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1,3,4,6,7,9-Hexabromo-2,3,4,5,6,7,8,9-octahydro-1*H*-trindene

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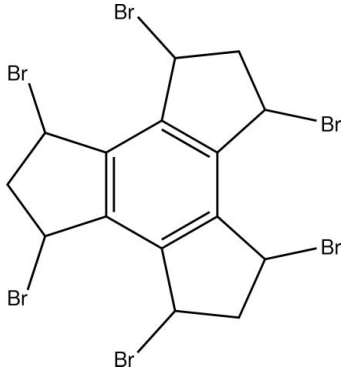
Key indicators: single-crystal X-ray study; T = 123 K; mean σ(C–C) = 0.010 Å; R factor = 0.046; wR factor = 0.114; data-to-parameter ratio = 16.0.

Each of the six Br atoms in the title compound, $C_{15}H_{12}Br_6$, connects to an α-C atom; a pair of adjacent Br atoms connecting to the same five-membered ring extends to one side of the molecular framework and the other two pairs to the other side. The β-C atoms extend in the opposite direction with respect to the pairs of Br atoms.

Related literature

For related literature, see: Ferrier *et al.* (2000); Katz & Slusarek (1980); Ranganathan *et al.* (1998).



Experimental

Crystal data

$C_{15}H_{12}Br_6$
M_r = 671.71
Monoclinic, $P2_1/n$
a = 9.907 (3) Å
b = 9.712 (2) Å
c = 18.010 (5) Å
β = 95.436 (5)°
V = 1725.0 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 13.96 mm^{-1}
T = 123 (2) K
0.3 × 0.2 × 0.2 mm

Data collection

Bruker SMART APEX 2000 CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
T_{min} = 0.102, T_{max} = 0.167
(expected range = 0.038–0.061)
8387 measured reflections
3031 independent reflections
2635 reflections with $I > 2\sigma(I)$
R_{int} = 0.048

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.114
S = 1.05
3031 reflections
190 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.76 e Å^{-3}

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ER2037).

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Ranganathan, S., Muraleedharan, K. M., Bharadwaj, P. & Madhusudananc, K. P. (1998). *Chem. Commun.* pp. 2239–2240.
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supplementary materials

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1,3,4,6,7,9-Hexabromo-2,3,4,5,6,7,8,9-octahydro-1*H*-trindene

S.-J. Zhou, S.-Y. Xie, R.-B. Huang and L.-S. Zheng

Comment

Trindane and its deratives play important roles in a lot of organic processes, for example, as precusor for synthesizing trindene trianion which is an anion ligand for bis(trindene)triiron, a special ferrocene with three iron atoms (Katz & Slusarek, 1980), as starting reagent of fullerene synthesis (Ferrier *et al.*, 2000) and in the other process(Ranganathan *et al.*, 1998).

The title compound, one of the tridane deratives, was synthesized and characterized by Katz & Slusarek (1980), but its crystal structure determination has not been carried out yet. In our organic synthesis of fullerene, we obtained single crystals of the compound and here we report its crystal structure.

There are six substituent bromine atoms in the molecule(Scheme). It may have a lot of isomers due to the bromine position and orientation. However, in the crystal, only one configuration of the molecule(Figure I) packs into solid. Every bromine atom connects to each α-carbon atom. a pair of adjacent bromine atoms extend to one side and the other two pairs to the other side of the framework. The bond lengths and bond angles of the compound remain the normal values.

Experimental

The title compound was prepared according to the literature (Katz & Slusarek, 1980). The colorless single crystals suitable for X-ray diffraction are crystallized from THF-ether(5:2,*v*/*v*) at room temperature in ten days.

Refinement

All H atoms were placed geometrically with C—H distances of 0.95 Å, N—H distances of 0.88Å and refined using a riding model with U_{iso} = 1.2 U_{eq}(C or N) of the parent N and phenyl C atom, at U_{iso} = 1.5 U_{eq}(C) of methyl C.

Figures

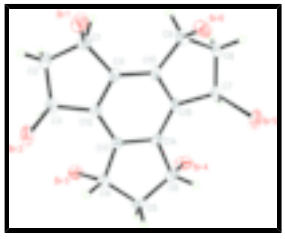


Fig. 1. *ORTEP* plot of the compound. The displacement ellipsoids are drawn at 50% probability level.

