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2-(3-Bromo-4-ethylphenyl)-2-methylpropanoic acid

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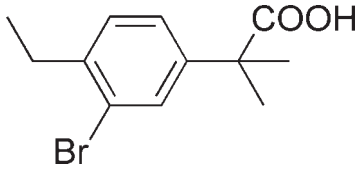
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.008 Å; R factor = 0.065; wR factor = 0.157; data-to-parameter ratio = 16.5.

In the title compound, $C_{12}H_{15}BrO_2$, the carboxyl group forms a dihedral angle of 78.4 (3)° with the benzene ring plane. In the crystal, molecules are linked into centrosymmetric dimers by pairs of O—H···O hydrogen bonds.

Related literature

For the preparation of pharmaceuticals and active agrochemical ingredients using 2-(3-bromo-4-ethylphenyl)-2-methylpropanoic acid, see: Wiegand *et al.* (2007). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{12}H_{15}BrO_2$
M_r = 271.15
Monoclinic, $P2_1/n$
a = 9.7370 (19) Å
b = 7.2930 (15) Å
c = 17.433 (4) Å
β = 90.98 (3)°
V = 1237.8 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 3.30 mm^{-1}
T = 298 K
0.20 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.558, T_{max} = 0.734
2389 measured reflections
2246 independent reflections
1171 reflections with $I > 2\sigma(I)$
R_{int} = 0.051
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.157
S = 1.00
2246 reflections
136 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.56 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1D···O2^{i}	0.82	1.88	2.696 (6)	178

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1985); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); 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: CI2903).

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supporting information

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2-(3-Bromo-4-ethylphenyl)-2-methylpropanoic acid

Rong Sun, Xiao-Yan Yang and Cheng Yao

S1. Comment

2-(3-Bromo-4-ethylphenyl)-2-methylpropanoic acid is one of the valuable intermediates for the preparation of pharmaceuticals and active agrochemical ingredients (Wiegand *et al.*, 2007). We report here the crystal structure of the title compound.

Bond lengths (Allen *et al.*, 1987) and angles in the title molecule (Fig.1) are within normal ranges. The plane of the carboxyl group forms a dihedral angle of 78.4 (3)° with the benzene plane.

In the crystal, molecules are linked into centrosymmetric dimers by pairs of O—H···O hydrogen bonds (Fig. 2).

S2. Experimental

The title compound was prepared by the hydrolyzation of methyl 2-(3-bromo-4-ethylphenyl)-2-methylpropanoate (10.42 g, 0.037 mol) in a solution of methanol (30 ml) and acetone (150 ml), catalyzed by KOH aqueous solution (73 ml, 1.0 mol/l) at room temperature (298 k). After stirring for 8 h, methanol and acetone were removed by reduced distillation to obtain an aqueous substrate. The substrate was washed with dichloromethane (4× 20 ml), and precipitated with concentrated hydrochloric acid. Then the precipitate was washed with water, collected and dried to give 2-(3-bromo-4-ethylphenyl)-2-methylpropanoic acid (4.07 g, 0.015 mol) with a yield of 41.0%. Single crystals of the compound were obtained by slow evaporation of an methanol solution at room temperature.

S3. Refinement

H atoms were positioned geometrically, with O-H = 0.82 Å and C-H = 0.93 and 0.96 Å for aromatic and methyl H, respectively, and constrained to ride on their parent atoms, with U_{iso}(H) = xU_{eq}(C,O), where x = 1.2 for aromatic H and x = 1.5 for other H atoms.

