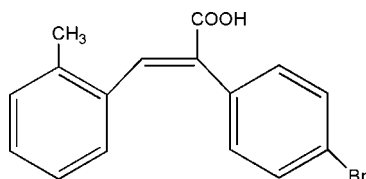# (*E*)-2-(4-Bromophenyl)-3-*o*-tolylacrylic acid

Mukhtiar Hussain,[a] Hanif Muhammad,[a] Saqib Ali[a]* and Don VanDerveer[b]

[a]Department of Chemistry, Quaid-I-Azam University, Islamabad 45320, Pakistan, and [b]Department of Chemistry, Clemson University, Clemson, SC 29634-0973, USA

Correspondence e-mail: drsa54@yahoo.com

The solid-state structure of the title compound, $C_{16}H_{13}BrO_2$, demonstrates an *E* configuration about the central C=C bond. Symmetry-related molecules are linked by O—H···O hydrogen bonds [O···O = 2.617 (3) Å], forming centrosymmetric carboxylic acid dimers.

Related literature

For related literature, see: Allen *et al.* (1987); Forgo *et al.* (2005); Hussain *et al.* (2006); Nodiff *et al.* (1971).

Experimental

Crystal data

$C_{16}H_{13}BrO_2$	Monoclinic, $P2_1/c$
M_r = 317.17	a = 6.0641 (12) Å
b = 15.208 (3) Å	Mo $K\alpha$ radiation
c = 15.187 (3) Å	μ = 2.93 mm^{-1}
β = 91.98 (3)°	T = 298 (2) K
V = 1399.7 (5) Å^3	$0.72 \times 0.48 \times 0.12$ mm
Z = 4	

Data collection

Rigaku AFC8 diffractometer with Mercury CCD	10277 measured reflections
Absorption correction: multi-scan (*REQAB*; Rigaku/MSC, 1999)	2454 independent reflections
T_{min} = 0.222, T_{max} = 0.706	2133 reflections with $I > 2\sigma(I)$
	R_{int} = 0.055

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041	175 parameters
$wR(F^2)$ = 0.098	H-atom parameters constrained
S = 1.11	$\Delta\rho_{max}$ = 0.43 e Å^{-3}
2454 reflections	$\Delta\rho_{min}$ = -0.40 e Å^{-3}

Data collection: *CrystalClear* (Rigaku/MSC, 2001); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.

Bruker (2000). *SHELXTL*. Version 6.10. Bruker AXS Inc., Madison, Wisconsin, USA.

Forgo, P., Felfoeldi, K. & Palinko, I. (2005). *J. Mol. Struct.* **744–747**, F273–F276.

Hussain, M., Hanif, M., Ali, S., Altaf, M. & Stoeckli-Evans, H. (2006). *Acta Cryst.* E**62**, o5020–o5021.

Nodiff, E. A., Tanabe, K., Seyfried, C., Matsuura, S., Kondo, Y., Chen, E. H. & Tyagi, M. P. (1971). *J. Med. Chem.* **14**, 921–925.

Rigaku/MSC (1999). *REQAB*. Rigaku/MSC, The Woodlands, Texas, USA.

Rigaku/MSC (2001). *CrystalClear*. Rigaku/MSC, The Woodlands, Texas, USA.

supplementary materials

(*E*)-2-(4-Bromophenyl)-3-*o*-tolylacrylic acid

M. Hussain, H. Muhammad, S. Ali and D. VanDerveer

Comment

Cinnamic Acid derived antibacterial drugs have increased the importance of these acids, especially halogenated Cinnamic acids. These acids have been revealed to enhance the efficiency of prototype medicinals (Nodiff *et al.*, 1971). The derivatives of these acids have been used as precursors in the shikimic acid metabolic pathways of higher plants (Forgo *et al.*, 2005). In an extension of such studies, in order to observe the effect of different substituents for improvement of the efficacy of these drugs, we have synthesized derivitives of these compounds and report the structure of compound (I). In the molecular structure of Compound (I), the central C8—C9 bond length of acrylate moiety proves a clear double bond character, and bonds C1—C8 and C9—C11 single bond character, which confirms localized bonding. In all the bond lengths and bond angles, a comparable behavior is observed as found in (Allen *et al.*, 1987), 3-(6-nitro-1, 3-benzodioxol-5-yl)-2-phenylacrylic acid, (II) (Hussain *et al.*, 2006). In the crystal structure of (I), Centro symmetric dimers are formed *via* O—H···O hydrogen bonds linking carboxylic acid groups. This arrangement is like that observed in the crystal structure of (II). In compound (I), these dimers are further connected by small links which form a slab like structure of the compound.

