1,6,7-Tris(bromomethyl)naphthalene

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The title compound, $C_{13}H_{11}Br_3$, has a planar framework of C atoms with no distortion towards helicity. The two adjacent bromine substituents point to opposite sides of the ring system. Two bromine–bromine contacts, with distances less than twice the van der Waals radius, link the molecules to form ribbons parallel to the a axis. Weak C—H$\cdots$Br hydrogen bonds and partial ring stacking complete the crystal packing.
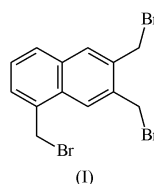
Comment

The title compound, (I), was synthesized as a potential precursor to naphthalenophanes (see, for example, Kuś & Jones, 2000).

(I)

The molecular structure of (I) is presented in Fig. 1. Bond lengths and angles may be regarded as normal. The naphthalene framework is planar (r.m.s. deviation 0.004 Å for all 11 C atoms); the level of substitution is thus not great enough to promote helicity, in contrast to the octakis-substituted analogue (Siman *et al.*, 2003). The C—Br groups are approximately perpendicular to the ring system (torsion angles in Table 1), with atoms Br1 and Br2 on one side of the plane, Br3 on the other.

The crystal packing involves two main types of interaction, weak C—H$\cdots$Br hydrogen bonds and Br$\cdots$Br interactions. Details of the H bonds are given in Table 2; none of the H$\cdots$Br distances is especially short, and two have narrow angles (<120°) at hydrogen. The Br$\cdots$Br contacts, Br1$\cdots$Br1^v 3.6974 (8) and Br2$\cdots$Br3^{vi} 3.5591 (6) Å [symmetry codes: (v) $-x, 2 - y, 1 - z$; (vi) $-1 + x, y, z$] may be compared with twice the van der Waals radius, 3.7 Å (Bondi, 1964). There is also a longer contact Br1$\cdots$Br3^i of 3.9048 (6) Å. The first two contacts are established as 'type II' and the third as 'type I', in the classification of Pedireddi *et al.* (1994), by the angles at bromine: 155.30 (10)° ($\times$ 2, by symmetry), 156.59 (10) and 147.56 (9)°, and 84.90 (10) and 142.03 (9)°, respectively.

The effect of the two shortest Br$\cdots$Br contacts is to link the molecules to form highly corrugated ribbons (Fig. 2), with overall direction parallel to the a axis. The hydrogen bonds (not shown) then link the ribbons in the c direction to establish the final three-dimensional packing, which also involves partial stacking (incomplete overlap in projection) of the ring systems.

Experimental

The title material was synthesized from 1,6,7-trimethylnaphthalene (Ried & Bodem, 1958) and recrystallized from ethanol/dichloromethane (1:2 v/v). ^{1}H NMR data (300 MHz, CDCl$_3$, TMS, p.p.m.): δ 4.87 (s, 2H), 4.91 (s, 2H), 4.92 (s, 2H), 7.43 (t, 1H), 7.55 (d, 1H), 7.79 (d, 1H), 7.89 (s, 1H) and 8.14 (s, 1H).

Crystal data

C$_{13}$H$_{11}$Br$_3$	$D_x = 2.109$ Mg m^{-3}
$M_r = 406.95$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 7411
$a = 7.7316\,(6)$ Å	reflections
$b = 9.3716\,(8)$ Å	$\theta = 2.4$–30.5°
$c = 17.7245\,(14)$ Å	$\mu = 9.41$ mm^{-1}
$\beta = 93.835\,(3)$°	$T = 133\,(2)$ K
$V = 1281.40\,(18)$ Å^3	Tablet, colourless
$Z = 4$	$0.18 \times 0.16 \times 0.08$ mm

