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1-Bromo-3,4-bis(bromomethyl)-2,5-dimethoxybenzene

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1,4-Dimethoxy-2,3-dimethylbenzene was brominated under radical conditions to give the title compound, $C_{10}H_{11}Br_3O_4$. The bond lengths and angles are generally within the normal ranges. The crystal packing is stabilized by weak intermolecular C—H···O and C—H···Br hydrogen bonds.

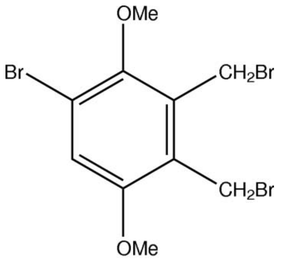
Key indicators

Single-crystal X-ray study
T = 122 K
Mean σ(C–C) = 0.004 Å
R factor = 0.041
wR factor = 0.078
Data-to-parameter ratio = 39.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

The title compound, (I), was prepared for use as a building block in the syntheses of nanotube interacting compounds. It crystallizes in the monoclinic space group $P2_1/c$ with two independent molecules in the asymmetric unit (Fig. 1). The bond lengths and angles in both independent molecules (Table 1) are generally within normal ranges.



(I)

In the crystal structure, there are weak intermolecular C—H···O (H7*AA*···O2*B* = 2.63 Å and H10*BB*···O1*A* = 2.66 Å) and C—H···Br (H10*AB*···Br2*A* = 2.89 Å) hydrogen bonds, which stabilize the crystal packing (Fig. 2).

Experimental

1,4-Dimethoxy-2,3-dimethylbenzene was prepared as described by Eskildsen *et al.* (2000). 1,4-Dimethoxy-2,3-dimethylbenzene (166.22 g, 1 mol) was dissolved in CCl_4 (4 l) and *N*-bromosuccinimide (711.96 g, 4 mol) was added. The reaction mixture was mechanically stirred and irradiated with a 500 W halogen lamp. The heat from the

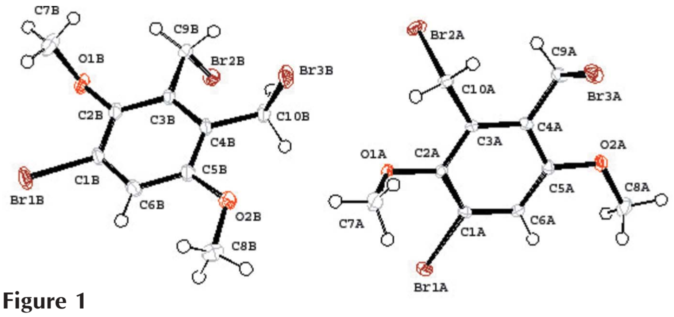


Figure 1
The structure of the asymmetric unit of (I) with 50% probability displacement ellipsoids. H atoms are shown as spheres of arbitrary radius.

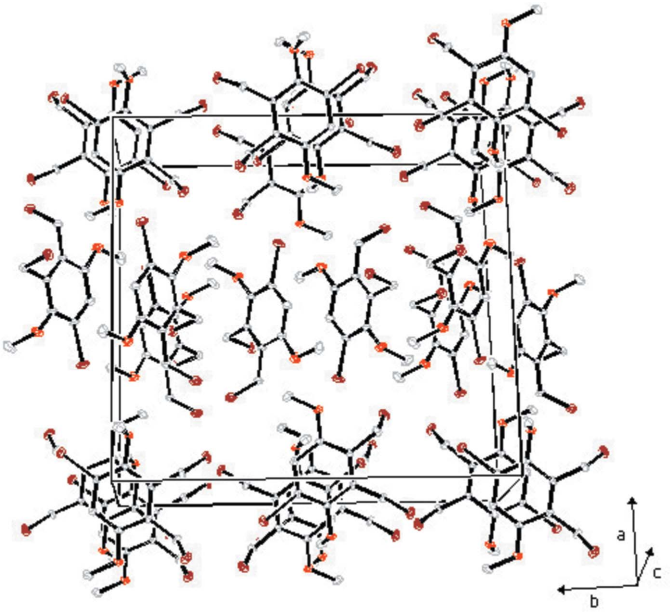


Figure 2
The crystal packing, viewed approximately along the *c* axis. H atoms have been omitted for clarity.

lamp made the reaction mixture reflux. After 8 h of reflux the mixture was filtered while hot to remove the precipitated succinimide. The solvent was removed *in vacuo*. Crystallization from EtOH yielded the title compound as a white powder (yield 205 g, 51.1%, m.p. 357–359 K); ^{1}H NMR (300 MHz, CDCl$_3$): δ 7.10 (1H, *s*), 4.65 (2H, *s*), 4.62(2H, *s*), 3.87 (3H, *s*), 3.70 (3H, *s*); ^{13}C NMR (100 MHz, CDCl$_3$): δ 154.2, 148.8, 132.8, 125.8, 118.1, 116.4, 61.5, 56.3, 23.3, 23.2; MS (EI+): 404 (9), 323 (100) and 227 (44%). Analysis calculated for $C_{10}H_{11}Br_3O_4$: C 29.95, H 2.49%; found: C 29.81, H 2.75%.

