Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-Ethyl 3-(3-bromophenyl)-2-cyanoacrylate

Yin Ye,* Wei-Li Shen and Xian-Wen Wei

College of Chemistry and Materials Science, Anhui Key Laboratory of Functional Molecular Solid, Anhui Normal University, Wuhu 241000, People's Republic of China
Correspondence e-mail: yeyin307@yahoo.com.cn

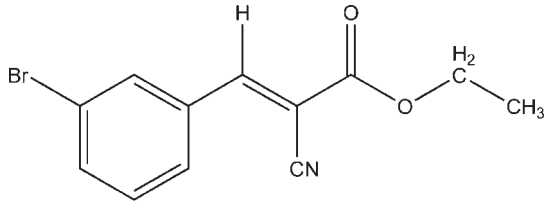
Received 16 September 2009; accepted 29 September 2009

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.034; wR factor = 0.081; data-to-parameter ratio = 18.5.

The title molecule, $C_{12}H_{10}BrNO_2$, adopts an *E* configuration with respect to the C=C bond of the acrylate unit. In the crystal structure, molecules are connected by a pair of intermolecular C—H···O hydrogen bonds, forming a centrosymmetric dimer.

Related literature

For the synthesis, see: Lapworth & Baker (1927). For the title compound as an intermediate in drug synthesis, see: Obniska *et al.* (2005).



Experimental

Crystal data

$C_{12}H_{10}BrNO_2$
M_r = 280.12
Monoclinic, $P2_1/c$
a = 7.6147 (7) Å
b = 21.6015 (19) Å
c = 7.6044 (7) Å
β = 110.370 (1)°
V = 1172.62 (18) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 3.49 mm^{-1}
T = 298 K
0.17 × 0.14 × 0.09 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.597, T_{max} = 0.751
10143 measured reflections
2698 independent reflections
1730 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.081
S = 1.00
2698 reflections
146 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6···O1^{i}	0.93	2.47	3.323 (3)	152

Symmetry code: (i) $-x+1, -y+1, -z+1$.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; 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*.

This work was supported by the Science and Technology Fund of Anhui Province for Outstanding Youth (No. 08040106906), the National Natural Science Foundation (No. 20671002) of China, the State Education Ministry (EYTP, SRF for ROCS, SRFDP 20070370001) and the Education Department (No. KJ2008B169) of Anhui Province.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2461).

References

Bruker (2000). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Lapworth, A. & Baker, W. (1927). *Org. Synth.* **7**, 20–21.
Obniska, J., Jurczyk, S., Zejc, A., Kamiński, K., Tatarczyńska, E. & Stachowicz, K. (2005). *Pharmacol. Rep.* **57**, 170–175.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2009). E**65**, o2636 [https://doi.org/10.1107/S1600536809039518]

(*E*)-Ethyl 3-(3-bromophenyl)-2-cyanoacrylate

Yin Ye, Wei-Li Shen and Xian-Wen Wei

S1. Comment

The title compound is an important intermediate in drugs synthesis (Obniska *et al.*, 2005). In this paper, we report the structure of the title compound (I). In (I), the molecule adopts an *E* configuration. Both the C7═C8 and the cyano (C12≡N1) groups deviate from the mean plane of the benzene C1–C6 ring. The crystal packing is stabilized by intermolecular non-classic C—H···O hydrogen bonds.

S2. Experimental

The compound (I) was obtained by reaction of 3-bromobenzaldehyde (36.8 g, 0.2 mol) and ethyl 2-cyanoacetate (22.6 g, 0.2 mol) in the absolute ethanol (180 ml) containing 3.2 ml triethylamine according to the reported method (Lapworth *et al.*, 1927). Single crystals suitable for X-ray diffraction were obtained by evaporation of an ethanol solution at room temperature.

S3. Refinement

H atoms bonded to C atoms were introduced at calculated positions (C—H = 0.93–0.97 Å) and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C).