1,3,4,6,7,9-Hexabromo-2,3,4,5,6,7,8,9-octahydro-1*H*-trindene

Crystal data

$C_{15}H_{12}Br_6$	F_{000} = 1248
M_r = 671.71	D_x = 2.586 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 2951 reflections
a = 9.907 (3) Å	θ = 2.9–27.7º
b = 9.712 (2) Å	μ = 13.96 mm^{-1}
c = 18.010 (5) Å	T = 123 (2) K
β = 95.436 (5)º	Prism, colourless
V = 1725.0 (8) Å^3	0.3 × 0.2 × 0.2 mm
Z = 4	

Data collection

Bruker SMART APEX 2000 CCD diffractometer	3031 independent reflections
Radiation source: fine-focus sealed tube	2635 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.048
T = 123(2) K	θ_{max} = 25.0º
ω scans	θ_{min} = 2.3º
Absorption correction: multi-scan (SADABS; Sheldrick, 2002)	h = −11→11
T_{min} = 0.102, T_{max} = 0.167	k = −11→11
8387 measured reflections	l = −16→21

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.046	H-atom parameters constrained
$wR(F^2)$ = 0.114	$w = 1/[\sigma^2(F_o^2) + 9.062P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.05	$(\Delta/\sigma)_{max} < 0.001$
3031 reflections	$\Delta\rho_{max}$ = 1.48 e Å^{-3}
190 parameters	$\Delta\rho_{min}$ = −0.76 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.16349 (8)	0.32182 (8)	0.04252 (4)	0.0277 (2)
Br2	0.09197 (8)	0.71829 (7)	0.17843 (4)	0.0258 (2)
Br3	0.03665 (8)	0.62691 (8)	0.39563 (5)	0.0300 (2)
Br4	0.06590 (9)	0.15916 (8)	0.44210 (4)	0.0306 (2)
Br5	−0.10614 (8)	−0.05081 (7)	0.28611 (4)	0.0272 (2)
Br6	−0.10332 (9)	0.03306 (7)	0.07974 (4)	0.0300 (2)
C1	−0.1293 (8)	0.3252 (7)	0.0519 (4)	0.0232 (16)
H1A	−0.1164	0.2798	0.0038	0.028*
H1B	−0.2048	0.3920	0.0437	0.028*
C2	−0.0001 (7)	0.3976 (7)	0.0825 (4)	0.0199 (15)
H2A	−0.0072	0.4987	0.0726	0.024*
C3	0.0076 (7)	0.3705 (6)	0.1638 (4)	0.0148 (14)
C4	0.0860 (7)	0.4371 (7)	0.2216 (4)	0.0163 (14)
C5	0.1874 (7)	0.5481 (6)	0.2182 (4)	0.0179 (15)
H5A	0.2587	0.5202	0.1853	0.021*
C6	0.2493 (7)	0.5680 (7)	0.2975 (4)	0.0204 (15)
H6A	0.2563	0.6673	0.3097	0.024*
H6B	0.3411	0.5271	0.3041	0.024*
C7	0.1545 (7)	0.4954 (7)	0.3481 (4)	0.0194 (15)
H7A	0.2078	0.4380	0.3866	0.023*
C8	0.0664 (7)	0.4060 (6)	0.2953 (4)	0.0157 (14)
C9	−0.0268 (7)	0.3070 (6)	0.3100 (4)	0.0159 (14)
C10	−0.0752 (7)	0.2678 (7)	0.3840 (4)	0.0203 (15)
H10A	−0.0969	0.3523	0.4123	0.024*
C11	−0.2034 (8)	0.1846 (7)	0.3634 (4)	0.0222 (16)
H11A	−0.2095	0.1067	0.3984	0.027*
H11B	−0.2848	0.2432	0.3651	0.027*
C12	−0.1929 (8)	0.1320 (6)	0.2849 (4)	0.0213 (16)
H12A	−0.2835	0.1309	0.2552	0.026*
C13	−0.0984 (7)	0.2346 (6)	0.2537 (4)	0.0154 (14)
C14	−0.0814 (7)	0.2663 (6)	0.1797 (4)	0.0175 (14)
C15	−0.1603 (7)	0.2185 (7)	0.1101 (4)	0.0206 (15)
H15A	−0.2594	0.2187	0.1166	0.025*

Atomic displacement parameters (Å^2)