Data collection

Bruker SMART 1000 CCD diffractometer	3737 independent reflections
ω and φ scans	2842 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan	$R_{int} = 0.092$
(SADABS; Bruker, 1998)	$\theta_{max} = 30.0$°
$T_{min} = 0.199$, $T_{max} = 0.471$	$h = -10 \rightarrow 10$
23006 measured reflections	$k = -13 \rightarrow 12$
	$l = -24 \rightarrow 24$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.037$	$w = 1/[\sigma^2(F_o^2) + (0.0518P)^2]$
$wR(F^2) = 0.094$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.97$	$(\Delta/\sigma)_{max} = 0.001$
3737 reflections	$\Delta\rho_{max} = 1.56$ e Å^{-3}
145 parameters	$\Delta\rho_{min} = -0.69$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Br1—C11	1.971 (3)	Br3—C13	1.986 (3)
Br2—C12	1.979 (3)		
C1—C11—Br1	110.9 (2)	C7—C13—Br3	111.6 (2)
C6—C12—Br2	112.5 (2)		
C2—C1—C11—Br1	93.4 (3)	C6—C7—C13—Br3	75.4 (3)
C7—C6—C12—Br2	72.9 (4)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C8—H8$\cdots$Br3^i	0.95	3.11	4.013 (3)	160
C11—H11$A\cdots$Br3^{ii}	0.99	3.02	3.908 (3)	150
C13—H13$B\cdots$Br2^{iii}	0.99	3.11	3.643 (3)	115
C12—H12$B\cdots$Br3^{iv}	0.99	3.09	3.672 (3)	119

Symmetry codes: (i) $1-x, \frac{1}{2}+y, \frac{3}{2}-z$; (ii) $x, \frac{3}{2}-y, z-\frac{1}{2}$; (iii) $-x, \frac{1}{2}+y, \frac{3}{2}-z$; (iv) $1-x, y-\frac{1}{2}, \frac{3}{2}-z$.

H atoms were included using a riding model with fixed C—H bond lengths of 0.95 (sp^2 carbon) or 0.99 Å (methylene). U_{iso}(H) values were fixed at $1.2U_{eq}$ of the parent atom. The largest maximum is 0.93 Å from Br2; all maxima > 1 e Å^{-3} are within 1 Å of Br atoms.
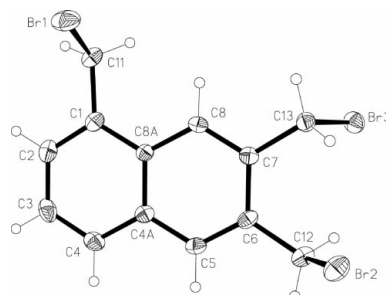

Figure 1
The molecule of the title compound in the crystal. Displacement ellipsoids are drawn at the 50% probability level. The H-atom radius is arbitrary.
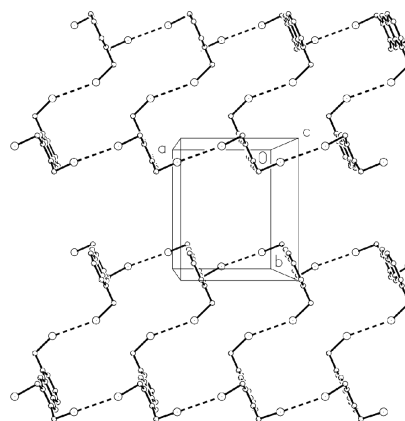

Figure 2
Packing diagram of the title compound, viewed perpendicular to the *ab* plane. Br$\cdots$Br interactions are indicated by dashed lines. H atoms have been omitted.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL*97.

References

Bondi, A. (1964). *J. Phys. Chem.* **68**, 441–451.
Bruker (1998). *SMART* (Version 5.0), *SAINT* (Version 4.0) and *SADABS* (Version 2.0). Bruker AXS Inc., Madison, Wisconsin, USA.
Kuś, P. & Jones, P. G. (2000). *Pol. J. Chem.* **74**, 649–657.
Pedireddi, V. R., Reddy, D. S., Goud, B. S., Craig, D. C., Rae, A. D. & Desiraju, G. R. (1994). *J. Chem. Soc. Perkin Trans.* 2, pp. 2353–2360.
Ried, W. & Bodem, H. (1958). *Chem. Ber.* **91**, 1981–1982.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Siemens (1994). *XP*. Version 5.03. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Siman, S., Marks, V., Gottlieb, H. E., Stranger, A. & Biali, S. E. (2003). *J. Org. Chem.* **68**, 637–640.