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(E)-1,2-Bis(3-bromo-4-methylphenyl)-ethene

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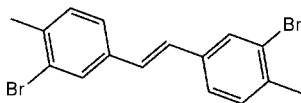
Received 15 December 2007; accepted 19 December 2007

 Key indicators: single-crystal X-ray study; $T = 173$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.025; wR factor = 0.054; data-to-parameter ratio = 16.9.

In the structure of the title compound, $\text{C}_{16}\text{H}_{14}\text{Br}_2$, the central $\text{C}=\text{C}$ bond length is 1.329 (4) Å and the two benzene rings are approximately coplanar with the double bond, with twist angles of 7.5 (2) and 13.6 (2)°.

Related literature

For related literature, see: Daik *et al.* (1998); Harada & Ogawa *et al.* (2004); Ogawa *et al.* (1992); Mallory *et al.* (2001).



Experimental

Crystal data

$\text{C}_{16}\text{H}_{14}\text{Br}_2$
 $M_r = 366.09$
 Monoclinic, $P2_1/c$
 $a = 6.3301$ (4) Å
 $b = 7.6499$ (5) Å
 $c = 28.164$ (2) Å
 $\beta = 91.208$ (1)°

$V = 1363.55$ (16) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 5.92$ mm⁻¹
 $T = 173$ (2) K
 $0.27 \times 0.19 \times 0.10$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 2004)
 $T_{\min} = 0.303$, $T_{\max} = 0.578$
 14025 measured reflections
 2793 independent reflections
 2393 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.027$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.025$
 $wR(F^2) = 0.054$
 $S = 1.08$
 2793 reflections
 165 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.42$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.32$ e Å⁻³

Data collection: APEX2 (Bruker, 2006); cell refinement: APEX2; data reduction: SAINT-Plus (Bruker, 2006); program(s) used to solve structure: SHELXS97 (Sheldrick, 1990); program(s) used to refine structure: SHELXTL (Sheldrick, 2003); molecular graphics: Mercury (Macrae *et al.*, 2006); software used to prepare material for publication: publCIF (Westrip, 2008).

Support provided by Dr Peter Dibble and the Natural Sciences and Engineering Research Council of Canada (NSERC) is gratefully acknowledged. The diffractometer was purchased with the help of NSERC and the University of Lethbridge.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2061).

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supporting information

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(E)-1,2-Bis(3-bromo-4-methylphenyl)ethene

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S1. Comment

The title compound, (I) (Fig. 1), was prepared by a Ti catalyzed McMurray coupling (Mallory *et al.*, 2001) with 99% E selectivity. The almost-planar molecules pack (Fig. 2) in slipped stacks with T-contacts typical for aromatic molecules. The only other isomeric stilbene for which a structure has been reported is *Z*-1,2-bis-(4-bromophenyl)-1,2-dimethyl-ethene (Daik *et al.*, 1998) for which C=C is 1.330 (10) and 1.344 (10) Å (for two independent molecules in an asymmetric unit). Unlike in I, the phenyl rings in this compound are twisted almost orthogonal to the double bond, perhaps because of steric interactions between methyl and phenyl groups. More structurally comparable alkenes include *E*-1,2-bis-(2,4-dimethylphenyl)ethene and *E*-1,2-bis-(2,4,5-trimethylphenyl)ethene (Ogawa *et al.*, 1992) for which C=C are 1.320 (4) and 1.327 (3) Å, respectively. A detailed study of geometric distortions in *trans*-stilbene has recently been published (Harada & Ogawa, 2004).

S2. Experimental

At 273 K under N₂, 0.18 ml (1.6 mmol) of TiCl₄ was stirred with 0.18 g (2.8 mmol) Zn dust in 25 ml of dry THF. To this mixture was added 0.25 g (1.3 mmol) of 3-bromo-4-methylbenzaldehyde and refluxed for 4 h before being quenched with 25 ml of 1.0 M HCl. After extracting with hexanes the organic phase was washed with brine solution and dried over MgSO₄. Removal of solvent resulted in a white powder that was recrystallized from ethyl acetate to give 0.11 g (yield = 58%) of the desired product as colorless crystals.

S3. Refinement

The H-atoms were included in the refinements at geometrically idealized positions with C—H distances 0.95 and 0.98 Å for non-methyl and methyl type H-atoms, respectively; U_{iso} values were $1.2U_{\text{eq}}$ of the carrier atom or $1.5U_{\text{eq}}$ for the non-methyl and methyl groups, respectively.

