0.0323 (12)	0.0133 (12)	0.0063 (10)	0.0200 (12)
C10	0.0412 (12)	0.0563 (15)	0.0310 (11)	0.0105 (11)	0.0116 (10)	0.0141 (11)

Geometric parameters (Å, °)

C11—C8	1.734 (3)	C7—C8	1.385 (4)
O1—C1	1.310 (3)	C8—C9	1.376 (4)
O2—C1	1.231 (3)	C9—C10	1.382 (4)
O1—H1	0.88 (4)	C3—H3A	0.9600
C1—C2	1.480 (3)	C3—H3B	0.9600
C2—C3	1.503 (4)	C3—H3C	0.9600
C2—C4	1.341 (3)	C4—H4	0.9300
C4—C5	1.467 (3)	C6—H6	0.9300
C5—C10	1.386 (4)	C7—H7	0.9300
C5—C6	1.397 (4)	C9—H9	0.9300
C6—C7	1.381 (4)	C10—H10	0.9300
C1—O1—H1	109 (3)	C5—C10—C9	121.4 (2)
O1—C1—O2	121.8 (2)	C2—C3—H3A	109.00
O1—C1—C2	116.5 (2)	C2—C3—H3B	109.00
O2—C1—C2	121.7 (2)	C2—C3—H3C	109.00
C1—C2—C4	118.9 (2)	H3A—C3—H3B	109.00
C3—C2—C4	126.8 (2)	H3A—C3—H3C	109.00
C1—C2—C3	114.2 (2)	H3B—C3—H3C	110.00
C2—C4—C5	128.1 (2)	C2—C4—H4	116.00
C4—C5—C10	118.9 (2)	C5—C4—H4	116.00
C6—C5—C10	118.1 (2)	C5—C6—H6	119.00

C4—C5—C6	122.9 (2)	C7—C6—H6	119.00
C5—C6—C7	121.0 (2)	C6—C7—H7	120.00
C6—C7—C8	119.2 (3)	C8—C7—H7	120.00
C11—C8—C7	118.7 (2)	C8—C9—H9	120.00
C11—C8—C9	120.3 (2)	C10—C9—H9	120.00
C7—C8—C9	120.9 (3)	C5—C10—H10	119.00
C8—C9—C10	119.2 (3)	C9—C10—H10	119.00
O1—C1—C2—C3	-174.4 (2)	C10—C5—C6—C7	1.3 (5)
O1—C1—C2—C4	9.9 (4)	C4—C5—C10—C9	177.8 (3)
O2—C1—C2—C3	6.7 (4)	C6—C5—C10—C9	-2.5 (5)
O2—C1—C2—C4	-169.1 (3)	C5—C6—C7—C8	0.4 (5)
C1—C2—C4—C5	177.8 (3)	C6—C7—C8—C11	179.4 (3)
C3—C2—C4—C5	2.6 (5)	C6—C7—C8—C9	-1.0 (5)
C2—C4—C5—C6	35.4 (4)	C11—C8—C9—C10	179.5 (3)
C2—C4—C5—C10	-145.0 (3)	C7—C8—C9—C10	-0.2 (5)
C4—C5—C6—C7	-179.0 (3)	C8—C9—C10—C5	2.0 (5)

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>
O1—H1...O2 ⁱ	0.88 (4)	1.76 (4)	2.643 (3)	176.4 (14)
C3—H3 <i>A</i> ...O2	0.96	2.41	2.765 (4)	101
C4—H4...O1	0.93	2.32	2.720 (3)	106
C9—H9...O2 ⁱⁱ	0.93	2.57	3.458 (3)	159
C3—H3 <i>a</i> ...C <i>g</i> ⁱⁱⁱ	0.96	2.84	3.638 (3)	141

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