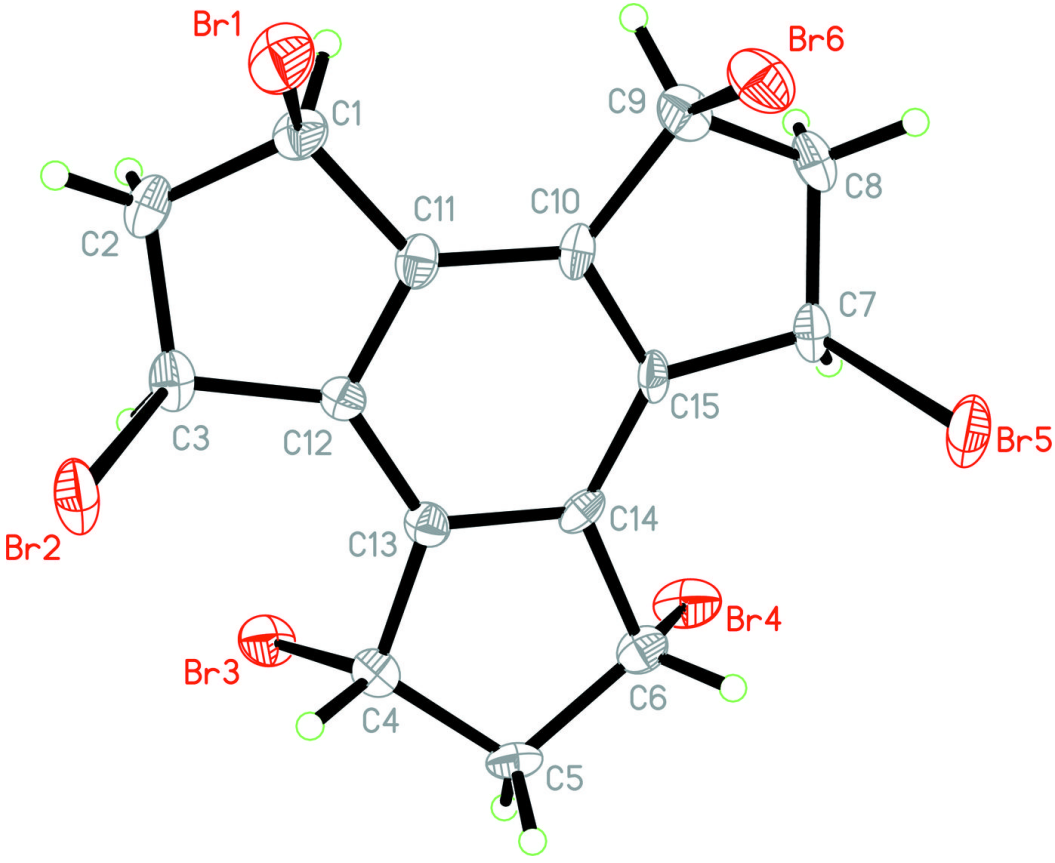
	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0306 (5)	0.0370 (4)	0.0168 (4)	0.0081 (3)	0.0087 (3)	0.0067 (3)
Br2	0.0264 (4)	0.0170 (3)	0.0333 (5)	−0.0006 (3)	0.0001 (3)	0.0054 (3)
Br3	0.0241 (4)	0.0306 (4)	0.0354 (5)	−0.0007 (3)	0.0043 (3)	−0.0164 (3)
Br4	0.0351 (5)	0.0385 (4)	0.0177 (4)	0.0050 (3)	−0.0009 (3)	0.0095 (3)
Br5	0.0330 (5)	0.0165 (3)	0.0329 (5)	−0.0028 (3)	0.0078 (3)	0.0005 (3)
Br6	0.0430 (5)	0.0245 (4)	0.0230 (4)	−0.0032 (3)	0.0051 (3)	−0.0097 (3)
C1	0.028 (4)	0.025 (4)	0.016 (4)	0.006 (3)	−0.003 (3)	0.001 (3)
C2	0.022 (4)	0.020 (3)	0.018 (4)	0.008 (3)	0.004 (3)	0.004 (3)
C3	0.014 (4)	0.014 (3)	0.016 (4)	0.001 (3)	0.003 (3)	−0.001 (3)
C4	0.016 (4)	0.016 (3)	0.017 (4)	0.000 (3)	0.004 (3)	−0.002 (3)
C5	0.018 (4)	0.015 (3)	0.021 (4)	0.002 (3)	0.004 (3)	0.000 (3)
C6	0.011 (4)	0.023 (3)	0.027 (4)	−0.001 (3)	−0.002 (3)	−0.001 (3)
C7	0.021 (4)	0.019 (3)	0.018 (4)	0.001 (3)	0.000 (3)	−0.006 (3)
C8	0.017 (4)	0.016 (3)	0.013 (4)	0.002 (3)	−0.003 (3)	−0.006 (3)
C9	0.017 (4)	0.019 (3)	0.011 (4)	0.005 (3)	0.002 (3)	0.007 (3)
C10	0.025 (4)	0.023 (3)	0.014 (4)	0.004 (3)	0.004 (3)	0.006 (3)
C11	0.026 (4)	0.022 (3)	0.021 (4)	0.003 (3)	0.011 (3)	0.006 (3)
C12	0.023 (4)	0.013 (3)	0.028 (4)	0.004 (3)	0.003 (3)	0.004 (3)
C13	0.021 (4)	0.015 (3)	0.010 (4)	0.006 (3)	0.001 (3)	0.001 (3)
C14	0.023 (4)	0.016 (3)	0.014 (4)	0.003 (3)	0.002 (3)	−0.001 (3)
C15	0.020 (4)	0.022 (3)	0.019 (4)	0.000 (3)	0.003 (3)	−0.007 (3)