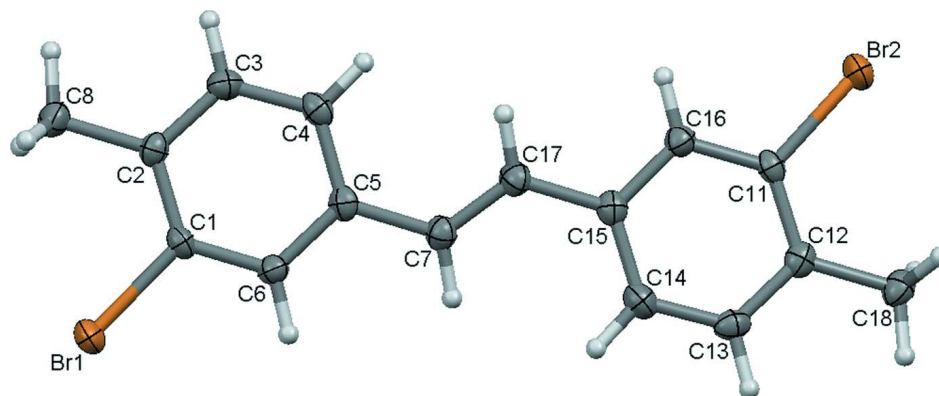


Figure 1

A view of (I) plotted with displacement ellipsoids at 50% probability level.

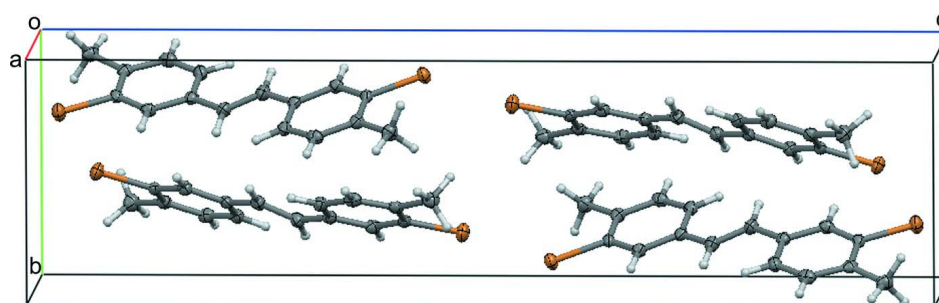


Figure 2

Unit-cell contents of (I) showing the herringbone arrangement of slipped-stacks with T-contacts between registers.

(E)-1,2-Bis(3-bromo-4-methylphenyl)ethene

Crystal data

$C_{16}H_{14}Br_2$

$M_r = 366.09$

Monoclinic, $P2_1/c$

Hall symbol: $-P2_1/c$

$a = 6.3301(4) \text{ \AA}$

$b = 7.6499(5) \text{ \AA}$

$c = 28.164(2) \text{ \AA}$

$\beta = 91.208(1)^\circ$

$V = 1363.55(16) \text{ \AA}^3$

$Z = 4$

$F(000) = 720$

$D_x = 1.783 \text{ Mg m}^{-3}$

Melting point: 424.75 K

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

$\mu = 5.92 \text{ mm}^{-1}$

$T = 173 \text{ K}$

Prism, colourless

$0.27 \times 0.19 \times 0.10 \text{ mm}$

Data collection

Bruker APEXII CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube, Bruker
D8

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 2004)

$T_{\min} = 0.303$, $T_{\max} = 0.578$

14025 measured reflections

2793 independent reflections

2393 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.027$

$\theta_{\max} = 26.4^\circ$, $\theta_{\min} = 2.8^\circ$

$h = -7 \rightarrow 7$

$k = -9 \rightarrow 9$

$l = -35 \rightarrow 35$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.025$
 $wR(F^2) = 0.054$
 $S = 1.09$
 2793 reflections
 165 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
 $w = 1/[\sigma^2(F_o^2) + (0.0179P)^2 + 1.6865P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.43 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.32 \text{ e } \text{Å}^{-3}$