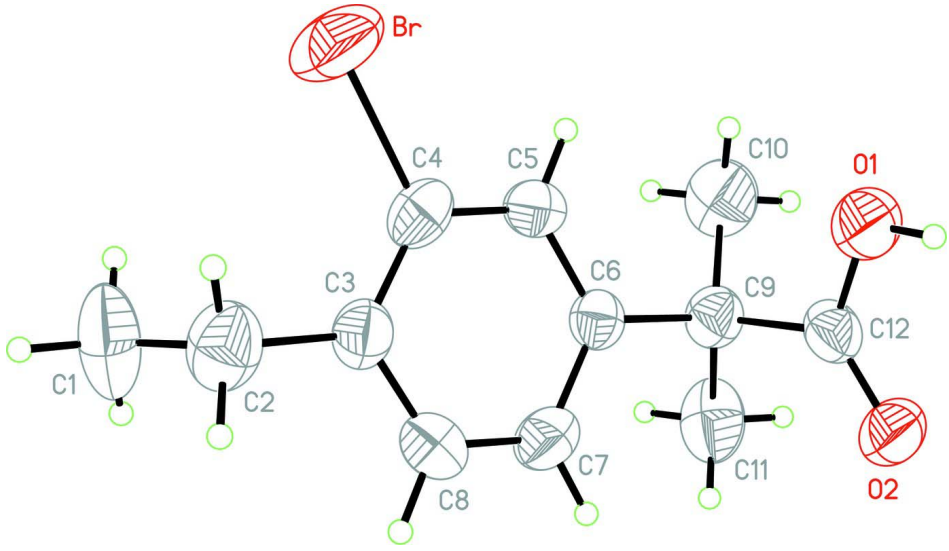

Figure 1

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

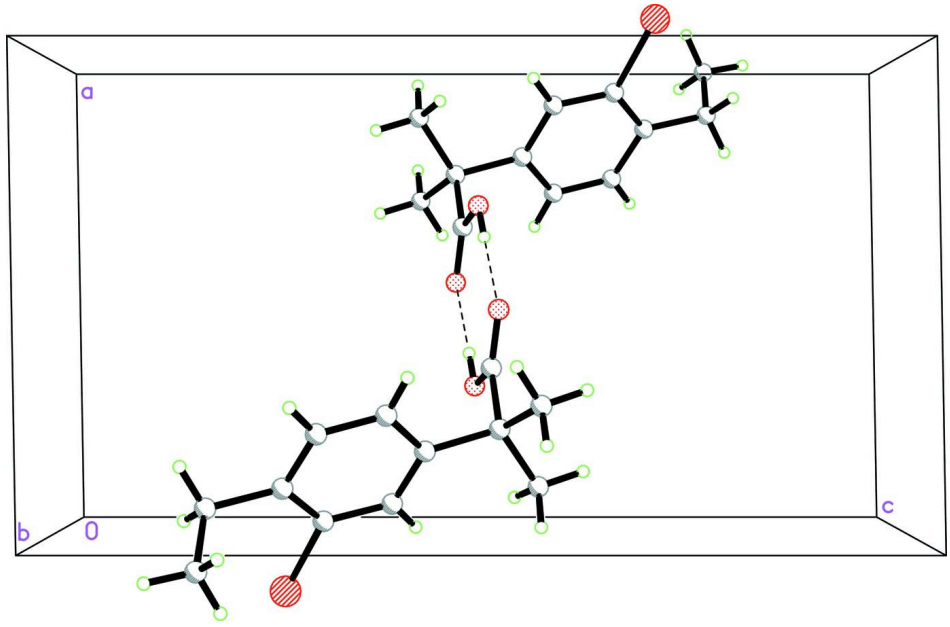

Figure 2

A packing diagram of the title compound. Hydrogen bonds are shown as dashed lines.

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Crystal data

$C_{12}H_{15}BrO_2$
$M_r = 271.15$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 9.7370\ (19)$ Å
$b = 7.2930\ (15)$ Å
$c = 17.433\ (4)$ Å
$\beta = 90.98\ (3)°$
$V = 1237.8\ (4)$ Å^3
$Z = 4$

$F(000) = 552$
$D_x = 1.455$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10–13°$
$\mu = 3.30$ mm^{-1}
$T = 298$ K
Block, colourless
0.20 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.558$, $T_{max} = 0.734$
2389 measured reflections
2246 independent reflections
1171 reflections with $I > 2\sigma(I)$
$R_{int} = 0.051$
$\theta_{max} = 25.3°$, $\theta_{min} = 2.4°$
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 8$
$l = -20 \rightarrow 20$
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.065$
$wR(F^2) = 0.157$
$S = 1.00$
2246 reflections
136 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
$w = 1/[\sigma^2(F_o^2) + (0.073P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.37$ e Å^{-3}
$\Delta\rho_{min} = -0.56$ e Å^{-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 $F^2 > \sigma(F^2)$ 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Br	0.59774 (8)	0.20831 (13)	0.22133 (4)	0.0837 (4)
O1	0.1885 (4)	0.0103 (6)	0.0039 (3)	0.0702 (14)
H1D	0.1216	−0.0561	0.0099	0.105*
C1	0.5163 (8)	0.6662 (12)	0.2979 (4)	0.095 (3)
H1A	0.5349	0.6986	0.3504	0.142*
H1B	0.4845	0.7723	0.2703	0.142*
H1C	0.5988	0.6212	0.2751	0.142*
O2	0.0282 (4)	0.2132 (6)	−0.0248 (3)	0.0621 (12)
C2	0.4083 (6)	0.5204 (10)	0.2946 (3)	0.0651 (19)
H2A	0.3259	0.5656	0.3186	0.078*
H2B	0.4402	0.4145	0.3234	0.078*