Experimental

Compound (I) was synthesized as essayed in (Nodiff *et al.*, 1971). A mixture of 2-Methyl benzaldehyde (6.6 mmol), (4-bromophenyl) acetic acid (6.6 mmol), potassium carbonate (15.4 mmol) and acetic anhydride (15.4 mmol) was slowly heated to 358 K and continued at that temperature for 24 h. To this hot solution, 10.0 ml distilled water and 5.0 ml 10% HCl were added sequentially. The solution was stirred for a further 2 h and after that filtered. Impurities were removed from the precipitated product by washing with water. Recrystallization was carried out in chloroform. (yield; 80%, m.p. 163–168 C.

Refinement

The molecules form hydrogen-bonded dimers across inversion centres. The O1—O2 distance is 2.617 (4) Å.
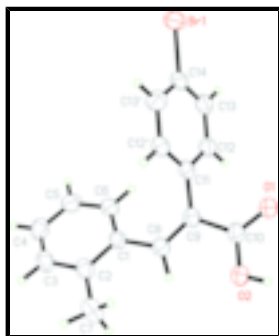
Figures

Fig. 1. A thermal ellipsoid perspective drawing of the molecule, 50% probability.
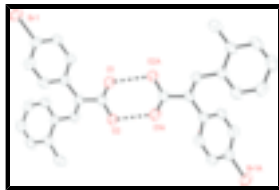
Fig. 2. Thermal ellipsoid perspective drawing showing the hydrogen-bonded dimer.

(*E*)-2-(4-Bromophenyl)-3-*o*-tolylacrylic acid

Crystal data

$C_{16}H_{13}BrO_2$	$F_{000} = 640$
$M_r = 317.17$	$D_x = 1.505$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 6.0641$ (12) Å	Cell parameters from 4981 reflections
$b = 15.208$ (3) Å	$\theta = 3.0–25.7°$
$c = 15.187$ (3) Å	$\mu = 2.93$ mm^{-1}
$\beta = 91.98$ (3)°	$T = 298$ (2) K
$V = 1399.7$ (5) Å^3	Plate, colourless
$Z = 4$	$0.72 \times 0.48 \times 0.12$ mm

Data collection

Rigaku Mercury CCD diffractometer	2454 independent reflections
Radiation source: sealed tube	2133 reflections with $I > 2\sigma(I)$
Monochromator: graphite monochromator	$R_{int} = 0.055$
Detector resolution: 14.6199 pixels mm^{-1}	$\theta_{max} = 25.0°$
$T = 298(2)$ K	$\theta_{min} = 3.0°$
dtprofit.ref scans	$h = -7 \rightarrow 7$
Absorption correction: multi-scan (REQAB; Rigaku/MSC, 1999)	$k = -18 \rightarrow 14$
$T_{min} = 0.222$, $T_{max} = 0.706$	$l = -18 \rightarrow 18$
10277 measured reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.041$	$w = 1/[\sigma^2(F_o^2) + (0.0375P)^2 + 1.3644P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.098$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.11$	$\Delta\rho_{max} = 0.43$ e Å^{-3}
2454 reflections	$\Delta\rho_{min} = -0.40$ e Å^{-3}
175 parameters	Extinction correction: SHELXTL (Bruker, 2000), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Primary atom site location: structure-invariant direct methods

Extinction coefficient: 0.0086 (17)