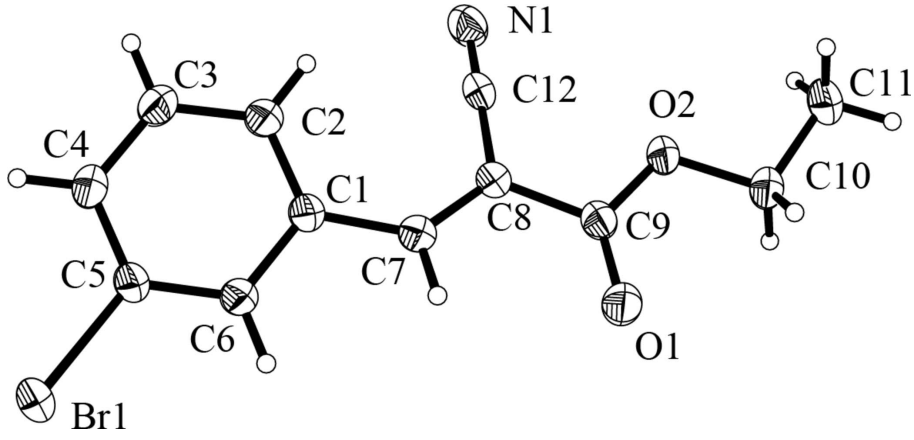


Figure 1

The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

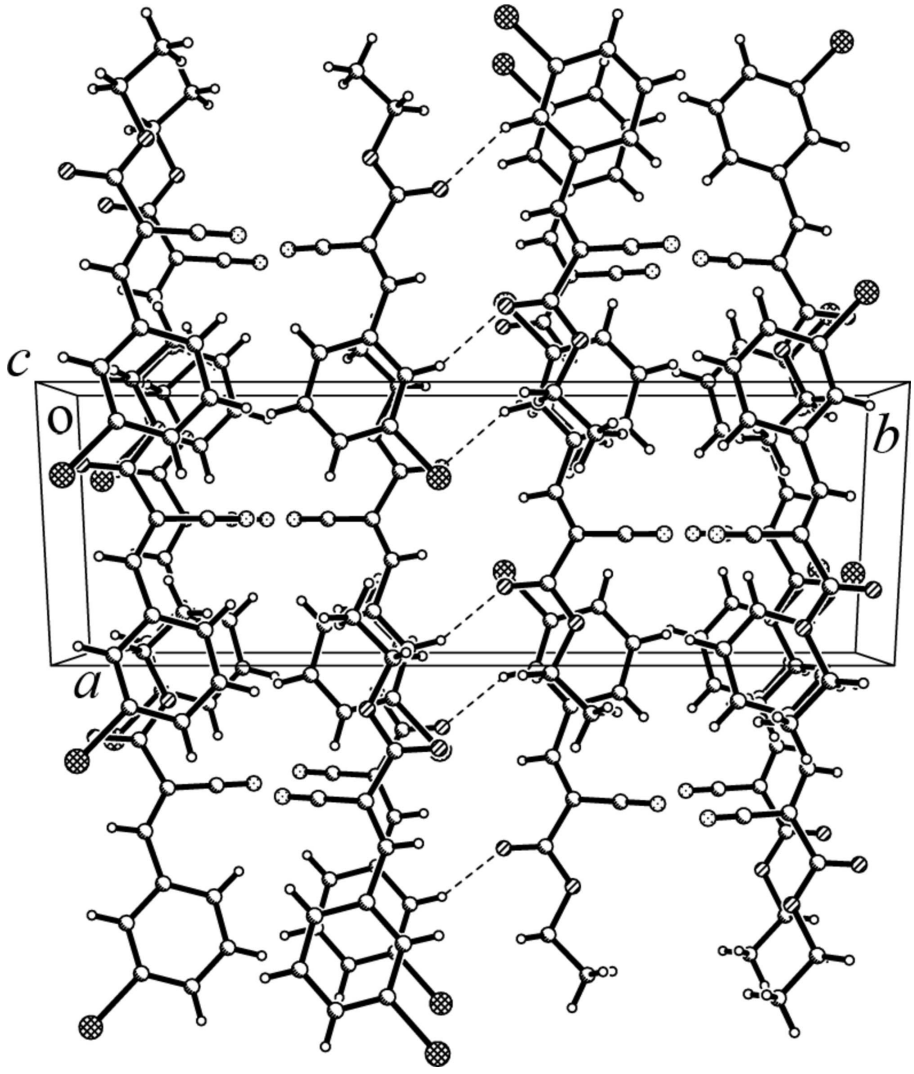


Figure 2

A packing diagram of (I) viewed down the *c* axis. Dotted lines show the C—H···O hydrogen bonds.