C3	0.3738 (6)	0.4625 (9)	0.2136 (3)	0.0456 (15)
C4	0.4468 (5)	0.3321 (9)	0.1736 (3)	0.0453 (15)
C5	0.4157 (5)	0.2833 (8)	0.0980 (3)	0.0420 (14)
H5A	0.4688	0.1960	0.0733	0.050*
C6	0.3068 (5)	0.3641 (8)	0.0596 (3)	0.0376 (14)
C7	0.2310 (6)	0.4946 (9)	0.0993 (3)	0.0551 (17)
H7A	0.1569	0.5521	0.0751	0.066*
C8	0.2650 (6)	0.5387 (9)	0.1738 (4)	0.0585 (18)
H8A	0.2115	0.6251	0.1987	0.070*
C9	0.2652 (6)	0.3128 (8)	−0.0232 (3)	0.0458 (15)
C10	0.3853 (7)	0.2252 (10)	−0.0659 (4)	0.069 (2)
H10A	0.3562	0.1951	−0.1173	0.103*
H10B	0.4141	0.1157	−0.0397	0.103*
H10C	0.4605	0.3102	−0.0675	0.103*
C11	0.2130 (7)	0.4781 (9)	−0.0680 (4)	0.067 (2)
H11A	0.1368	0.5316	−0.0419	0.100*
H11B	0.1840	0.4401	−0.1185	0.100*
H11C	0.2853	0.5669	−0.0720	0.100*
C12	0.1475 (6)	0.1738 (8)	−0.0153 (3)	0.0456 (15)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br	0.0699 (5)	0.1100 (7)	0.0703 (5)	0.0306 (5)	−0.0219 (4)	0.0089 (5)
O1	0.055 (3)	0.041 (3)	0.113 (4)	−0.007 (2)	−0.016 (3)	0.011 (3)
C1	0.103 (6)	0.102 (7)	0.078 (5)	−0.038 (6)	−0.012 (5)	−0.032 (5)
O2	0.046 (3)	0.046 (3)	0.094 (3)	−0.001 (2)	−0.016 (2)	0.007 (2)
C2	0.063 (4)	0.082 (5)	0.050 (4)	−0.007 (4)	0.000 (3)	−0.003 (4)
C3	0.042 (3)	0.053 (4)	0.041 (3)	−0.003 (3)	−0.002 (3)	−0.005 (3)
C4	0.035 (3)	0.054 (4)	0.047 (3)	−0.007 (3)	−0.002 (3)	0.010 (3)
C5	0.041 (3)	0.040 (3)	0.045 (3)	0.004 (3)	0.002 (3)	0.000 (3)
C6	0.038 (3)	0.036 (3)	0.039 (3)	−0.005 (3)	0.001 (3)	0.001 (3)
C7	0.046 (4)	0.065 (5)	0.054 (4)	0.011 (3)	−0.013 (3)	−0.006 (4)
C8	0.059 (4)	0.054 (5)	0.062 (4)	0.009 (3)	−0.003 (3)	−0.019 (3)
C9	0.048 (3)	0.048 (4)	0.041 (3)	0.000 (3)	−0.005 (3)	−0.001 (3)
C10	0.067 (4)	0.090 (6)	0.049 (4)	0.000 (4)	0.008 (3)	−0.009 (4)
C11	0.089 (5)	0.064 (5)	0.047 (4)	−0.014 (4)	−0.017 (4)	0.013 (4)
C12	0.057 (4)	0.034 (4)	0.045 (3)	0.001 (3)	−0.011 (3)	−0.001 (3)

Geometric parameters (Å, º)