Secondary atom site location: difference Fourier map

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 $F^2 > 2sigma(F^2)$ 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	$U_{iso}*/U_{eq}$
Br1	0.34229 (6)	0.49805 (2)	0.61545 (2)	0.04791 (18)
O1	0.0639 (4)	0.89770 (16)	0.50704 (15)	0.0498 (6)
O2	0.2513 (4)	1.00144 (14)	0.43676 (16)	0.0423 (5)
H2	0.1630	1.0340	0.4617	0.051*
C1	0.6456 (5)	0.8217 (2)	0.30564 (17)	0.0333 (6)
C2	0.8343 (5)	0.8622 (2)	0.27221 (19)	0.0384 (7)
C3	0.9804 (5)	0.8094 (3)	0.2261 (2)	0.0449 (8)
H3	1.1139	0.8351	0.2056	0.054*
C4	0.9393 (6)	0.7225 (3)	0.2094 (2)	0.0495 (9)
H4	1.0415	0.6885	0.1766	0.059*
C5	0.7502 (6)	0.6839 (2)	0.2397 (2)	0.0467 (8)
H5	0.7189	0.6232	0.2271	0.056*
C6	0.6057 (5)	0.7333 (2)	0.2884 (2)	0.0408 (7)
H6	0.4762	0.7059	0.3106	0.049*
C7	0.8753 (6)	0.9589 (3)	0.2828 (2)	0.0510 (9)
H7A	1.0260	0.9718	0.2701	0.076*
H7B	0.8473	0.9761	0.3422	0.076*
H7C	0.7790	0.9909	0.2428	0.076*
C8	0.4923 (5)	0.8763 (2)	0.35516 (18)	0.0343 (7)
H8	0.4826	0.9369	0.3376	0.041*
C9	0.3640 (4)	0.8527 (2)	0.42163 (18)	0.0325 (6)
C10	0.2132 (5)	0.9190 (2)	0.45829 (19)	0.0371 (7)
C11	0.3587 (5)	0.7651 (2)	0.46472 (17)	0.0321 (6)
C12'	0.5391 (5)	0.7379 (2)	0.51683 (18)	0.0352 (7)
H12'	0.6678	0.7746	0.5218	0.042*
C12	0.1737 (5)	0.7111 (2)	0.45780 (19)	0.0371 (7)
H12	0.0482	0.7294	0.4221	0.045*
C13'	0.5357 (5)	0.6588 (2)	0.56153 (19)	0.0397 (7)
H13'	0.6609	0.6404	0.5973	0.048*
C13	0.1680 (5)	0.6314 (2)	0.5017 (2)	0.0398 (7)

H13	0.0408	0.5940	0.4961	0.048*
C14	0.3499 (5)	0.6066 (2)	0.55412 (18)	0.0362 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0670 (3)	0.0365 (2)	0.0413 (2)	0.00595 (16)	0.01678 (16)	0.00548 (14)
O1	0.0596 (15)	0.0398 (14)	0.0518 (14)	0.0139 (11)	0.0273 (11)	0.0084 (11)
O2	0.0446 (12)	0.0343 (12)	0.0493 (13)	0.0058 (10)	0.0193 (10)	−0.0007 (10)
C1	0.0336 (15)	0.0408 (17)	0.0258 (13)	0.0039 (13)	0.0046 (11)	0.0038 (12)
C2	0.0329 (15)	0.052 (2)	0.0303 (15)	0.0013 (14)	0.0023 (11)	0.0037 (14)
C3	0.0361 (16)	0.065 (2)	0.0344 (16)	0.0060 (15)	0.0106 (13)	0.0085 (16)
C4	0.051 (2)	0.065 (3)	0.0337 (16)	0.0210 (18)	0.0112 (14)	0.0043 (16)
C5	0.066 (2)	0.0388 (19)	0.0359 (16)	0.0105 (16)	0.0116 (15)	0.0028 (14)
C6	0.0455 (17)	0.0439 (19)	0.0338 (15)	0.0043 (14)	0.0124 (13)	0.0040 (14)
C7	0.0441 (19)	0.060 (2)	0.0496 (19)	−0.0080 (17)	0.0098 (15)	−0.0006 (18)
C8	0.0363 (15)	0.0363 (17)	0.0306 (14)	0.0026 (13)	0.0047 (11)	0.0010 (13)
C9	0.0313 (14)	0.0366 (16)	0.0297 (14)	0.0011 (12)	0.0040 (11)	−0.0023 (12)
C10	0.0375 (15)	0.0411 (19)	0.0332 (15)	0.0067 (13)	0.0086 (12)	0.0008 (13)
C11	0.0339 (14)	0.0372 (17)	0.0256 (13)	0.0035 (12)	0.0084 (11)	−0.0021 (12)
C12'	0.0350 (15)	0.0399 (18)	0.0309 (14)	−0.0007 (13)	0.0047 (11)	−0.0021 (13)
C12	0.0302 (15)	0.0437 (19)	0.0377 (16)	0.0036 (13)	0.0040 (12)	0.0004 (14)
C13'	0.0461 (18)	0.0446 (19)	0.0281 (14)	0.0048 (14)	−0.0016 (12)	−0.0018 (13)
C13	0.0335 (15)	0.0417 (18)	0.0448 (17)	−0.0037 (13)	0.0103 (12)	−0.0014 (15)
C14	0.0458 (17)	0.0364 (17)	0.0272 (14)	0.0047 (13)	0.0126 (12)	0.0002 (12)

Geometric parameters (Å, °)

