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2,4,6-Tris(bromomethyl)-1,3,5-trimethylbenzene

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Key indicators

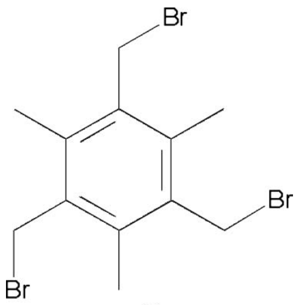
Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.005 Å
R factor = 0.028
wR factor = 0.069
Data-to-parameter ratio = 20.6

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

The title compound, $C_{12}H_{15}Br_3$, has been synthesized and its structure is reported here. The bulky Br atoms are accommodated above and below the plane of the benzene ring.

Comment

Although the synthesis of the title compound, (I), has been reported previously (Závada *et al.*, 1994), the crystal structure had not been determined. The compound is an important precursor in the synthesis of tripodal and hexapodal ligands, the latter requiring further bromination to furnish hexakis(bromomethyl)benzene.



(I)

The bulky Br atoms are accommodated both above and below the plane of the benzene ring, with two bromine substituents on one side of the plane and one on the other (Fig. 1). The C—Br distances are summarized in Table 1, all being in the range 1.979 (3)–1.982 (3) Å. These bond lengths lie towards the longer end of the distribution when compared with 1.97 (3) Å reported by Allen *et al.* (1987) as the mean length for similar bonds. Presumably this is due to the steric crowding around the benzene ring.

The molecules stack along [100] with their ring planes at an angle of 49.9° with respect to this direction (Fig. 2). Adjacent molecules are related by a centre of symmetry and in this way there are collectively either two or four Br atoms between adjacent molecules in the stack. The Br atoms themselves form planes between the bands defined by the aromatic ring stacks (Fig. 2). These planes in which the Br atoms lie are parallel to the *ac* plane.

Experimental

The title compound was synthesized by a modification of the method of Závada *et al.* (1994). The crystals form as colourless blocks upon slow evaporation of a solution of 1,2-dichloroethane. In addition to the crystal structure, the compound was also characterized by ^{1}H

NMR [($CDCl_3$, 300 MHz): δ 4.57 (*s*, 6H, CH_2), 2.46 (*s*, 9H, CH_3)] and the melting point was determined to be 457–458 K (literature 459–461 K; Závada *et al.*, 1976).

Crystal data

$C_{12}H_{15}Br_3$
$M_r = 398.97$
Triclinic, $P\bar{1}$
$a = 9.3961$ (16) Å
$b = 9.4512$ (16) Å
$c = 9.4846$ (16) Å
$\alpha = 117.452$ (2)°
$\beta = 109.115$ (2)°
$\gamma = 101.278$ (3)°

$V = 643.08$ (19) Å^3
$Z = 2$
$D_x = 2.060$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 9.38$ mm^{-1}
$T = 173$ (2) K
Block, colourless
$0.60 \times 0.30 \times 0.24$ mm

Data collection

Bruker APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{min} = 0.027$, $T_{max} = 0.106$

7135 measured reflections
2864 independent reflections
2471 reflections with $I > 2\sigma(I)$
$R_{int} = 0.038$
$\theta_{max} = 28.2°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.069$
$S = 1.04$
2864 reflections
139 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0264P)^2 + 0.3185P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.80$ e Å^{-3}
$\Delta\rho_{min} = -0.71$ e Å^{-3}

Table 1
Selected bond lengths (Å).

Br1—C7	1.980 (3)	Br3—C11	1.979 (3)
Br2—C9	1.982 (3)		

H atoms were refined in calculated positions, using a riding model [C—H = 0.98–0.99 Å, and $U_{iso}(H) = 1.5U_{eq}$(methyl C) or $1.2U_{eq}$(other C)].

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001; Atwood & Barbour, 2003); software used to prepare material for publication: *publCIF* (Westrip, 2006).

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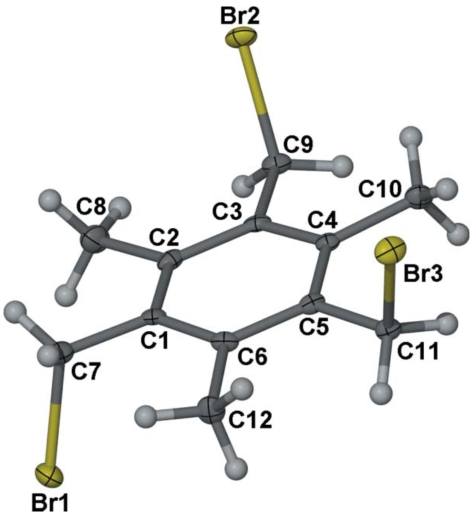

Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

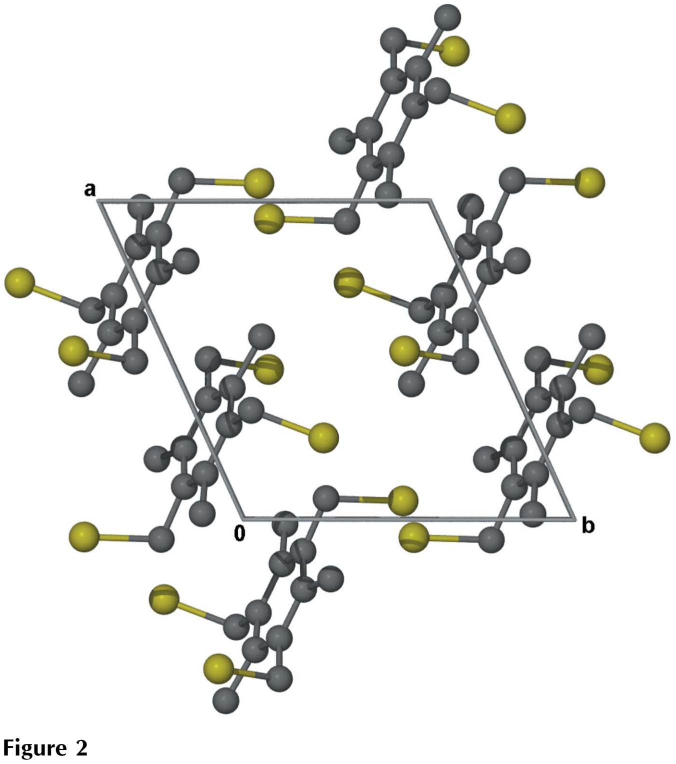

Figure 2
Packing diagram for (I), viewed along [001], showing the Br atoms lying in planes (here viewed end on) that are parallel to the *ac* plane. H atoms have been omitted for clarity.

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