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3-Hydroxyadamantane-1-acetic acid

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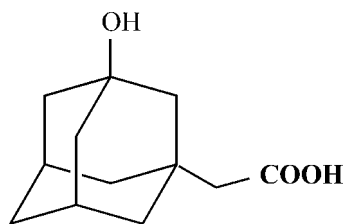
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.048; wR factor = 0.130; data-to-parameter ratio = 17.5.

The crystal structure of the title adamantane derivative, $\text{C}_{12}\text{H}_{18}\text{O}_3$, has been determined by X-ray diffraction. The structure is stabilized by intermolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds, forming a chain.

Related literature

For related literature, see: Lu & Yang (1996); Tukada & Mochizuki (2005); Zhao *et al.* (2003).



Experimental

Crystal data

$\text{C}_{12}\text{H}_{18}\text{O}_3$
 $M_r = 210.26$
 Triclinic, $P\bar{1}$
 $a = 6.5120$ (9) Å
 $b = 7.9485$ (11) Å

$c = 11.5469$ (15) Å
 $\alpha = 106.919$ (10)°
 $\beta = 94.838$ (10)°
 $\gamma = 104.443$ (7)°
 $V = 545.73$ (13) Å³

$Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹

$T = 296$ (2) K
 $0.30 \times 0.13 \times 0.10$ mm

Data collection

Bruker APEXII area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.98$, $T_{\max} = 0.99$

8786 measured reflections
 2488 independent reflections
 1574 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.033$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.130$
 $S = 1.03$
 2488 reflections
 142 parameters
 2 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.18$ e Å⁻³
 $\Delta\rho_{\min} = -0.17$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O1}-\text{H1}\cdots\text{O3}^{\text{i}}$	0.815 (15)	1.995 (16)	2.7959 (18)	168 (2)
$\text{O2}-\text{H2}\cdots\text{O1}^{\text{ii}}$	0.850 (16)	1.811 (16)	2.649 (2)	168 (2)

Symmetry codes: (i) $x - 1, y - 1, z$; (ii) $-x + 1, -y + 2, -z$.

Data collection: APEX2 (Bruker, 2006); cell refinement: SAINT (Bruker, 2006); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2572).

References

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 Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
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 Tukada, H. & Mochizuki, K. (2005). *J. Mol. Struct.* **655**, 473–478.
 Zhao, G. L., Feng, Y. L., Hu, X. C. & Kong, L. C. (2003). *Chin. J. Appl. Chem.* **20**, 802–808.

supporting information

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3-Hydroxyadamantane-1-acetic acid

Xiao-Hong Geng, Li-Chun Kong and Yun-Long Feng

S1. Comment

Adamantane and its derivatives have an extensive application in the field of medicine. For instance, adamantaneamine has an obvious effect on controlling the exuviating the influenza A virus and it can alleviate the Parkinson symptom (Lu *et al.*, 1996). A large number of compounds containing amantadine have been synthesized (Tukada & Mochizuki, 2005; Zhao *et al.*, 2003). Here we report the synthesis and crystal structure of the title compound (I), illustrated in Fig. 1.