Br1—C14	1.896 (3)	C7—H7B	0.9599
O1—C10	1.233 (4)	C7—H7C	0.9599
O2—C10	1.318 (4)	C8—C9	1.344 (4)
O2—H2	0.8299	C8—H8	0.9600
C1—C6	1.390 (5)	C9—C10	1.483 (4)
C1—C2	1.410 (4)	C9—C11	1.485 (4)
C1—C8	1.472 (4)	C11—C12'	1.391 (4)
C2—C3	1.401 (5)	C11—C12	1.391 (4)
C2—C7	1.499 (5)	C12'—C13'	1.381 (5)
C3—C4	1.367 (5)	C12'—H12'	0.9600
C3—H3	0.9600	C12—C13	1.384 (5)
C4—C5	1.382 (5)	C12—H12	0.9600
C4—H4	0.9600	C13'—C14	1.380 (4)
C5—C6	1.387 (5)	C13'—H13'	0.9600
C5—H5	0.9600	C13—C14	1.391 (4)
C6—H6	0.9600	C13—H13	0.9600
C7—H7A	0.9599		
C10—O2—H2	109.5	C9—C8—H8	115.7
C6—C1—C2	119.6 (3)	C1—C8—H8	115.7
C6—C1—C8	122.2 (3)	C8—C9—C10	118.7 (3)

C2—C1—C8	118.2 (3)	C8—C9—C11	126.4 (3)
C3—C2—C1	117.7 (3)	C10—C9—C11	114.9 (2)
C3—C2—C7	120.7 (3)	O1—C10—O2	122.5 (3)
C1—C2—C7	121.6 (3)	O1—C10—C9	121.4 (3)
C4—C3—C2	122.2 (3)	O2—C10—C9	116.0 (3)
C4—C3—H3	118.9	C12'—C11—C12	119.0 (3)
C2—C3—H3	118.9	C12'—C11—C9	119.3 (3)
C3—C4—C5	119.8 (3)	C12—C11—C9	121.7 (3)
C3—C4—H4	120.1	C13'—C12'—C11	120.9 (3)
C5—C4—H4	120.1	C13'—C12'—H12'	119.6
C4—C5—C6	119.6 (3)	C11—C12'—H12'	119.6
C4—C5—H5	120.2	C13—C12—C11	120.9 (3)
C6—C5—H5	120.2	C13—C12—H12	119.5
C5—C6—C1	121.0 (3)	C11—C12—H12	119.5
C5—C6—H6	119.5	C14—C13'—C12'	119.2 (3)
C1—C6—H6	119.5	C14—C13'—H13'	120.4
C2—C7—H7A	109.5	C12'—C13'—H13'	120.4
C2—C7—H7B	109.5	C12—C13—C14	118.8 (3)
H7A—C7—H7B	109.5	C12—C13—H13	120.6
C2—C7—H7C	109.5	C14—C13—H13	120.6
H7A—C7—H7C	109.5	C13'—C14—C13	121.3 (3)
H7B—C7—H7C	109.5	C13'—C14—Br1	119.7 (2)
C9—C8—C1	128.7 (3)	C13—C14—Br1	119.0 (2)
C6—C1—C2—C3	−3.1 (4)	C8—C9—C10—O2	−14.9 (4)
C8—C1—C2—C3	179.2 (3)	C11—C9—C10—O2	164.3 (3)
C6—C1—C2—C7	175.1 (3)	C8—C9—C11—C12'	69.4 (4)
C8—C1—C2—C7	−2.6 (4)	C10—C9—C11—C12'	−109.6 (3)
C1—C2—C3—C4	3.4 (4)	C8—C9—C11—C12	−113.7 (3)
C7—C2—C3—C4	−174.8 (3)	C10—C9—C11—C12	67.3 (4)
C2—C3—C4—C5	−1.2 (5)	C12—C11—C12'—C13'	−0.3 (4)
C3—C4—C5—C6	−1.3 (5)	C9—C11—C12'—C13'	176.7 (3)
C4—C5—C6—C1	1.5 (5)	C12'—C11—C12—C13	−0.1 (4)
C2—C1—C6—C5	0.8 (4)	C9—C11—C12—C13	−177.0 (3)
C8—C1—C6—C5	178.3 (3)	C11—C12'—C13'—C14	−0.1 (4)
C6—C1—C8—C9	33.4 (5)	C11—C12—C13—C14	0.9 (4)
C2—C1—C8—C9	−149.0 (3)	C12'—C13'—C14—C13	0.9 (4)
C1—C8—C9—C10	−176.8 (3)	C12'—C13'—C14—Br1	−179.8 (2)
C1—C8—C9—C11	4.2 (5)	C12—C13—C14—C13'	−1.3 (4)
C8—C9—C10—O1	166.2 (3)	C12—C13—C14—Br1	179.4 (2)
C11—C9—C10—O1	−14.7 (4)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O1[i]	0.83	1.80	2.617 (3)	167

Symmetry codes: (i) −*x*, −*y*+2, −*z*+1.

Fig. 1

Fig. 2