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	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.08255 (4)	0.93057 (4)	0.562520 (9)	0.02689 (8)
C1	0.0145 (4)	0.8529 (3)	0.62456 (8)	0.0197 (5)
C2	-0.1806 (4)	0.7739 (3)	0.63183 (9)	0.0217 (5)
C3	-0.2158 (4)	0.7151 (3)	0.67779 (10)	0.0241 (6)
H3	-0.3476	0.6620	0.6844	0.029*
C4	-0.0673 (4)	0.7309 (3)	0.71400 (9)	0.0237 (6)
H4	-0.0972	0.6867	0.7446	0.028*
C5	0.1280 (4)	0.8119 (3)	0.70600 (9)	0.0213 (6)
C6	0.1650 (4)	0.8749 (3)	0.66049 (9)	0.0212 (5)
H6	0.2939	0.9332	0.6541	0.025*
C7	0.2912 (4)	0.8315 (4)	0.74343 (9)	0.0241 (6)
H7	0.4101	0.9015	0.7361	0.029*
C8	-0.3452 (4)	0.7506 (4)	0.59307 (10)	0.0282 (6)
H8A	-0.2872	0.6792	0.5676	0.034*
H8B	-0.3865	0.8653	0.5804	0.034*
H8C	-0.4693	0.6922	0.6060	0.034*
Br2	0.44176 (4)	0.73105 (4)	0.971236 (9)	0.02871 (8)
C11	0.5297 (4)	0.7870 (3)	0.90906 (9)	0.0205 (5)
C12	0.7254 (4)	0.8675 (3)	0.90274 (9)	0.0218 (5)
C13	0.7768 (4)	0.9044 (3)	0.85612 (10)	0.0243 (6)
H13	0.9102	0.9558	0.8501	0.029*
C14	0.6420 (4)	0.8695 (4)	0.81816 (9)	0.0253 (6)
H14	0.6834	0.8991	0.7869	0.030*
C15	0.4454 (4)	0.7913 (3)	0.82503 (9)	0.0218 (6)
C16	0.3931 (4)	0.7476 (3)	0.87155 (9)	0.0224 (6)

H16	0.2629	0.6906	0.8774	0.027*
C17	0.2894 (4)	0.7613 (4)	0.78658 (9)	0.0250 (6)
H17	0.1761	0.6842	0.7932	0.030*
C18	0.8708 (4)	0.9153 (4)	0.94351 (10)	0.0279 (6)
H18A	0.8014	1.0011	0.9638	0.033*
H18B	1.0011	0.9656	0.9313	0.033*
H18C	0.9047	0.8104	0.9621	0.033*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.02994 (15)	0.03138 (16)	0.01930 (14)	−0.00196 (12)	−0.00088 (10)	0.00574 (11)
C1	0.0243 (14)	0.0192 (13)	0.0157 (12)	0.0034 (11)	0.0018 (10)	0.0006 (10)
C2	0.0207 (13)	0.0217 (14)	0.0226 (13)	0.0025 (11)	−0.0021 (10)	−0.0016 (11)
C3	0.0214 (13)	0.0219 (14)	0.0291 (15)	0.0000 (11)	0.0031 (11)	0.0012 (11)
C4	0.0295 (15)	0.0221 (14)	0.0196 (13)	0.0031 (11)	0.0033 (11)	−0.0001 (11)
C5	0.0252 (14)	0.0196 (13)	0.0190 (13)	0.0025 (11)	0.0001 (11)	−0.0042 (10)
C6	0.0193 (13)	0.0224 (14)	0.0218 (13)	−0.0019 (11)	0.0014 (10)	−0.0017 (11)
C7	0.0253 (14)	0.0256 (15)	0.0213 (14)	0.0010 (11)	0.0001 (11)	−0.0028 (11)
C8	0.0247 (14)	0.0314 (16)	0.0283 (15)	−0.0022 (12)	−0.0052 (11)	−0.0001 (12)
Br2	0.03120 (16)	0.03709 (17)	0.01781 (14)	−0.00261 (12)	−0.00030 (11)	0.00279 (12)
C11	0.0259 (14)	0.0184 (13)	0.0172 (13)	0.0044 (11)	0.0003 (10)	0.0014 (10)
C12	0.0221 (13)	0.0169 (13)	0.0262 (14)	0.0042 (10)	−0.0029 (11)	−0.0014 (11)
C13	0.0192 (13)	0.0218 (14)	0.0321 (15)	0.0008 (11)	0.0027 (11)	0.0016 (11)
C14	0.0298 (15)	0.0249 (14)	0.0213 (14)	0.0026 (12)	0.0030 (11)	0.0018 (11)
C15	0.0251 (14)	0.0185 (13)	0.0217 (13)	0.0028 (11)	−0.0017 (11)	−0.0011 (10)
C16	0.0216 (13)	0.0216 (14)	0.0240 (14)	−0.0008 (11)	−0.0008 (10)	0.0009 (11)
C17	0.0287 (15)	0.0243 (14)	0.0218 (14)	−0.0022 (12)	−0.0013 (11)	−0.0023 (11)
C18	0.0272 (15)	0.0248 (15)	0.0314 (15)	−0.0020 (12)	−0.0052 (12)	−0.0019 (12)