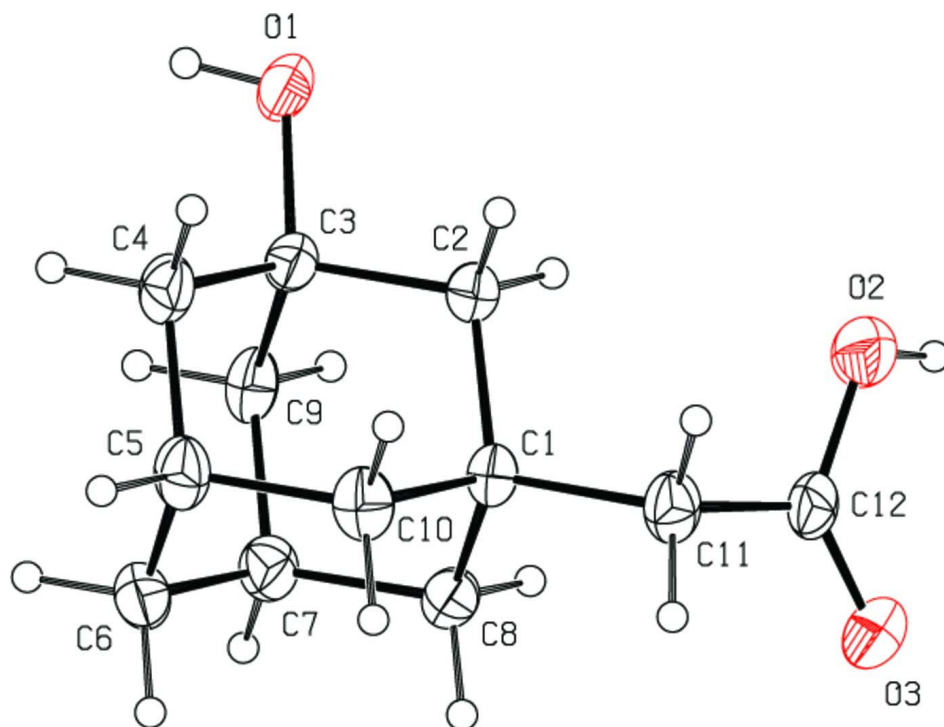
The title compound (I) is an adamantane derivative. Single-crystal X-ray diffraction analyses demonstrate that hydrogen bonding produces an extensive polymeric network since the hydroxyl group substituents are simultaneously hydrogen bonded to the OH of the carboxyl group on an adjacent molecule and the carbonyl group of a different neighbor forming a 12-membered ring as shown in Fig. 2. A 16-membered ring is formed by intermolecular hydrogen bonding between the carbonyl O and hydroxyl H atoms of two molecules. The alternating 12- and 16-membered rings make the compound to form a one-dimensional network.

S2. Experimental

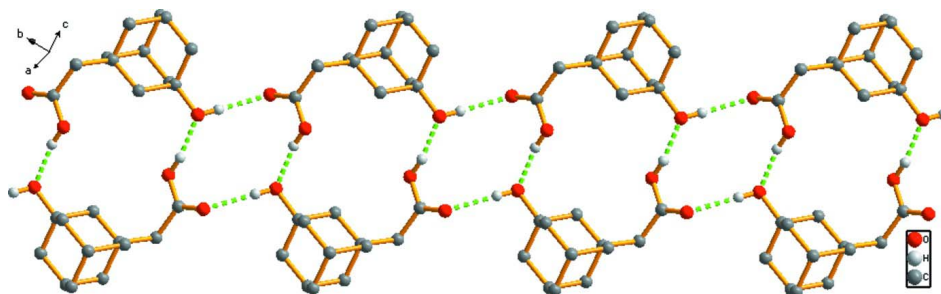
The 3-hydroxy-1-adamantaneacetic was obtained from Zhejiang Key Laboratory for Reactive Chemistry on Solid Surface. Crystals suitable for single-crystal X-ray diffraction were grown by slow evaporation of distilled water.

S3. Refinement

The H atoms bonded to C atoms were positioned geometrically [$C-H = 0.97 \text{ \AA}$, $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$]. The H atoms bonded to O atoms were located in a difference Fourier maps and refined with O—H distance restraints of 0.82 and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{O})$.

**Figure 1**

A view of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are shown at the 30% probability level.

**Figure 2**

A packing diagram for the title compound. The O—H...O interactions are depicted by dashed lines.