(*E*)-Ethyl 3-(3-bromophenyl)-2-cyanoacrylate

Crystal data

$C_{12}H_{10}BrNO_2$
$M_r = 280.12$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 7.6147\ (7)$ Å
$b = 21.6015\ (19)$ Å
$c = 7.6044\ (7)$ Å
$\beta = 110.370\ (1)°$
$V = 1172.62\ (18)$ Å^3
$Z = 4$
$F(000) = 560$
$D_x = 1.587$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2803 reflections
$\theta = 2.9–25.8°$
$\mu = 3.49$ mm^{-1}
$T = 298$ K
Block, colorless
0.17 × 0.14 × 0.09 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.597, T_{max} = 0.751
10143 measured reflections
2698 independent reflections
1730 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
θ_{max} = 27.6°, θ_{min} = 1.9°
h = −9→9
k = −28→28
l = −8→9

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.081
S = 1.00
2698 reflections
146 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
$w = 1/[\sigma^2(F_o^2) + (0.0337P)^2 + 0.2149P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
Br1	1.32627 (4)	0.459872 (14)	0.80815 (5)	0.06743 (14)
C5	1.1401 (4)	0.40161 (12)	0.6781 (4)	0.0491 (7)
C6	0.9564 (4)	0.42010 (12)	0.6053 (4)	0.0464 (6)
H6	0.9240	0.4608	0.6193	0.056*
C7	0.6257 (4)	0.39971 (12)	0.4366 (4)	0.0481 (6)
H7	0.6060	0.4373	0.4862	0.058*
C1	0.8179 (3)	0.37725 (11)	0.5098 (4)	0.0460 (6)
N1	0.4728 (3)	0.27353 (12)	0.1299 (4)	0.0694 (7)
C8	0.4721 (3)	0.37490 (11)	0.3089 (3)	0.0438 (6)
C4	1.1939 (4)	0.34169 (14)	0.6612 (4)	0.0626 (8)
H4	1.3192	0.3301	0.7101	0.075*
C2	0.8709 (4)	0.31640 (13)	0.4958 (4)	0.0629 (8)
H2	0.7806	0.2870	0.4362	0.075*
C10	−0.0327 (4)	0.40634 (13)	0.0878 (4)	0.0579 (8)
H10A	−0.0360	0.4404	0.0035	0.069*
H10B	−0.0578	0.4225	0.1957	0.069*

C11	−0.1756 (4)	0.35851 (14)	−0.0101 (5)	0.0682 (9)
H11A	−0.1452	0.3412	−0.1122	0.102*
H11B	−0.2972	0.3774	−0.0577	0.102*
H11C	−0.1759	0.3263	0.0768	0.102*
C3	1.0573 (4)	0.29951 (14)	0.5701 (5)	0.0754 (10)
H3	1.0912	0.2588	0.5582	0.090*
O2	0.1493 (2)	0.37612 (8)	0.1469 (2)	0.0517 (5)
O1	0.2787 (3)	0.46073 (9)	0.3104 (3)	0.0703 (6)
C12	0.4724 (3)	0.31789 (13)	0.2110 (4)	0.0500 (7)
C9	0.2926 (4)	0.40931 (13)	0.2581 (4)	0.0493 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.04546 (19)	0.0595 (2)	0.0794 (2)	−0.00844 (14)	−0.00088 (15)	−0.00303 (16)
C5	0.0422 (15)	0.0497 (16)	0.0478 (16)	−0.0039 (12)	0.0061 (13)	0.0000 (12)
C6	0.0477 (16)	0.0381 (14)	0.0488 (16)	0.0000 (11)	0.0109 (12)	0.0000 (12)
C7	0.0445 (15)	0.0409 (14)	0.0548 (17)	−0.0001 (11)	0.0119 (13)	−0.0013 (12)
C1	0.0394 (14)	0.0436 (14)	0.0476 (16)	−0.0031 (11)	0.0058 (12)	0.0004 (12)
N1	0.0519 (15)	0.0609 (16)	0.092 (2)	−0.0110 (12)	0.0204 (14)	−0.0237 (15)
C8	0.0381 (14)	0.0438 (14)	0.0474 (16)	−0.0023 (11)	0.0123 (12)	−0.0003 (12)
C4	0.0421 (16)	0.0558 (17)	0.076 (2)	0.0073 (13)	0.0024 (15)	−0.0037 (15)
C2	0.0503 (17)	0.0469 (16)	0.074 (2)	−0.0019 (13)	−0.0010 (15)	−0.0078 (14)
C10	0.0349 (15)	0.0605 (18)	0.073 (2)	0.0057 (13)	0.0125 (14)	−0.0015 (15)
C11	0.0390 (16)	0.077 (2)	0.079 (2)	−0.0025 (15)	0.0092 (15)	0.0018 (17)
C3	0.0539 (19)	0.0490 (17)	0.102 (3)	0.0099 (14)	0.0000 (18)	−0.0102 (17)
O2	0.0350 (10)	0.0511 (11)	0.0634 (12)	−0.0005 (8)	0.0098 (9)	−0.0070 (9)
O1	0.0505 (12)	0.0600 (13)	0.0904 (16)	0.0028 (10)	0.0119 (11)	−0.0251 (11)
C12	0.0329 (14)	0.0529 (16)	0.0594 (18)	−0.0069 (12)	0.0098 (13)	−0.0009 (14)
C9	0.0410 (15)	0.0534 (17)	0.0505 (17)	−0.0041 (12)	0.0120 (13)	−0.0033 (13)

