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1,3-Bis(4-bromophenyl)propane

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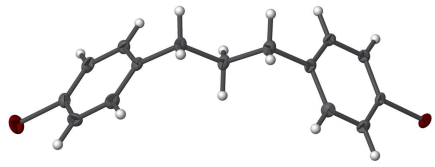
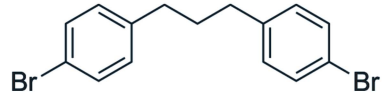
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{15}H_{14}Br_2$, obtained through the reduction of 4,4′-dibromochalcone, has monoclinic $P2_1$ symmetry at 100 K. No directional interactions could be identified in the crystal.

3D view



Chemical scheme



Structure description

The title compound (Fig. 1) crystallizes in the monoclinic space group $P2_1$ with one molecule per asymmetric unit. The 4-bromophenyl substituents are located in the anti positions of the propane linker, with C4—C1—C2—C3 and C1—C2—C3–C10 torsion angles of −174.5 (3) and 179.5 (3)°, respectively. The phenyl rings are oriented in a nearly perpendicular arrangement to the propane chain as shown by the dihedral angles between the C1–C2–C3 plane and the phenyl rings of 74.7 (3)° (C4–C9) and 87.6 (3)° (C10–C15).

Despite the presence of multiple aromatic rings within the molecule, there are no obvious π-stacking interactions due to the kinked arrangement of the propane linker. The only interactions present are typical van der Waals interactions.

A search in the Cambridge Structural Database (CSD, Version 5.38, last update November 2016; Groom *et al.*, 2016) revealed that a structurally similar 1,3-bis(4-bromophenyl)acetone has been reported (Varughese & Draper, 2010)

Synthesis and crystallization

The title compound was prepared *via* a modified literature procedure (Murata *et al.*, 2004). Triethylsilane (14.1 ml, 87.4 mmol) was added dropwise to a stirring suspension of 1,3-bis(4-bromophenyl)-2-propen-1-one (7.99 g, 21.9 mmol) in trifluoroacetic acid

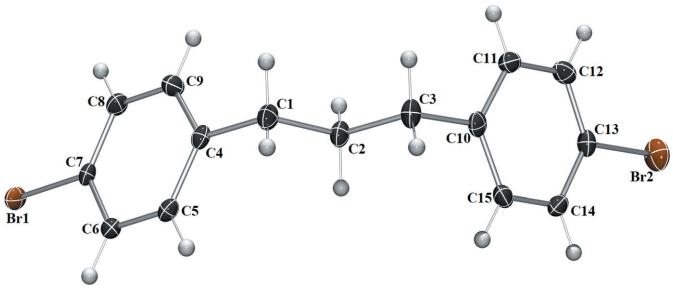


Figure 1
The molecular structure of 1,3-bis(4-bromophenyl)propane. Displacement ellipsoids are shown at the 50% probability level.

(20 ml) under N_2 at 0°C. The reaction mixture was stirred and slowly warmed to room temperature over 18 h. The resulting white precipitate was filtered, taken up in dichloromethane (50 ml), dried over anhydrous $MgSO_4$, filtered, and residual solvent was removed *in vacuo*. The crude, oily product solidified upon standing over 48 h. The waxy solid was recrystallized by dissolving in boiling hexanes (25 ml) and cooling (5°C). Vacuum filtration, washing with cold hexanes (10 ml), and removal of residual solvent *in vacuo* afforded the title compound as a pale yellow solid (4.57 g, 59.1%). Crystals suitable for single-crystal X-ray diffraction were obtained from the slow evaporation of methanol. ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.41 (*d*, 4H, J = 8.0 Hz), 7.05 (*d*, 4H, J = 8.0 Hz), 2.59 (*t*, 4H, J = 7.5 Hz), 1.91 (*p*, 2H, J = 8.0 Hz). ^{13}C NMR (500 MHz, $CDCl_3$): δ 141.0, 131.5, 130.3, 119.7, 34.8, 32.7.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1.

Table 1
Experimental details.

Crystal data	
Chemical formula	$C_{15}H_{14}Br_2$
M_r	354.08
Crystal system, space group	Monoclinic, $P2_1$
Temperature (K)	100
a, b, c (Å)	7.4526 (13), 5.8441 (10), 16.278 (3)
β (°)	101.808 (2)
V (Å^3)	694.0 (2)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	5.82
Crystal size (mm)	0.47 × 0.25 × 0.12
Data collection	
Diffractometer	Bruker SMART APEX CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2017)
T_{min}, T_{max}	0.25, 0.55
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	14936, 3562, 3421
R_{int}	0.034
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.676
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.023, 0.055, 1.38
No. of reflections	3562
No. of parameters	154
No. of restraints	1
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.68, −0.35
Absolute structure	Flack x determined using 1492 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	0.019 (9)

Computer programs: *APEX3* and *SAINT* (Bruker, 2017), *SHELXT* (Sheldrick, 2015*a*), *SHELXL* (Sheldrick, 2015*b*), *ORTEP-3 for Windows* (Farrugia, 2012), *Mercury* (Macrae *et al.*, 2008) and *SHELXL* (Sheldrick, 2008).