Br—C4	1.904 (6)	C5—H5A	0.93
O1—C12	1.299 (7)	C6—C7	1.395 (8)
O1—H1D	0.82	C6—C9	1.539 (7)
C1—C2	1.496 (9)	C7—C8	1.373 (8)
C1—H1A	0.96	C7—H7A	0.93
C1—H1B	0.96	C8—H8A	0.93
C1—H1C	0.96	C9—C11	1.520 (8)

O2—C12	1.205 (7)	C9—C10	1.536 (9)
C2—C3	1.505 (8)	C9—C12	1.538 (8)
C2—H2A	0.97	C10—H10A	0.96
C2—H2B	0.97	C10—H10B	0.96
C3—C8	1.374 (8)	C10—H10C	0.96
C3—C4	1.383 (8)	C11—H11A	0.96
C4—C5	1.394 (8)	C11—H11B	0.96
C5—C6	1.376 (7)	C11—H11C	0.96

C12—O1—H1D	109.5	C8—C7—H7A	119.7
C2—C1—H1A	109.5	C6—C7—H7A	119.7
C2—C1—H1B	109.5	C7—C8—C3	123.7 (6)
H1A—C1—H1B	109.5	C7—C8—H8A	118.2
C2—C1—H1C	109.5	C3—C8—H8A	118.2
H1A—C1—H1C	109.5	C11—C9—C10	109.3 (5)
H1B—C1—H1C	109.5	C11—C9—C12	109.0 (5)
C1—C2—C3	112.4 (6)	C10—C9—C12	110.1 (5)
C1—C2—H2A	109.1	C11—C9—C6	111.7 (5)
C3—C2—H2A	109.1	C10—C9—C6	111.5 (5)
C1—C2—H2B	109.1	C12—C9—C6	105.1 (4)
C3—C2—H2B	109.1	C9—C10—H10A	109.5
H2A—C2—H2B	107.8	C9—C10—H10B	109.5
C8—C3—C4	115.0 (5)	H10A—C10—H10B	109.5
C8—C3—C2	121.2 (6)	C9—C10—H10C	109.5
C4—C3—C2	123.8 (5)	H10A—C10—H10C	109.5
C3—C4—C5	123.2 (5)	H10B—C10—H10C	109.5
C3—C4—Br	120.3 (4)	C9—C11—H11A	109.5
C5—C4—Br	116.5 (5)	C9—C11—H11B	109.5
C6—C5—C4	120.2 (5)	H11A—C11—H11B	109.5
C6—C5—H5A	119.9	C9—C11—H11C	109.5
C4—C5—H5A	119.9	H11A—C11—H11C	109.5
C5—C6—C7	117.5 (5)	H11B—C11—H11C	109.5
C5—C6—C9	122.6 (5)	O2—C12—O1	123.0 (6)
C7—C6—C9	119.9 (5)	O2—C12—C9	123.3 (5)
C8—C7—C6	120.5 (6)	O1—C12—C9	113.7 (5)

C1—C2—C3—C8	−95.0 (8)	C2—C3—C8—C7	178.3 (6)
C1—C2—C3—C4	84.7 (8)	C5—C6—C9—C11	−144.6 (6)
C8—C3—C4—C5	1.5 (9)	C7—C6—C9—C11	36.7 (7)
C2—C3—C4—C5	−178.3 (6)	C5—C6—C9—C10	−22.0 (8)
C8—C3—C4—Br	−178.4 (4)	C7—C6—C9—C10	159.4 (6)
C2—C3—C4—Br	1.9 (8)	C5—C6—C9—C12	97.3 (6)
C3—C4—C5—C6	−0.9 (9)	C7—C6—C9—C12	−81.4 (6)
Br—C4—C5—C6	178.9 (4)	C11—C9—C12—O2	−18.2 (8)
C4—C5—C6—C7	0.2 (8)	C10—C9—C12—O2	−138.1 (6)
C4—C5—C6—C9	−178.5 (5)	C6—C9—C12—O2	101.7 (6)
C5—C6—C7—C8	−0.2 (9)	C11—C9—C12—O1	163.1 (5)
C9—C6—C7—C8	178.5 (6)	C10—C9—C12—O1	43.2 (7)

C6—C7—C8—C3	0.9 (11)	C6—C9—C12—O1	−77.0 (6)
C4—C3—C8—C7	−1.5 (10)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*D*···O2^{i}	0.82	1.88	2.696 (6)	178

Symmetry code: (i) −*x*, −*y*, −*z*.