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## Structure Reports

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## 1-(Bromomethyl)adamantane

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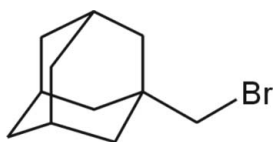
Received 15 June 2011; accepted 17 June 2011

Key indicators: single-crystal X-ray study;  $T = 120$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.017;  $wR$  factor = 0.048; data-to-parameter ratio = 14.9.

The title compound,  $\text{C}_{11}\text{H}_{17}\text{Br}$ , has crystallographically imposed mirror symmetry in the solid state with molecules bisected by mirror planes parallel to the crystallographic  $ac$  plane (five C atoms, three H atoms and the Br atom lie on the mirror plane). The asymmetric unit contains one half-molecule. The crystal packing is stabilized only *via* weak non-specific van der Waals interactions.

## Related literature

For the synthetic procedure, see: Nordlander *et al.* (1966). For the structure of a related non-polar adamantane derivate, see: Rouchal *et al.* (2010).



## Experimental

## Crystal data

 $\text{C}_{11}\text{H}_{17}\text{Br}$  $M_r = 229.16$ 

Monoclinic,  $C2/m$   
 $a = 10.7250$  (3) Å  
 $b = 7.0066$  (3) Å  
 $c = 13.4479$  (4) Å  
 $\beta = 101.801$  (3)°  
 $V = 989.19$  (6) Å<sup>3</sup>

$Z = 4$   
 Mo  $K\alpha$  radiation  
 $\mu = 4.10$  mm<sup>-1</sup>  
 $T = 120$  K  
 $0.40 \times 0.40 \times 0.30$  mm

## Data collection

Oxford Diffraction Xcalibur  
 Sapphire2 diffractometer  
 Absorption correction: multi-scan  
 (*CrysAlis RED*; Oxford  
 Diffraction, 2009)  
 $T_{\min} = 0.480$ ,  $T_{\max} = 1.000$

5102 measured reflections  
 951 independent reflections  
 900 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.014$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.017$   
 $wR(F^2) = 0.048$   
 $S = 1.08$   
 951 reflections

64 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\max} = 0.28$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.29$  e Å<sup>-3</sup>

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2009); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JH2298).

## References

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**supplementary materials**

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## 1-(Bromomethyl)adamantane

J. Cernochová, A. Cablová, M. Rouchal, M. Necas and R. Vícha

### Comment

The title compound is well known and widely used in chemistry of adamantane derivatives as convenient source of 1-adamantylmethyl substituent (Nordlander *et al.*, 1966). Although this compound is very easy to purify *via* crystallization or sublimation, it is prone to form soft thin plates. Due to this fact hand in hand with relatively low melting point (314–316 K), the crystal structure was not published yet. We successfully prepared sufficiently thick plates usable for XRD analyses *via* very slow partial evaporation of solvents from the solution of the title compound in DMF, petroleum ether, and ethyl acetate at room temperature. The molecule of the title compound contains the adamantane moiety consisting from three fused cyclohexane rings in classical chair conformation. The value of C—C—C angles varies within the range of 108.35 (6)–110.27 (12)°. No specific interactions, in addition to the van der Waals interactions, were observed to stabilize the packing of the molecules in the crystal.

### Experimental

The title compound was prepared according to slightly modified previously published procedure (Nordlander *et al.*, 1966). The mixture of starting 1-adamantylmethanol (23.3 g, 0.14 mol), ZnBr<sub>2</sub> (80.7 g, 0.36 mol), and azeotropic hydrobromic acid (412 cm<sup>3</sup>) was refluxed until the GC analyses showed complete disappearing of starting alcohol. Mixture was extracted with hexane:diethyl ether (1:1, v:v), collected organic portions were successively washed with 10% sodium bicarbonate solution and brine and dried over Na<sub>2</sub>SO<sub>4</sub>. The evaporation of solvent yielded of 26.5 g (83%) of pale yellow soft plates.