3-Hydroxyadamantane-1-acetic acid

Crystal data

$C_{12}H_{18}O_3$
 $M_r = 210.26$
 Triclinic, $P\bar{1}$
 Hall symbol: $-P\ 1$
 $a = 6.5120$ (9) Å
 $b = 7.9485$ (11) Å
 $c = 11.5469$ (15) Å
 $\alpha = 106.919$ (10)°
 $\beta = 94.838$ (10)°
 $\gamma = 104.443$ (7)°
 $V = 545.73$ (13) Å³

$Z = 2$
 $F(000) = 228$
 $D_x = 1.280$ Mg m⁻³
 Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
 Cell parameters from 1598 reflections
 $\theta = 1.9$ – 27.5 °
 $\mu = 0.09$ mm⁻¹
 $T = 296$ K
 Block, colourless
 $0.30 \times 0.13 \times 0.10$ mm

Data collection

Bruker APEXII area-detector
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 ω scans
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.98$, $T_{\max} = 0.99$

8786 measured reflections
 2488 independent reflections
 1574 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.033$
 $\theta_{\text{max}} = 27.5^\circ$, $\theta_{\text{min}} = 1.9^\circ$
 $h = -8 \rightarrow 8$
 $k = -10 \rightarrow 10$
 $l = -15 \rightarrow 15$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.130$
 $S = 1.03$
 2488 reflections
 142 parameters
 2 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H atoms treated by a mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.052P)^2 + 0.1083P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.18 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.17 \text{ e } \text{\AA}^{-3}$

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.0947 (2)	0.76522 (17)	0.09607 (12)	0.0581 (4)
H1	0.024 (3)	0.690 (3)	0.1232 (19)	0.070*
O2	0.5777 (2)	1.30404 (19)	0.01092 (13)	0.0620 (4)
H2	0.694 (3)	1.295 (3)	-0.015 (2)	0.074*
O3	0.7982 (2)	1.51594 (19)	0.17220 (13)	0.0647 (4)
C4	-0.0106 (3)	0.9805 (3)	0.26199 (16)	0.0462 (4)
H4B	-0.0679	0.8858	0.2976	0.055*
H4A	-0.1258	0.9836	0.2043	0.055*
C9	0.3516 (3)	0.9321 (2)	0.28586 (16)	0.0444 (4)
H9B	0.4665	0.9027	0.2431	0.053*
H9A	0.2979	0.8377	0.3223	0.053*
C3	0.1714 (3)	0.9376 (2)	0.19572 (15)	0.0392 (4)
C2	0.2580 (3)	1.0849 (2)	0.13783 (15)	0.0387 (4)
H2B	0.1441	1.0871	0.0790	0.046*
H2A	0.3724	1.0562	0.0943	0.046*