Geometric parameters (Å, °)

Br1—C2	1.976 (7)	C6—C7	1.540 (9)
Br2—C5	2.003 (7)	C6—H6A	0.9900
Br3—C7	1.980 (7)	C6—H6B	0.9900
Br4—C10	1.971 (7)	C7—C8	1.504 (9)
Br5—C12	1.972 (6)	C7—H7A	1.0000
Br6—C15	1.979 (7)	C8—C9	1.376 (9)
C1—C2	1.518 (11)	C9—C13	1.376 (10)
C1—C15	1.525 (10)	C9—C10	1.506 (9)
C1—H1A	0.9900	C10—C11	1.521 (10)
C1—H1B	0.9900	C10—H10A	1.0000
C2—C3	1.482 (10)	C11—C12	1.517 (10)
C2—H2A	1.0000	C11—H11A	0.9900
C3—C14	1.389 (9)	C11—H11B	0.9900
C3—C4	1.396 (10)	C12—C13	1.511 (9)
C4—C8	1.393 (9)	C12—H12A	1.0000
C4—C5	1.479 (9)	C13—C14	1.392 (9)
C5—C6	1.512 (10)	C14—C15	1.488 (10)
C5—H5A	1.0000	C15—H15A	1.0000
C2—C1—C15	106.4 (6)	C9—C8—C4	119.5 (6)
C2—C1—H1A	110.4	C9—C8—C7	129.8 (6)

C15—C1—H1A	110.4	C4—C8—C7	110.7 (6)
C2—C1—H1B	110.4	C8—C9—C13	121.5 (6)
C15—C1—H1B	110.4	C8—C9—C10	128.6 (6)
H1A—C1—H1B	108.6	C13—C9—C10	109.7 (6)
C3—C2—C1	103.8 (6)	C9—C10—C11	104.4 (6)
C3—C2—Br1	109.1 (5)	C9—C10—Br4	109.6 (5)
C1—C2—Br1	112.8 (5)	C11—C10—Br4	112.2 (5)
C3—C2—H2A	110.3	C9—C10—H10A	110.2
C1—C2—H2A	110.3	C11—C10—H10A	110.2
Br1—C2—H2A	110.3	Br4—C10—H10A	110.2
C14—C3—C4	120.2 (6)	C12—C11—C10	106.0 (5)
C14—C3—C2	111.1 (6)	C12—C11—H11A	110.5
C4—C3—C2	128.7 (6)	C10—C11—H11A	110.5
C8—C4—C3	119.4 (6)	C12—C11—H11B	110.5
C8—C4—C5	110.7 (6)	C10—C11—H11B	110.5
C3—C4—C5	129.7 (6)	H11A—C11—H11B	108.7
C4—C5—C6	105.7 (5)	C13—C12—C11	103.0 (5)
C4—C5—Br2	108.5 (5)	C13—C12—Br5	108.2 (5)
C6—C5—Br2	111.5 (4)	C11—C12—Br5	111.2 (5)
C4—C5—H5A	110.4	C13—C12—H12A	111.4
C6—C5—H5A	110.4	C11—C12—H12A	111.4
Br2—C5—H5A	110.4	Br5—C12—H12A	111.4
C5—C6—C7	106.6 (6)	C9—C13—C14	119.5 (6)
C5—C6—H6A	110.4	C9—C13—C12	110.9 (6)
C7—C6—H6A	110.4	C14—C13—C12	129.6 (6)
C5—C6—H6B	110.4	C3—C14—C13	119.7 (6)
C7—C6—H6B	110.4	C3—C14—C15	110.2 (6)
H6A—C6—H6B	108.6	C13—C14—C15	129.5 (6)
C8—C7—C6	103.9 (6)	C14—C15—C1	104.1 (6)
C8—C7—Br3	108.4 (5)	C14—C15—Br6	112.1 (5)
C6—C7—Br3	112.2 (5)	C1—C15—Br6	110.3 (5)
C8—C7—H7A	110.7	C14—C15—H15A	110.1
C6—C7—H7A	110.7	C1—C15—H15A	110.1
Br3—C7—H7A	110.7	Br6—C15—H15A	110.1

Fig. 1


Br1
C1
C2
C11
C10
C9
Br6
C8
C7
C3
C12
C15
Br5
Br2
C13
C14
Br4
Br3
C4
C6
C5