### Figures

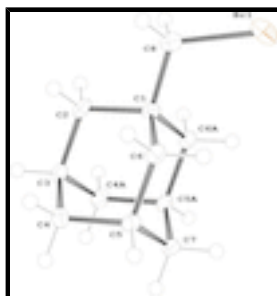


Fig. 1. Molecular structure of of the title compound with atoms represented as 50% probability ellipsoids. Symmetry code used to generate the complete molecule:  $x, -y, z$ .

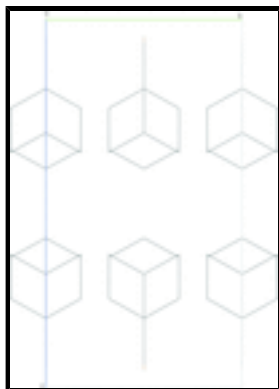


Fig. 2. The unit cell viewed along the *a*-axis. H-atoms have been omitted for clarity.

### 1-(bromomethyl)adamantane

#### Crystal data

$C_{11}H_{17}Br$

$M_r = 229.16$

Monoclinic,  $C2/m$

Hall symbol:  $-C\ 2y$

$a = 10.7250\ (3)\ \text{\AA}$

$b = 7.0066\ (3)\ \text{\AA}$

$c = 13.4479\ (4)\ \text{\AA}$

$\beta = 101.801\ (3)^\circ$

$V = 989.19\ (6)\ \text{\AA}^3$

$Z = 4$

$F(000) = 472$

$D_x = 1.539\ \text{Mg m}^{-3}$

Melting point: 315 K

Mo  $K\alpha$  radiation,  $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 4311 reflections

$\theta = 3.1\text{--}27.2^\circ$

$\mu = 4.10\ \text{mm}^{-1}$

$T = 120\ \text{K}$

Block, colourless

$0.40 \times 0.40 \times 0.30\ \text{mm}$

#### Data collection

Oxford Diffraction Xcalibur Sapphire2 diffractometer

Radiation source: Enhance (Mo) X-ray Source graphite

Detector resolution:  $8.4353\ \text{pixels mm}^{-1}$

$\omega$  scans

Absorption correction: multi-scan (*Crys.Alis RED*; Oxford Diffraction, 2009)

$T_{\min} = 0.480$ ,  $T_{\max} = 1.000$

5102 measured reflections

951 independent reflections

900 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.014$

$\theta_{\max} = 25.0^\circ$ ,  $\theta_{\min} = 3.1^\circ$

$h = -12 \rightarrow 12$

$k = -8 \rightarrow 4$

$l = -15 \rightarrow 15$

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.017$

$wR(F^2) = 0.048$

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$S = 1.08$	$w = 1/[\sigma^2(F_o^2) + (0.029P)^2 + 0.8238P]$
951 reflections	where $P = (F_o^2 + 2F_c^2)/3$
64 parameters	$(\Delta/\sigma)_{\max} < 0.001$
0 restraints	$\Delta\rho_{\max} = 0.28 \text{ e } \text{\AA}^{-3}$
	$\Delta\rho_{\min} = -0.29 \text{ e } \text{\AA}^{-3}$

*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  $F^2 > 2\sigma(F^2)$  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 ( $\text{\AA}^2$ )*

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Br1	0.35255 (2)	0.0000	0.046661 (18)	0.02707 (11)	
C1	0.1776 (2)	0.0000	0.18798 (16)	0.0146 (5)	
C2	0.0362 (2)	0.0000	0.19634 (17)	0.0175 (5)	
H2A	-0.0068	0.1144	0.1621	0.021*	0.50
H2B	-0.0068	-0.1144	0.1621	0.021*	0.50
C3	0.0259 (2)	0.0000	0.30840 (17)	0.0188 (5)	
H3	-0.0660	0.0000	0.3130	0.023*	
C4	0.09066 (14)	0.1784 (3)	0.36097 (12)	0.0199 (4)	
H4A	0.0834	0.1794	0.4332	0.024*	
H4B	0.0483	0.2943	0.3279	0.024*	
C5	0.23165 (14)	0.1783 (3)	0.35368 (12)	0.0169 (4)	
H5	0.2742	0.2948	0.3878	0.020*	
C6	0.24109 (14)	0.1789 (2)	0.24137 (12)	0.0163 (3)	
H6A	0.1988	0.2942	0.2077	0.020*	
H6B	0.3318	0.1824	0.2360	0.020*	
C7	0.2966 (2)	0.0000	0.40589 (17)	0.0183 (5)	
H7A	0.3878	0.0000	0.4021	0.022*	
H7B	0.2908	0.0000	0.4784	0.022*	
C8	0.1790 (2)	0.0000	0.07521 (18)	0.0202 (5)	
H8A	0.1329	0.1141	0.0435	0.024*	0.50
H8B	0.1329	-0.1141	0.0435	0.024*	0.50