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full crystallographic data

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Crystal data

$C_{15}H_{14}Br_2$
M_r = 354.08
Monoclinic, $P2_1$
a = 7.4526 (13) Å
b = 5.8441 (10) Å
c = 16.278 (3) Å
β = 101.808 (2)°
V = 694.0 (2) Å^3
Z = 2
$F(000)$ = 348
D_x = 1.694 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9704 reflections
θ = 2.6–29.7°
μ = 5.82 mm^{-1}
T = 100 K
Flat prism, clear colourless
0.47 × 0.25 × 0.12 mm

Data collection

Bruker SMART APEX CCD diffractometer
Radiation source: fine focus sealed tube
Graphite monochromator
Detector resolution: 8.3333 pixels mm^{-1}
ω Scans scans
Absorption correction: multi-scan (SADABS; Bruker, 2017)
T_{min} = 0.25, T_{max} = 0.55
14936 measured reflections
3562 independent reflections
3421 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 28.7°, θ_{min} = 2.6°
h = −10→10
k = −7→7
l = −21→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.055
S = 1.38
3562 reflections
154 parameters
1 restraint
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2)]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.68 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}
Absolute structure: Flack x determined using 1492 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.019 (9)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. The hydrogen atoms were included in calculated positions and refined with a riding model: C–H = 0.95 and 0.98 Å for aromatic and methyl H atoms, respectively, and and U_{iso}(H) = 1.2 U_{eq}(C-aromatic) and U_{iso}(H) = 1.5 U_{eq}(*C*-methyl).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.72512 (6)	0.64324 (6)	0.32150 (2)	0.02990 (11)
Br2	0.07038 (4)	0.07332 (6)	0.93844 (2)	0.01940 (9)
C1	0.7965 (5)	0.8018 (7)	0.7041 (2)	0.0203 (7)
H1A	0.773435	0.961347	0.714801	0.024*
H1B	0.91894	0.764318	0.734497	0.024*
C2	0.6578 (4)	0.6530 (7)	0.73599 (18)	0.0185 (6)
H2A	0.688304	0.493461	0.72972	0.022*
H2B	0.53722	0.679954	0.701533	0.022*
C3	0.6507 (5)	0.6981 (6)	0.8280 (2)	0.0186 (7)
H3A	0.770709	0.669457	0.862731	0.022*
H3B	0.620835	0.857725	0.834565	0.022*
C4	0.7860 (5)	0.7678 (6)	0.6108 (2)	0.0172 (7)
C5	0.6973 (5)	0.9269 (6)	0.5527 (2)	0.0194 (7)
H5	0.649793	1.05945	0.571618	0.023*
C6	0.6785 (5)	0.8907 (6)	0.4664 (2)	0.0205 (7)
H6	0.619388	0.997936	0.42798	0.025*
C7	0.7494 (5)	0.6922 (6)	0.43923 (19)	0.0191 (7)
C8	0.8384 (5)	0.5303 (6)	0.4947 (2)	0.0203 (8)
H8	0.885231	0.397812	0.475442	0.024*
C9	0.8562 (4)	0.5710 (7)	0.5808 (2)	0.0193 (6)
H9	0.916431	0.463847	0.618886	0.023*
C10	0.5111 (4)	0.5499 (6)	0.85791 (18)	0.0157 (6)
C11	0.5604 (5)	0.3371 (6)	0.8952 (2)	0.0165 (7)
H11	0.68188	0.289513	0.903494	0.02*
C12	0.4309 (4)	0.1948 (6)	0.92003 (19)	0.0162 (7)
H12	0.465409	0.054546	0.945371	0.019*
C13	0.2489 (4)	0.2666 (6)	0.90622 (19)	0.0154 (6)
C14	0.1967 (5)	0.4775 (6)	0.8701 (2)	0.0192 (7)
H14	0.075257	0.525136	0.862057	0.023*
C15	0.3282 (4)	0.6167 (6)	0.84615 (19)	0.0195 (7)
H15	0.293275	0.757895	0.821671	0.023*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0370 (2)	0.0355 (2)	0.01707 (16)	−0.00535 (17)	0.00528 (14)	−0.00373 (15)
Br2	0.01583 (15)	0.01890 (15)	0.02574 (17)	−0.00157 (13)	0.00954 (12)	0.00104 (12)
C1	0.0195 (18)	0.0252 (19)	0.0174 (15)	−0.0039 (14)	0.0069 (14)	−0.0002 (14)
C2	0.0182 (16)	0.0209 (16)	0.0183 (14)	−0.0031 (14)	0.0080 (12)	−0.0013 (14)
C3	0.0183 (17)	0.0195 (19)	0.0196 (15)	−0.0027 (14)	0.0074 (13)	−0.0016 (12)
C4	0.0133 (16)	0.0207 (17)	0.0189 (15)	−0.0030 (13)	0.0063 (13)	0.0019 (13)