C8	0.5237 (3)	1.2664 (2)	0.32771 (15)	0.0413 (4)
H8B	0.5804	1.3850	0.3913	0.050*
H8A	0.6399	1.2391	0.2854	0.050*
C7	0.4377 (3)	1.1188 (2)	0.38625 (16)	0.0441 (4)
H7A	0.5541	1.1154	0.4444	0.053*
C6	0.2554 (3)	1.1612 (3)	0.45363 (16)	0.0494 (5)
H6B	0.2012	1.0676	0.4906	0.059*
H6A	0.3086	1.2787	0.5185	0.059*
C5	0.0756 (3)	1.1668 (2)	0.36319 (16)	0.0458 (5)
H5A	-0.0411	1.1949	0.4065	0.055*
C10	0.1616 (3)	1.3152 (2)	0.30564 (17)	0.0464 (5)
H10B	0.2146	1.4338	0.3694	0.056*
H10A	0.0462	1.3203	0.2491	0.056*
C1	0.3444 (3)	1.2738 (2)	0.23674 (14)	0.0352 (4)
C11	0.4224 (3)	1.4276 (2)	0.18016 (16)	0.0457 (5)
H11A	0.3065	1.4201	0.1186	0.055*
H11B	0.4522	1.5452	0.2441	0.055*
C12	0.6181 (3)	1.4222 (2)	0.12218 (16)	0.0436 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0652 (10)	0.0397 (7)	0.0542 (8)	-0.0080 (6)	0.0268 (7)	0.0076 (6)
O2	0.0614 (10)	0.0572 (9)	0.0552 (9)	0.0018 (7)	0.0240 (7)	0.0091 (7)
O3	0.0584 (10)	0.0567 (9)	0.0637 (9)	-0.0088 (7)	0.0151 (7)	0.0171 (7)
C4	0.0361 (10)	0.0536 (11)	0.0504 (11)	0.0072 (8)	0.0157 (8)	0.0217 (9)
C9	0.0466 (11)	0.0412 (10)	0.0567 (11)	0.0145 (8)	0.0201 (9)	0.0279 (8)
C3	0.0391 (10)	0.0350 (9)	0.0390 (9)	0.0028 (7)	0.0129 (7)	0.0099 (7)
C2	0.0364 (10)	0.0412 (9)	0.0369 (9)	0.0050 (7)	0.0088 (7)	0.0147 (7)
C8	0.0393 (10)	0.0392 (9)	0.0429 (9)	0.0043 (8)	0.0046 (8)	0.0162 (8)
C7	0.0427 (11)	0.0492 (10)	0.0432 (10)	0.0095 (8)	0.0026 (8)	0.0237 (8)
C6	0.0646 (13)	0.0448 (10)	0.0387 (10)	0.0092 (9)	0.0153 (9)	0.0171 (8)
C5	0.0464 (11)	0.0484 (10)	0.0510 (11)	0.0180 (9)	0.0251 (9)	0.0202 (9)
C10	0.0498 (11)	0.0471 (10)	0.0519 (11)	0.0210 (9)	0.0181 (9)	0.0218 (9)
C1	0.0360 (9)	0.0351 (9)	0.0392 (9)	0.0098 (7)	0.0110 (7)	0.0179 (7)
C11	0.0564 (12)	0.0384 (10)	0.0507 (10)	0.0156 (9)	0.0175 (9)	0.0228 (8)
C12	0.0575 (13)	0.0317 (9)	0.0449 (10)	0.0066 (9)	0.0153 (9)	0.0208 (8)

Geometric parameters (Å, °)

O1—C3	1.447 (2)	C8—C7	1.532 (2)
O1—H1	0.815 (15)	C8—H8B	0.9700
O2—C12	1.311 (2)	C8—H8A	0.9700
O2—H2	0.850 (16)	C7—C6	1.527 (3)
O3—C12	1.210 (2)	C7—H7A	0.9800
C4—C3	1.519 (2)	C6—C5	1.520 (3)
C4—C5	1.530 (2)	C6—H6B	0.9700
C4—H4B	0.9700	C6—H6A	0.9700