*Atomic displacement parameters ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Br1	0.03772 (17)	0.02270 (17)	0.02531 (16)	0.000	0.01703 (11)	0.000

## supplementary materials

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C1	0.0154 (10)	0.0148 (12)	0.0125 (11)	0.000	0.0004 (8)	0.000
C2	0.0128 (10)	0.0162 (12)	0.0204 (12)	0.000	-0.0037 (9)	0.000
C3	0.0106 (10)	0.0236 (13)	0.0214 (12)	0.000	0.0015 (9)	0.000
C4	0.0163 (7)	0.0228 (10)	0.0203 (8)	0.0031 (7)	0.0034 (6)	-0.0032 (7)
C5	0.0148 (7)	0.0176 (9)	0.0173 (8)	-0.0026 (7)	0.0010 (6)	-0.0046 (7)
C6	0.0157 (7)	0.0145 (8)	0.0178 (8)	-0.0012 (7)	0.0014 (6)	-0.0001 (7)
C7	0.0132 (10)	0.0273 (14)	0.0135 (11)	0.000	0.0004 (8)	0.000
C8	0.0230 (11)	0.0195 (13)	0.0167 (11)	0.000	0.0010 (9)	0.000

### Geometric parameters (Å, °)

Br1—C8	1.975 (2)	C4—H4A	0.9900
C1—C8	1.520 (3)	C4—H4B	0.9900
C1—C6	1.533 (2)	C5—C7	1.529 (2)
C1—C6 <sup>i</sup>	1.533 (2)	C5—C6	1.534 (2)
C1—C2	1.544 (3)	C5—H5	1.0000
C2—C3	1.533 (3)	C6—H6A	0.9900
C2—H2A	0.9900	C6—H6B	0.9900
C2—H2B	0.9900	C7—C5 <sup>i</sup>	1.529 (2)
C3—C4	1.531 (2)	C7—H7A	0.9900
C3—C4 <sup>i</sup>	1.531 (2)	C7—H7B	0.9900
C3—H3	1.0000	C8—H8A	0.9900
C4—C5	1.535 (2)	C8—H8B	0.9900
C8—C1—C6	111.91 (12)	C7—C5—C6	109.78 (14)
C8—C1—C6 <sup>i</sup>	111.91 (12)	C7—C5—C4	109.45 (14)
C6—C1—C6 <sup>i</sup>	109.67 (17)	C6—C5—C4	109.09 (12)
C8—C1—C2	106.47 (17)	C7—C5—H5	109.5
C6—C1—C2	108.36 (12)	C6—C5—H5	109.5
C6 <sup>i</sup> —C1—C2	108.36 (12)	C4—C5—H5	109.5
C3—C2—C1	109.91 (17)	C1—C6—C5	110.28 (14)
C3—C2—H2A	109.7	C1—C6—H6A	109.6
C1—C2—H2A	109.7	C5—C6—H6A	109.6
C3—C2—H2B	109.7	C1—C6—H6B	109.6
C1—C2—H2B	109.7	C5—C6—H6B	109.6
H2A—C2—H2B	108.2	H6A—C6—H6B	108.1
C4—C3—C4 <sup>i</sup>	109.48 (18)	C5 <sup>i</sup> —C7—C5	109.57 (17)
C4—C3—C2	109.69 (12)	C5 <sup>i</sup> —C7—H7A	109.8
C4 <sup>i</sup> —C3—C2	109.69 (12)	C5—C7—H7A	109.8
C4—C3—H3	109.3	C5 <sup>i</sup> —C7—H7B	109.8
C4 <sup>i</sup> —C3—H3	109.3	C5—C7—H7B	109.8
C2—C3—H3	109.3	H7A—C7—H7B	108.2
C3—C4—C5	109.26 (14)	C1—C8—Br1	113.35 (15)
C3—C4—H4A	109.8	C1—C8—H8A	108.9
C5—C4—H4A	109.8	Br1—C8—H8A	108.9
C3—C4—H4B	109.8	C1—C8—H8B	108.9
C5—C4—H4B	109.8	Br1—C8—H8B	108.9
H4A—C4—H4B	108.3	H8A—C8—H8B	107.7