C5	0.0191 (18)	0.0160 (16)	0.0243 (17)	0.0020 (13)	0.0076 (14)	0.0006 (13)
C6	0.0187 (17)	0.0202 (18)	0.0215 (17)	0.0020 (14)	0.0016 (14)	0.0052 (14)
C7	0.0194 (17)	0.0236 (19)	0.0152 (14)	−0.0042 (14)	0.0054 (12)	−0.0007 (12)
C8	0.0207 (17)	0.017 (2)	0.0266 (18)	0.0016 (13)	0.0123 (14)	−0.0007 (13)
C9	0.0185 (15)	0.0197 (16)	0.0209 (16)	0.0021 (16)	0.0069 (13)	0.0069 (15)
C10	0.0158 (14)	0.0184 (17)	0.0140 (14)	−0.0026 (13)	0.0055 (12)	−0.0041 (12)
C11	0.0135 (16)	0.0200 (17)	0.0164 (15)	0.0015 (13)	0.0045 (13)	−0.0024 (13)
C12	0.0173 (16)	0.0161 (18)	0.0159 (14)	0.0020 (13)	0.0052 (12)	0.0004 (12)
C13	0.0142 (16)	0.0189 (17)	0.0147 (14)	−0.0004 (12)	0.0070 (12)	−0.0014 (12)
C14	0.0145 (16)	0.0224 (17)	0.0218 (16)	0.0044 (14)	0.0066 (13)	0.0020 (14)
C15	0.0196 (16)	0.0171 (19)	0.0226 (15)	0.0021 (13)	0.0061 (13)	0.0035 (13)

Geometric parameters (Å, º)

Br1—C7	1.909 (3)	C6—C7	1.384 (5)
Br2—C13	1.899 (3)	C6—H6	0.93
C1—C4	1.519 (4)	C7—C8	1.380 (5)
C1—C2	1.521 (5)	C8—C9	1.400 (5)
C1—H1A	0.97	C8—H8	0.93
C1—H1B	0.97	C9—H9	0.93
C2—C3	1.532 (4)	C10—C15	1.392 (4)
C2—H2A	0.97	C10—C11	1.400 (5)
C2—H2B	0.97	C11—C12	1.395 (5)
C3—C10	1.509 (5)	C11—H11	0.93
C3—H3A	0.97	C12—C13	1.393 (4)
C3—H3B	0.97	C12—H12	0.93
C4—C9	1.392 (5)	C13—C14	1.387 (5)
C4—C5	1.393 (5)	C14—C15	1.390 (5)
C5—C6	1.399 (5)	C14—H14	0.93
C5—H5	0.93	C15—H15	0.93
C4—C1—C2	111.5 (3)	C8—C7—C6	121.9 (3)
C4—C1—H1A	109.3	C8—C7—Br1	119.2 (3)
C2—C1—H1A	109.3	C6—C7—Br1	118.8 (3)
C4—C1—H1B	109.3	C7—C8—C9	118.2 (3)
C2—C1—H1B	109.3	C7—C8—H8	120.9
H1A—C1—H1B	108.0	C9—C8—H8	120.9
C1—C2—C3	113.4 (3)	C4—C9—C8	121.7 (3)
C1—C2—H2A	108.9	C4—C9—H9	119.1
C3—C2—H2A	108.9	C8—C9—H9	119.1
C1—C2—H2B	108.9	C15—C10—C11	118.1 (3)
C3—C2—H2B	108.9	C15—C10—C3	121.0 (3)
H2A—C2—H2B	107.7	C11—C10—C3	120.8 (3)
C10—C3—C2	112.4 (3)	C12—C11—C10	121.3 (3)
C10—C3—H3A	109.1	C12—C11—H11	119.4
C2—C3—H3A	109.1	C10—C11—H11	119.4
C10—C3—H3B	109.1	C13—C12—C11	118.8 (3)
C2—C3—H3B	109.1	C13—C12—H12	120.6

H3A—C3—H3B	107.9	C11—C12—H12	120.6
C9—C4—C5	118.3 (3)	C14—C13—C12	121.0 (3)
C9—C4—C1	120.9 (3)	C14—C13—Br2	119.6 (3)
C5—C4—C1	120.8 (3)	C12—C13—Br2	119.4 (2)
C4—C5—C6	121.1 (3)	C13—C14—C15	119.1 (3)
C4—C5—H5	119.4	C13—C14—H14	120.4
C6—C5—H5	119.4	C15—C14—H14	120.4
C7—C6—C5	118.8 (3)	C14—C15—C10	121.6 (3)
C7—C6—H6	120.6	C14—C15—H15	119.2
C5—C6—H6	120.6	C10—C15—H15	119.2