Geometric parameters (Å, º)

Br1—C5	1.896 (2)	C4—H4	0.9300
C5—C6	1.372 (3)	C2—C3	1.382 (4)
C5—C4	1.377 (4)	C2—H2	0.9300
C6—C1	1.401 (3)	C10—O2	1.454 (3)
C6—H6	0.9300	C10—C11	1.497 (4)
C7—C8	1.344 (3)	C10—H10A	0.9700
C7—C1	1.456 (3)	C10—H10B	0.9700
C7—H7	0.9300	C11—H11A	0.9600
C1—C2	1.390 (4)	C11—H11B	0.9600
N1—C12	1.140 (3)	C11—H11C	0.9600
C8—C12	1.439 (4)	C3—H3	0.9300
C8—C9	1.484 (4)	O2—C9	1.333 (3)
C4—C3	1.374 (4)	O1—C9	1.197 (3)
C6—C5—C4	122.0 (2)	O2—C10—C11	107.1 (2)

C6—C5—Br1	119.3 (2)	O2—C10—H10A	110.3
C4—C5—Br1	118.7 (2)	C11—C10—H10A	110.3
C5—C6—C1	119.6 (2)	O2—C10—H10B	110.3
C5—C6—H6	120.2	C11—C10—H10B	110.3
C1—C6—H6	120.2	H10A—C10—H10B	108.6
C8—C7—C1	130.5 (2)	C10—C11—H11A	109.5
C8—C7—H7	114.8	C10—C11—H11B	109.5
C1—C7—H7	114.8	H11A—C11—H11B	109.5
C2—C1—C6	118.6 (2)	C10—C11—H11C	109.5
C2—C1—C7	124.5 (2)	H11A—C11—H11C	109.5
C6—C1—C7	116.9 (2)	H11B—C11—H11C	109.5
C7—C8—C12	123.9 (2)	C4—C3—C2	121.3 (3)
C7—C8—C9	118.6 (2)	C4—C3—H3	119.3
C12—C8—C9	117.5 (2)	C2—C3—H3	119.3
C3—C4—C5	118.2 (3)	C9—O2—C10	115.9 (2)
C3—C4—H4	120.9	N1—C12—C8	178.3 (3)
C5—C4—H4	120.9	O1—C9—O2	124.2 (3)
C3—C2—C1	120.2 (3)	O1—C9—C8	124.0 (2)
C3—C2—H2	119.9	O2—C9—C8	111.8 (2)
C1—C2—H2	119.9		

C4—C5—C6—C1	−0.3 (4)	C7—C1—C2—C3	−179.9 (3)
Br1—C5—C6—C1	−179.72 (19)	C5—C4—C3—C2	0.5 (5)
C5—C6—C1—C2	1.6 (4)	C1—C2—C3—C4	0.9 (5)
C5—C6—C1—C7	179.8 (2)	C11—C10—O2—C9	−171.7 (2)
C8—C7—C1—C2	−17.9 (5)	C10—O2—C9—O1	0.5 (4)
C8—C7—C1—C6	164.1 (3)	C10—O2—C9—C8	−179.3 (2)
C1—C7—C8—C12	−1.8 (5)	C7—C8—C9—O1	7.3 (4)
C1—C7—C8—C9	−178.8 (3)	C12—C8—C9—O1	−169.9 (3)
C6—C5—C4—C3	−0.8 (5)	C7—C8—C9—O2	−172.8 (2)
Br1—C5—C4—C3	178.7 (2)	C12—C8—C9—O2	9.9 (3)
C6—C1—C2—C3	−1.9 (5)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O1^{i}	0.93	2.47	3.323 (3)	152

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