Symmetry codes: (i)  $x, -y, z$ .

Fig. 1

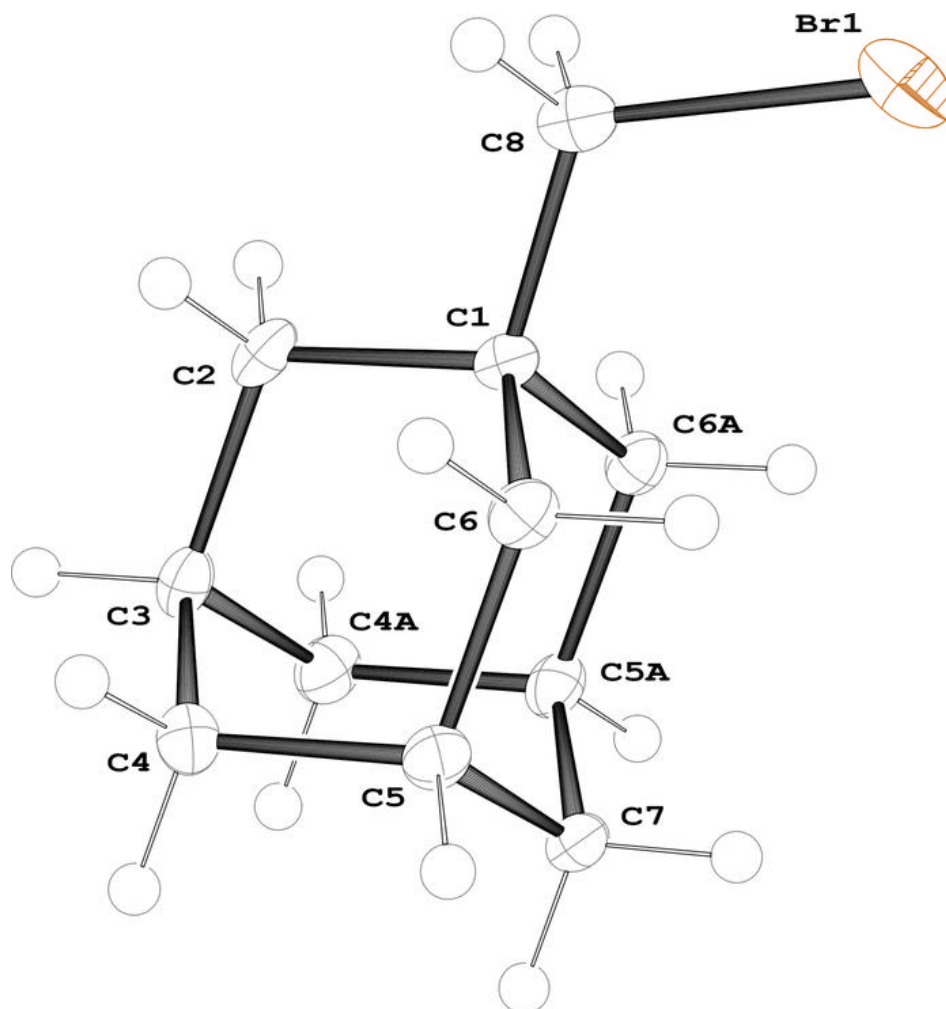


Fig. 2

