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# 1-(Bromomethyl)adamantane

**Jarmila Černochová,[a] Andrea Čablová,[a] Michal Rouchal,[a] Marek Nečas[b] and Robert Vícha[a]***

[a]Department of Chemistry, Faculty of Technology, Tomas Bata University in Zlin, Nám. T. G. Masaryka 275, Zlín,762 72, Czech Republic, and [b]Department of Chemistry, Faculty of Science, Masaryk University, Kamenice 5, Brno-Bohunice, 625 00, Czech Republic
Correspondence e-mail: rvicha@ft.utb.cz

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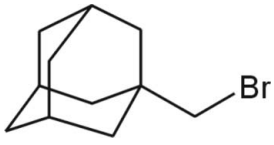
Key indicators: single-crystal X-ray study; $T$ = 120 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.017; $wR$ factor = 0.048; data-to-parameter ratio = 14.9.

The title compound, $C_{11}H_{17}Br$, 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*

$C_{11}H_{17}Br$ — $M_r$ = 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$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 4.10 mm$^{-1}$
$T$ = 120 K
0.40 × 0.40 × 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 $I > 2\sigma(I)$
$R_{int}$ = 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$^{-3}$
$\Delta\rho_{min}$ = −0.29 e Å$^{-3}$

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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# supporting information

*Acta Cryst.* (2011). E**67**, o1820 [doi:10.1107/S1600536811023695]

## 1-(Bromomethyl)adamantane

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### S1. Comment

The title compound is well known and widely used in chemistry of adamantane derivates 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.

### S2. 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), $ZnBr_2$ (80.7 g, 0.36 mol), and azeotropic hydrobromic acid (412 $cm^3$) 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 $Na_2SO_4$. The evaporation of solvent yielded of 26.5 g (83%) of pale yellow soft plates.

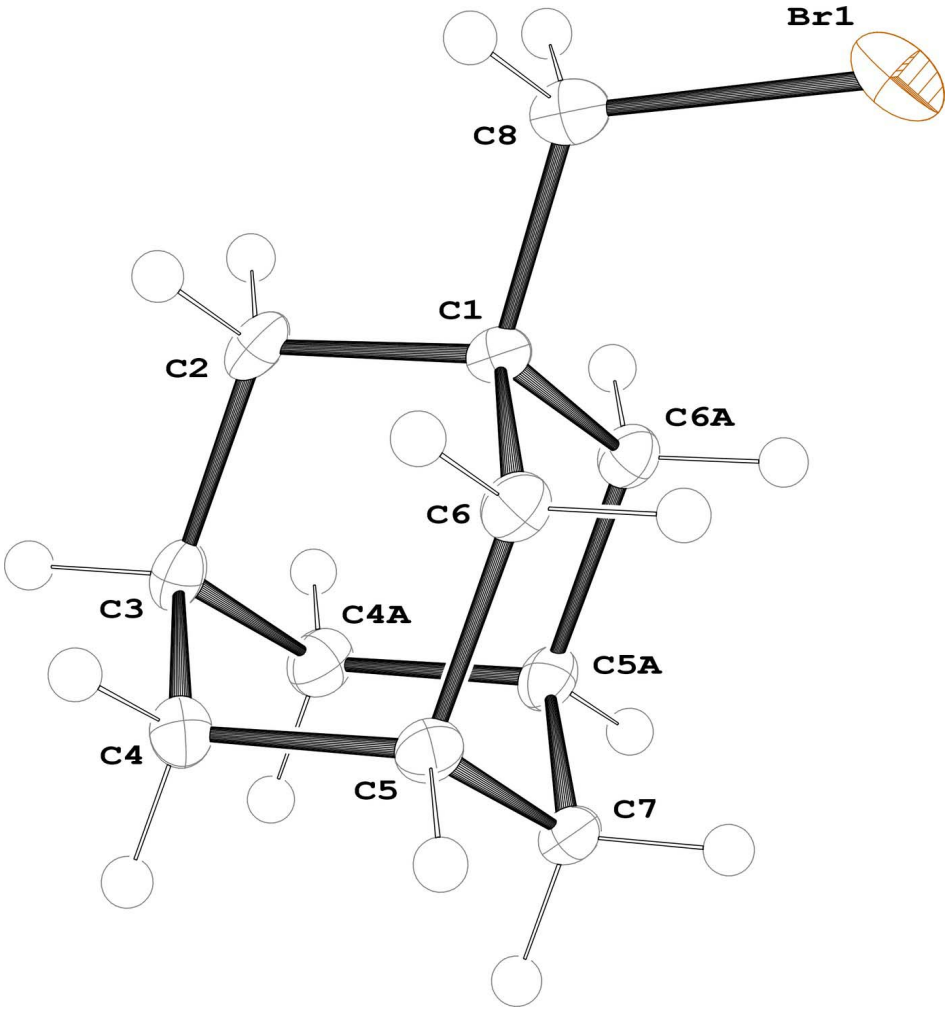


**Figure 1**

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*.