Geometric parameters (Å, °)

Br1—C1	1.904 (2)	Br2—C11	1.898 (3)
C1—C6	1.386 (3)	C11—C16	1.385 (4)
C1—C2	1.393 (4)	C11—C12	1.398 (4)
C2—C3	1.393 (4)	C12—C13	1.389 (4)
C2—C8	1.504 (4)	C12—C18	1.502 (4)
C3—C4	1.378 (4)	C13—C14	1.380 (4)
C3—H3	0.9500	C13—H13	0.9500
C4—C5	1.405 (4)	C14—C15	1.398 (4)
C4—H4	0.9500	C14—H14	0.9500
C5—C6	1.394 (4)	C15—C16	1.399 (4)
C5—C7	1.468 (4)	C15—C17	1.468 (4)
C6—H6	0.9500	C16—H16	0.9500
C7—C17	1.329 (4)	C17—H17	0.9500
C7—H7	0.9500	C18—H18A	0.9800
C8—H8A	0.9800	C18—H18B	0.9800
C8—H8B	0.9800	C18—H18C	0.9800

C8—H8C	0.9800		
C6—C1—C2	122.9 (2)	C16—C11—C12	122.7 (2)
C6—C1—Br1	117.82 (19)	C16—C11—Br2	117.7 (2)
C2—C1—Br1	119.25 (19)	C12—C11—Br2	119.67 (19)
C3—C2—C1	115.9 (2)	C13—C12—C11	115.9 (2)
C3—C2—C8	120.9 (2)	C13—C12—C18	121.4 (2)
C1—C2—C8	123.2 (2)	C11—C12—C18	122.7 (2)
C4—C3—C2	122.6 (3)	C14—C13—C12	122.7 (3)
C4—C3—H3	118.7	C14—C13—H13	118.7
C2—C3—H3	118.7	C12—C13—H13	118.7
C3—C4—C5	120.6 (2)	C13—C14—C15	120.9 (2)
C3—C4—H4	119.7	C13—C14—H14	119.6
C5—C4—H4	119.7	C15—C14—H14	119.6
C6—C5—C4	117.7 (2)	C14—C15—C16	117.5 (2)
C6—C5—C7	119.7 (2)	C14—C15—C17	123.5 (2)
C4—C5—C7	122.6 (2)	C16—C15—C17	119.0 (2)
C1—C6—C5	120.2 (2)	C11—C16—C15	120.4 (2)
C1—C6—H6	119.9	C11—C16—H16	119.8
C5—C6—H6	119.9	C15—C16—H16	119.8
C17—C7—C5	126.6 (3)	C7—C17—C15	126.4 (3)
C17—C7—H7	116.7	C7—C17—H17	116.8
C5—C7—H7	116.7	C15—C17—H17	116.8
C2—C8—H8A	109.5	C12—C18—H18A	109.5
C2—C8—H8B	109.5	C12—C18—H18B	109.5
H8A—C8—H8B	109.5	H18A—C18—H18B	109.5
C2—C8—H8C	109.5	C12—C18—H18C	109.5
H8A—C8—H8C	109.5	H18A—C18—H18C	109.5
H8B—C8—H8C	109.5	H18B—C18—H18C	109.5
Br2—C11—C12—C18	-1.0 (3)	C6—C5—C7—C17	-171.7 (3)
Br1—C1—C2—C8	-1.7 (4)	C16—C15—C17—C7	162.7 (3)
C4—C5—C7—C17	8.2 (4)	C5—C7—C17—C15	-175.4 (2)
C14—C15—C17—C7	-14.1 (4)		