C4—H4A	0.9700	C5—C10	1.529 (2)
C9—C3	1.519 (2)	C5—H5A	0.9800
C9—C7	1.528 (2)	C10—C1	1.536 (2)
C9—H9B	0.9700	C10—H10B	0.9700
C9—H9A	0.9700	C10—H10A	0.9700
C3—C2	1.525 (2)	C1—C11	1.546 (2)
C2—C1	1.532 (2)	C11—C12	1.493 (2)
C2—H2B	0.9700	C11—H11A	0.9700
C2—H2A	0.9700	C11—H11B	0.9700
C8—C1	1.528 (2)		
C3—O1—H1	107.3 (15)	C9—C7—H7A	109.5
C12—O2—H2	110.6 (16)	C8—C7—H7A	109.5
C3—C4—C5	109.05 (14)	C5—C6—C7	109.37 (13)
C3—C4—H4B	109.9	C5—C6—H6B	109.8
C5—C4—H4B	109.9	C7—C6—H6B	109.8
C3—C4—H4A	109.9	C5—C6—H6A	109.8
C5—C4—H4A	109.9	C7—C6—H6A	109.8
H4B—C4—H4A	108.3	H6B—C6—H6A	108.2
C3—C9—C7	109.47 (13)	C6—C5—C10	109.57 (15)
C3—C9—H9B	109.8	C6—C5—C4	109.75 (14)
C7—C9—H9B	109.8	C10—C5—C4	109.33 (14)
C3—C9—H9A	109.8	C6—C5—H5A	109.4
C7—C9—H9A	109.8	C10—C5—H5A	109.4
H9B—C9—H9A	108.2	C4—C5—H5A	109.4
O1—C3—C9	110.50 (14)	C5—C10—C1	110.38 (13)
O1—C3—C4	110.82 (13)	C5—C10—H10B	109.6
C9—C3—C4	109.93 (14)	C1—C10—H10B	109.6
O1—C3—C2	106.48 (13)	C5—C10—H10A	109.6
C9—C3—C2	109.35 (13)	C1—C10—H10A	109.6
C4—C3—C2	109.69 (14)	H10B—C10—H10A	108.1
C3—C2—C1	110.46 (12)	C8—C1—C2	108.35 (13)
C3—C2—H2B	109.6	C8—C1—C10	108.54 (13)
C1—C2—H2B	109.6	C2—C1—C10	108.65 (14)
C3—C2—H2A	109.6	C8—C1—C11	111.84 (14)
C1—C2—H2A	109.6	C2—C1—C11	111.65 (13)
H2B—C2—H2A	108.1	C10—C1—C11	107.71 (13)
C1—C8—C7	110.32 (13)	C12—C11—C1	115.10 (13)
C1—C8—H8B	109.6	C12—C11—H11A	108.5
C7—C8—H8B	109.6	C1—C11—H11A	108.5
C1—C8—H8A	109.6	C12—C11—H11B	108.5
C7—C8—H8A	109.6	C1—C11—H11B	108.5
H8B—C8—H8A	108.1	H11A—C11—H11B	107.5
C6—C7—C9	108.98 (14)	O3—C12—O2	122.43 (17)
C6—C7—C8	109.93 (14)	O3—C12—C11	123.93 (17)
C9—C7—C8	109.30 (13)	O2—C12—C11	113.63 (17)
C6—C7—H7A	109.5		

C7—C9—C3—O1	176.98 (12)	C3—C4—C5—C10	60.50 (18)
C7—C9—C3—C4	-60.38 (17)	C6—C5—C10—C1	60.28 (19)
C7—C9—C3—C2	60.09 (17)	C4—C5—C10—C1	-60.04 (19)
C5—C4—C3—O1	-177.88 (13)	C7—C8—C1—C2	-59.16 (17)
C5—C4—C3—C9	59.68 (17)	C7—C8—C1—C10	58.66 (17)
C5—C4—C3—C2	-60.59 (18)	C7—C8—C1—C11	177.35 (14)
O1—C3—C2—C1	-179.80 (13)	C3—C2—C1—C8	59.39 (17)
C9—C3—C2—C1	-60.39 (17)	C3—C2—C1—C10	-58.36 (17)
C4—C3—C2—C1	60.23 (18)	C3—C2—C1—C11	-177.01 (14)
C3—C9—C7—C6	60.25 (16)	C5—C10—C1—C8	-59.19 (18)
C3—C9—C7—C8	-59.91 (18)	C5—C10—C1—C2	58.44 (18)
C1—C8—C7—C6	-59.55 (18)	C5—C10—C1—C11	179.54 (14)
C1—C8—C7—C9	60.02 (18)	C8—C1—C11—C12	53.8 (2)
C9—C7—C6—C5	-60.32 (17)	C2—C1—C11—C12	-67.8 (2)
C8—C7—C6—C5	59.45 (18)	C10—C1—C11—C12	172.94 (15)
C7—C6—C5—C10	-59.72 (18)	C1—C11—C12—O3	-98.6 (2)
C7—C6—C5—C4	60.34 (18)	C1—C11—C12—O2	80.82 (19)
C3—C4—C5—C6	-59.71 (17)		

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1 \cdots O3 ⁱ	0.82 (2)	2.00 (2)	2.7959 (18)	168 (2)
O2—H2 \cdots O1 ⁱⁱ	0.85 (2)	1.81 (2)	2.649 (2)	168 (2)

Symmetry codes: (i) $x-1, y-1, z$; (ii) $-x+1, -y+2, -z$.