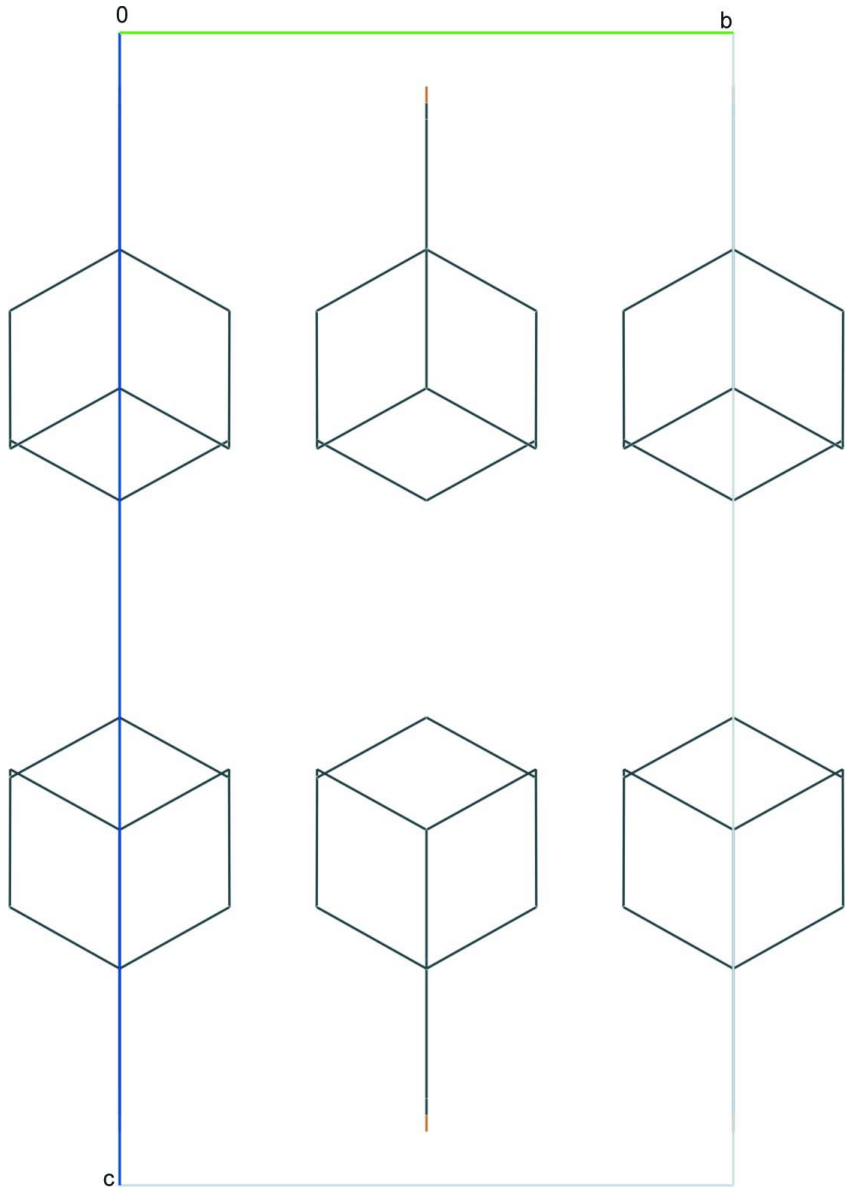


**Figure 2**

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, *C*2/*m*
Hall symbol: -C 2y
*a* = 10.7250 (3) Å
*b* = 7.0066 (3) Å
*c* = 13.4479 (4) Å
$\beta$ = 101.801 (3)°
*V* = 989.19 (6) Å$^3$
*Z* = 4
*F*(000) = 472
$D_x$ = 1.539 Mg m$^{-3}$
Melting point: 315 K
Mo *Kα* radiation, $\lambda$ = 0.71073 Å
Cell parameters from 4311 reflections
$\theta$ = 3.1–27.2°
$\mu$ = 4.10 mm$^{-1}$
*T* = 120 K
Block, colourless
0.40 × 0.40 × 0.30 mm

*Data collection*

Oxford Diffraction Xcalibur Sapphire2 diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 8.4353 pixels mm$^{-1}$
$\omega$ scans
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 $I > 2\sigma(I)$
$R_{int}$ = 0.014
$\theta_{max}$ = 25.0°, $\theta_{min}$ = 3.1°
$h = -12 \rightarrow 12$
$k = -8 \rightarrow 4$
$l = -15 \rightarrow 15$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.017
$wR(F^2)$ = 0.048
$S$ = 1.08
951 reflections
64 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.029P)^2 + 0.8238P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.28 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.29 e Å$^{-3}$

*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 (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ | 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 (Å²)*

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

| | | | |
|---|---|---|---|
| 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$^{i}$ | 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$^{i}$ | 1.529 (2) |
| C3—C4 | 1.531 (2) | C7—H7A | 0.9900 |
| C3—C4$^{i}$ | 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$^{i}$ | 111.91 (12) | C7—C5—C4 | 109.45 (14) |
| C6—C1—C6$^{i}$ | 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$^{i}$—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$^{i}$ | 109.48 (18) | C5$^{i}$—C7—C5 | 109.57 (17) |
| C4—C3—C2 | 109.69 (12) | C5$^{i}$—C7—H7A | 109.8 |

| | | | |
|---|---|---|---|
| C4$^{i}$—C3—C2 | 109.69 (12) | C5—C7—H7A | 109.8 |
| C4—C3—H3 | 109.3 | C5$^{i}$—C7—H7B | 109.8 |
| C4$^{i}$—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 code: (i) $x$, $-y$, $z$.