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1,5-Dibromo-2,6-dimethylnaphthalene

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Key indicators

Single-crystal X-ray study
T = 133 K
Mean σ(C–C) = 0.003 Å
R factor = 0.024
wR factor = 0.054
Data-to-parameter ratio = 24.6

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

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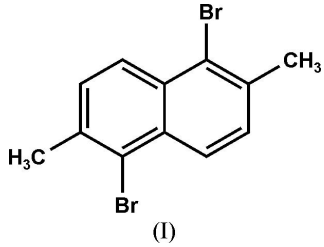
The molecule of the title compound, $C_{12}H_{10}Br_2$, displays crystallographic inversion symmetry and is essentially planar. Bond lengths show the typical naphthalene pattern. The crystal packing shows no short H···Br, Br···Br or C—H···π contacts.

Comment

The electrophilic bromination of 2,6-dimethylnaphthalene in tetrachloromethane in the presence of iron leads to the title compound, (I), in very good yield (*ca* 90%). This compound is also present (as a contaminant) on monobromination of 2,6-dimethylnaphthalene (Veselý & Štursa, 1932; Gore & Yusuf, 1971). It has been used as a starting material for the synthesis of naphthodifuran, which can be used in Diels–Alder reactions or to prepare chrysene derivatives and some interesting cyclophanes (Thibault *et al.*, 2003; Blank & Haenel, 1983). Its spectroscopic characterization was described by Casarini *et al.* (1991). In view of our interest in the structure of bromine-substituted naphthalenes (unpublished results), we decided to determine the crystal structure.



(I)

The molecule is shown in Fig. 1; it displays crystallographic inversion symmetry and is thus essentially planar except for the methyl H atoms. Bond lengths and angles may be regarded as normal, *e.g.* the typical naphthalene bond-length pattern.

The crystal packing (Fig. 2) is surprisingly devoid of short contacts. The shortest H···Br contact is H3···Br(x, $1-y$, $\frac{1}{2}+z$) of 3.20 Å and the shortest Br···Br contact is given by the b-axis repeat of 4.01 Å. There are no short C—H···π interactions. Adjacent molecules in Fig. 2 are displaced in height with respect to each other and subtend an interplanar angle of 55.88 (6)°.

Experimental

The title compound was synthesized, starting from 2,6-dimethylnaphthalene, according to the method of Veselý & Štursa (1932). It crystallizes well from the reaction mixture, but as needles unsuitable for structure determination. The analytical and spectroscopic data are consistent with the literature (Casarini *et al.*, 1991). Single crystals

were grown by slow evaporation of a chloroform solution. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.18 (*d*, 2H, J = 8.4 Hz), 7.40 (*d*, 2H, J = 8.4 Hz), 2.62 (*s*, 6H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 24.13, 124.09, 126.49, 129.92, 132.09, 136.02.

Crystal data

$C_{12}H_{10}Br_2$
M_r = 314.02
Monoclinic, $C2/c$
a = 20.543 (3) Å
b = 4.0082 (8) Å
c = 12.813 (2) Å
β = 95.497 (6)°
V = 1050.2 (3) Å^3
Z = 4
D_x = 1.986 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4270 reflections
θ = 2.7–30.7°
μ = 7.67 mm^{-1}
T = 133 (2) K
Prism, colourless
0.22 × 0.11 × 0.08 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.383, T_{max} = 0.541
8223 measured reflections
1597 independent reflections
1327 reflections with $I > 2\sigma(I)$
R_{int} = 0.028
θ_{max} = 30.5°
$h = -28 \rightarrow 28$
$k = -5 \rightarrow 5$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.054
S = 1.08
1597 reflections
65 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0241P)^2 + 1.2229P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.54 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Br—C1	1.9141 (19)	C3—C4	1.371 (3)
C1—C2	1.379 (3)	C4—C5	1.423 (3)
C1—C5^{i}	1.426 (3)	C5—C5^{i}	1.431 (4)
C2—C3	1.420 (3)		
C2—C1—C5^{i}	123.59 (18)	C5^{i}—C1—Br	118.40 (14)
C2—C1—Br	118.01 (15)		

Symmetry code: (i) $\frac{1}{2} - x, \frac{1}{2} - y, 1 - z$.

Methyl H atoms were identified in difference syntheses, idealized and then refined using a rigid methyl group (C—H = 0.98 Å and H—C—H = 109.5°) allowed to rotate but not tip. Other H atoms were included using a riding model, with C—H = 0.95 Å. U_{iso}(H) values were fixed at 1.2U_{eq}(C) of the parent C atom.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL97*.

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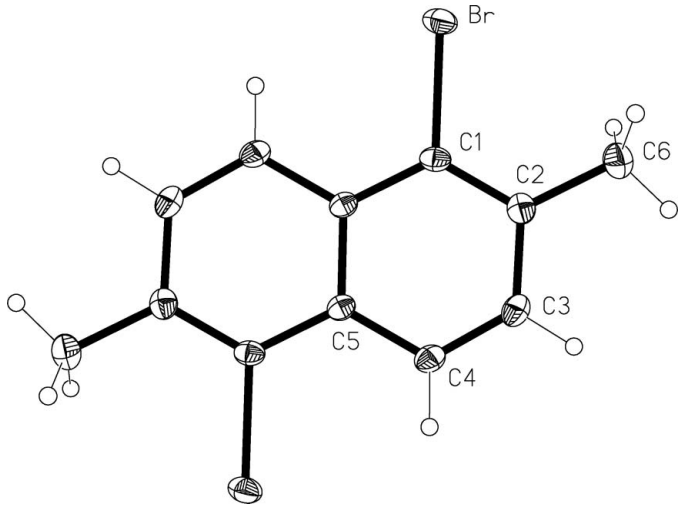

Figure 1
The molecule of the title compound in the crystal structure. Displacement ellipsoids are drawn at the 50% probability level. H-atom radii are arbitrary. [Symmetry code: (i) $\frac{1}{2} - x, \frac{1}{2} - y, 1 - z$.]

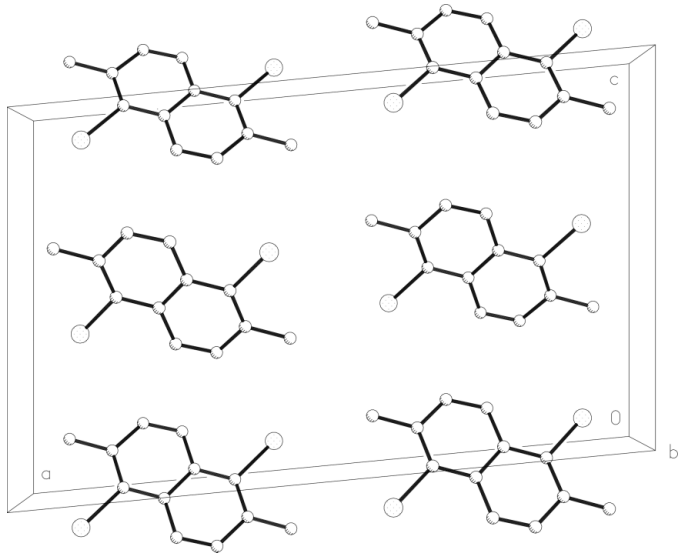

Figure 2
Packing of the title compound, viewed parallel to the short b axis. H atoms have been omitted.

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