Crystal data

$C_{10}H_{11}Br_3O_2$
$M_r = 402.91$
Monoclinic, $P2_1/c$
$a = 16.5660$ (9) Å
$b = 17.1420$ (16) Å
$c = 8.6720$ (12) Å
$\beta = 98.672$ (7)°
$V = 2434.5$ (4) Å^3
$Z = 8$
$D_x = 2.199$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 47424 reflections
$\theta = 1.2$–35.0°
$\mu = 9.92$ mm^{-1}
$T = 122$ (2) K
Prism, white
0.36 × 0.26 × 0.16 mm

Data collection

Nonius KappaCCD area-detector diffractometer
ω and φ scans
Absorption correction: Gaussian integration (Coppens, 1970)
$T_{\min} = 0.195$, $T_{\max} = 0.419$
106756 measured reflections
10710 independent reflections
8305 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.057$
$\theta_{\max} = 35.0°$
$h = -26 \rightarrow 26$
$k = -27 \rightarrow 27$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.078$
$S = 1.16$
10710 reflections
271 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0187P)^2 + 7.2256P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 1.33$ e Å^{-3}
$\Delta\rho_{\min} = -1.14$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Br1*A*—C1*A*	1.884 (2)	C6*A*—C1*A*	1.385 (3)
Br2*B*—C9*B*	1.979 (3)	C6*A*—C5*A*	1.390 (4)
Br2*A*—C10*A*	1.962 (3)	C10*A*—C3*A*	1.489 (4)
Br3*A*—C9*A*	1.977 (3)	O2*B*—C5*B*	1.360 (3)
Br3*B*—C10*B*	1.972 (3)	O2*B*—C8*B*	1.427 (4)
Br1*B*—C1*B*	1.888 (3)	C9*B*—C3*B*	1.494 (4)
O1*A*—C2*A*	1.373 (3)	C3*B*—C2*B*	1.403 (4)
O1*A*—C7*A*	1.442 (3)	C3*B*—C4*B*	1.397 (4)
O1*B*—C2*B*	1.373 (3)	O2*A*—C5*A*	1.359 (3)
O1*B*—C7*B*	1.438 (4)	O2*A*—C8*A*	1.428 (3)
C2*A*—C1*A*	1.392 (4)	C10*B*—C4*B*	1.490 (4)
C2*A*—C3*A*	1.401 (4)	C1*B*—C2*B*	1.384 (4)
C4*A*—C5*A*	1.403 (4)	C1*B*—C6*B*	1.393 (4)
C4*A*—C3*A*	1.404 (3)	C6*B*—C5*B*	1.388 (4)
C4*A*—C9*A*	1.490 (4)	C4*B*—C5*B*	1.405 (4)

C2*A*—O1*A*—C7*A*	112.4 (2)	C2*B*—C1*B*—C6*B*	121.6 (2)
C2*B*—O1*B*—C7*B*	114.0 (2)	C2*B*—C1*B*—Br1*B*	119.8 (2)
O1*A*—C2*A*—C1*A*	121.4 (2)	C6*B*—C1*B*—Br1*B*	118.6 (2)
O1*A*—C2*A*—C3*A*	119.6 (2)	C5*B*—C6*B*—C1*B*	118.8 (3)
C1*A*—C2*A*—C3*A*	119.0 (2)	C6*A*—C1*A*—C2*A*	121.7 (2)
C5*A*—C4*A*—C3*A*	119.1 (2)	C6*A*—C1*A*—Br1*A*	119.15 (19)
C5*A*—C4*A*—C9*A*	119.9 (2)	C2*A*—C1*A*—Br1*A*	119.14 (19)
C3*A*—C4*A*—C9*A*	121.0 (2)	O1*B*—C2*B*—C1*B*	121.3 (2)
C4*A*—C9*A*—Br3*A*	111.76 (18)	O1*B*—C2*B*—C3*B*	119.3 (3)
C1*A*—C6*A*—C5*A*	119.1 (2)	C1*B*—C2*B*—C3*B*	119.3 (3)
C3*A*—C10*A*—Br2*A*	111.47 (18)	C3*B*—C4*B*—C5*B*	119.2 (2)
C2*A*—C3*A*—C4*A*	120.3 (2)	C3*B*—C4*B*—C10*B*	122.0 (3)
C2*A*—C3*A*—C10*A*	118.1 (2)	C5*B*—C4*B*—C10*B*	118.8 (2)
C4*A*—C3*A*—C10*A*	121.6 (2)	O2*B*—C5*B*—C6*B*	123.9 (3)
C5*B*—O2*B*—C8*B*	117.9 (2)	O2*B*—C5*B*—C4*B*	115.1 (2)
C3*B*—C9*B*—Br2*B*	109.76 (19)	C6*B*—C5*B*—C4*B*	121.0 (3)
C2*B*—C3*B*—C4*B*	120.1 (3)	O2*A*—C5*A*—C6*A*	123.6 (2)
C2*B*—C3*B*—C9*B*	118.0 (2)	O2*A*—C5*A*—C4*A*	115.6 (2)
C5*A*—O2*A*—C8*A*	117.1 (2)	C6*A*—C5*A*—C4*A*	120.8 (2)
C4*B*—C10*B*—Br3*B*	111.07 (19)		

H atoms were placed in geometrically idealized positions and allowed to ride on their parent atoms, with C—H = 0.95–0.99 Å and $U_{\rm iso}$(H) = 1.2–1.5$U_{\rm eq}$(C). The maximum positive and minimum negative residual electron-density peaks are situated 0.77 Å from atom Br3*B* and 0.57 Å from Br1*B*.

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *DIRAX* (Duisenberg, 1992); data reduction: *EVALCCD* (Duisenberg *et al.*, 2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97*.

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