

Mo Aung Myint, Allan G. Blackman* and Eng Wui Tan

Department of Chemistry, University of Otago,
 PO Box 56, Dunedin, New Zealand

Correspondence e-mail:
 blackman@alkali.otago.ac.nz

Key indicators

Single-crystal X-ray study
 T = 85 K
 Mean $\sigma(\text{C}-\text{C}) = 0.001 \text{ \AA}$
 R factor = 0.033
 wR factor = 0.097
 Data-to-parameter ratio = 24.2

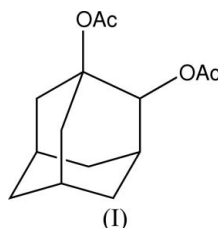
For details of how these key indicators were
 automatically derived from the article, see
<http://journals.iucr.org/e>.

(±)-Adamantane-1,2-diyl diacetate

The first structural analysis of a 1,2-disubstituted adamantane, (±)-adamantane-1,2-diyl diacetate, $\text{C}_{14}\text{H}_{20}\text{O}_4$, is reported. The molecules pack in columns which are held together by inter- and intracolumn $\text{C}-\text{H}\cdots\text{O}$ interactions.

Comment

The title compound, (I) (Fig. 1), was prepared as an intermediate in the synthesis of adamantane-1,2-diol as part of a study of binding selectivity of molecularly imprinted polymers with conformationally restricted molecules. Compound (I) crystallizes in the monoclinic space group $P2_1/c$, confirming the presence of both enantiomers in the crystalline material. The C—C bond distances within the adamantane cage range from 1.5293 (12) to 1.5402 (12) Å, hence showing a greater variety of values than those in unsubstituted adamantane (1.528–1.530 Å; Amoureux & Foulon, 1987). The molecules are arranged in columns, which run parallel to the *a* axis. The only intracolumn interaction of any note is that between carbonyl atom O3 and atom H10B (2.682 Å) of a neighbouring molecule at $(-1 + x, y, z)$. Carbonyl atom O4 is involved in interactions with H6($x, \frac{1}{2} - y, -\frac{1}{2} + z$) (2.664 Å) and H9($-x, -y, 1 - z$) (2.646 Å) of molecules in adjacent columns, while O3 also engages in an intercolumn interaction with H2A($-1 - x, -y, 2 - z$) (2.450 Å).



Experimental

Adamantane-1,2-diacetate was prepared as described previously (McKervey *et al.*, 1971; Janjatovic & Majerski, 1980) and was crystallized from a chloroform/2-propanol mixture as large colourless blocks, which were cut to provide suitable crystals for structural analysis.

Crystal data

$\text{C}_{14}\text{H}_{20}\text{O}_4$
 $M_r = 252.30$
 Monoclinic, $P2_1/c$
 $a = 8.699 (5) \text{ \AA}$
 $b = 12.618 (5) \text{ \AA}$
 $c = 11.835 (5) \text{ \AA}$
 $\beta = 102.934 (5)^\circ$
 $V = 1266.1 (10) \text{ \AA}^3$
 $Z = 4$

$D_x = 1.324 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 Cell parameters from 9961 reflections
 $\theta = 2.9\text{--}33.3^\circ$
 $\mu = 0.10 \text{ mm}^{-1}$
 $T = 85 (2) \text{ K}$
 Irregular chip, colourless
 $0.43 \times 0.23 \times 0.22 \text{ mm}$

Received 31 August 2005
 Accepted 1 September 2005
 Online 7 September 2005

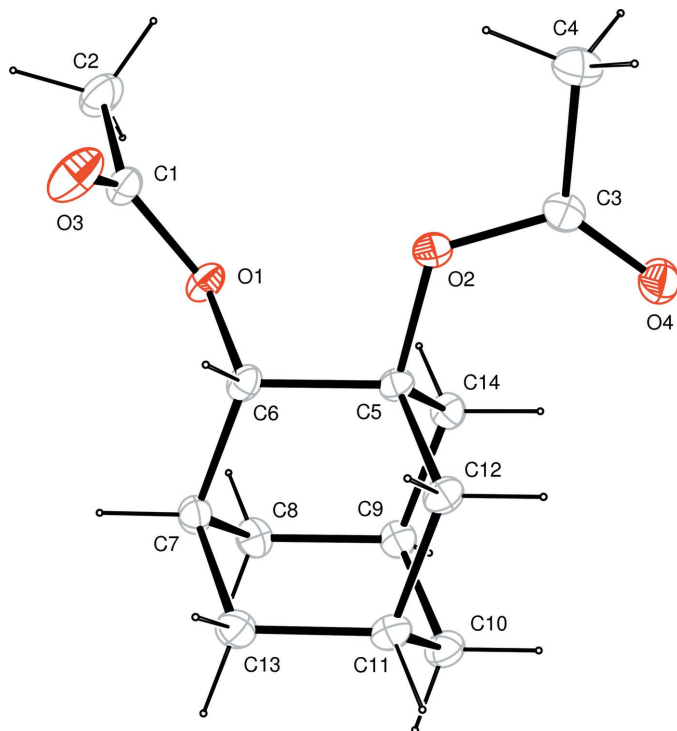


Figure 1
The structure of (I), with displacement ellipsoids drawn at the 50% probability level.

Data collection

Bruker Kappa-APEX-II area-detector diffractometer
 φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2001)
 $T_{\min} = 0.919$, $T_{\max} = 1.000$
26090 measured reflections

3997 independent reflections
3699 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.022$
 $\theta_{\text{max}} = 33.3^\circ$
 $h = -12 \rightarrow 12$
 $k = -14 \rightarrow 18$
 $l = -17 \rightarrow 17$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.033$
 $wR(F^2) = 0.097$
 $S = 1.03$
3997 reflections
165 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0517P)^2 + 0.3507P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.44 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.16 \text{ e } \text{\AA}^{-3}$

All H atoms were included in calculated positions (C–H = 0.96 Å for methyl H atoms, 0.97 Å for methylene H atoms and 0.98 Å for methine H atoms) and were refined as riding atoms with $U_{\text{iso}}(\text{H}) =$

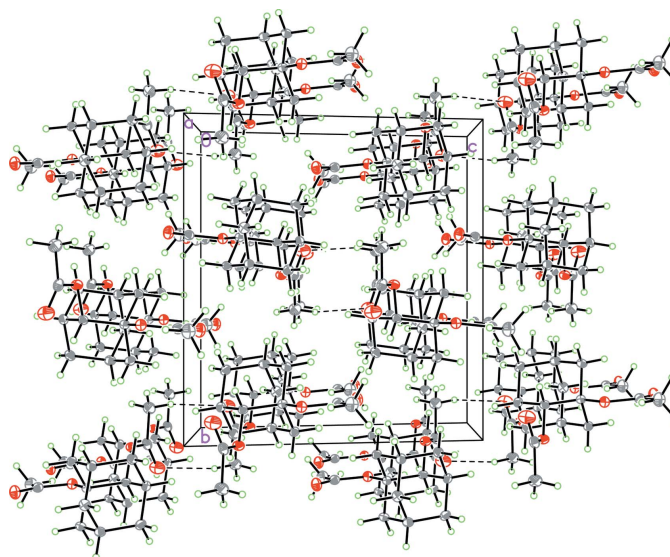


Figure 2
Packing diagram viewed down the a axis. Close interactions are indicated as dashed lines.

$1.2U_{\text{eq}}$ (parent atom, methylene and methine H atoms) or $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}$ (parent atom, methyl H atoms).

Data collection: APEX-II (Bruker, 2004); cell refinement: APEX-II and SAINT (Bruker, 2001); data reduction: SAINT; program(s) used to solve structure: SIR97 (Altomare *et al.*, 1999); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 in WinGX (Farrugia, 1999); software used to prepare material for publication: WinGX.

We thank Fonterra and the Foundation of Research, Science and Technology (FRST) for their support of this research.

References

- Altomare, A., Burla, M. C., Camalli, M., Casciarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
Amoureux, J. P. & Foulon, M. (1987). *Acta Cryst.* **B43**, 470–479.
Bruker (2001). SAINT and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2004). APEX-II. Version 1.0-27. Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Janjatovic, J. & Majerski, Z. (1980). *J. Org. Chem.* **45**, 4892–4898.
McKervey, M. A., Cuddy, B. D. & Grant, D. (1971). *J. Chem. Soc. Sect. C*, pp. 3173–3179.
Sheldrick, G. M. (1997). SHELXL97. University of Göttingen, Germany.