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5-*endo*,6-*endo*-Bis(acetoxymethyl)bicyclo[2.2.1]-hept-2-ene

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
R factor = 0.044
wR factor = 0.128
Data-to-parameter ratio = 16.4

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

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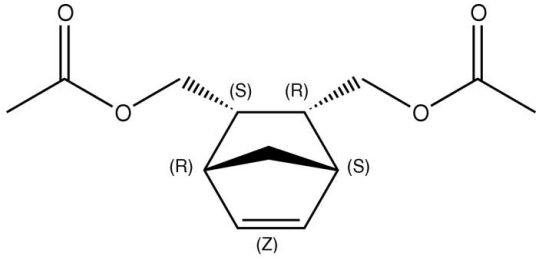
The structure of the title compound (I) $C_{13}H_{18}O_4$ contains three rings in a bicyclo[2.2.1] system (two five-membered rings and one six-membered ring).

Comment

The title compound, (I), was prepared by the reaction of 5-*endo*,6-*endo*-bis(hydroxymethyl)bicyclo[2.2.1]hept-2-ene and acetic acid anhydride (Bailey & Lawson, 1955). The crystal structure determination was performed to elucidate the molecular conformation of (I), which, as expected, was found to consist of a bicyclo[2.2.1] ring system and two acetoxymethyl groups. The acetoxymethyl groups are extended away from one another. In the bicyclo[2.2.1] system, both five-membered rings have envelope conformations [C5—C6—C7—C8 = 0.3 (2)° and C5—C4—C10—C8 = 1.38 (15)°], atom C9 being the out-of-plane atom in both rings. The six-membered ring displays a normal boat conformation.



(I)

Experimental

To 5-*endo*,6-*endo*-di(hydroxymethyl)bicyclo[2.2.1]hept-2-ene (7 g, 45 mmol) in acetic anhydride (100 ml) was added sodium acetate (37 mg, 0.45 mmol). The solvent was removed *in vacuo* after stirring the reaction mixture at 358 K for 12 h. The residue was dissolved in ethyl acetate, filtrated and dried over anhydrous sodium sulfate. Crystallization of the residue from ethanol yielded 7.63 g (70%) of (I) as colourless prisms. M.p. 343 K. IR (CCl_4, χm^{-1}): 1740 (CO). ^{1}H NMR ($CDCl_3$, p.p.m.): δ 6.17 (*t*, 2H), 3.74–3.91 (*m*, 4H), 2.92 (*m*, 2H), 2.53 (*m*, 2H), 2.06 (*s*, 6H), 1.54–1.33 (*m*, 2H).

Crystal data

$C_{13}H_{18}O_4$
M_r = 238.27
Monoclinic, $P2_1/n$
a = 10.114 (2) Å
b = 6.5143 (14) Å
c = 19.311 (4) Å
β = 93.372 (3)°
V = 1270.1 (5) Å^3
Z = 4
D_x = 1.246 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 2313 reflections
θ = 3.3–25.5°
μ = 0.09 mm^{-1}
T = 294 (2) K
Prism, colourless
0.28 × 0.22 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer	2561 independent reflections
φ and ω scans	1745 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$R_{int} = 0.029$
$T_{min} = 0.976$, $T_{max} = 0.984$	$\theta_{max} = 26.4^\circ$
6832 measured reflections	$h = -12 \rightarrow 12$
	$k = -8 \rightarrow 7$
	$l = -12 \rightarrow 24$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0663P)^2 + 0.2238P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.044$	$(\Delta/\sigma)_{max} = 0.003$
$wR(F^2) = 0.128$	$\Delta\rho_{max} = 0.24$ e Å^{-3}
$S = 1.00$	$\Delta\rho_{min} = -0.26$ e Å^{-3}
2561 reflections	
156 parameters	
H-atom parameters constrained	

All H atoms were positioned geometrically and refined as riding (C—H = 0.93–0.98 Å). For the CH and CH_2 groups, U_{iso}(H) values were set equal to $1.2U_{eq}$(carrier atom), and for the methyl groups, they were set equal to $1.5U_{eq}$(carrier atom).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

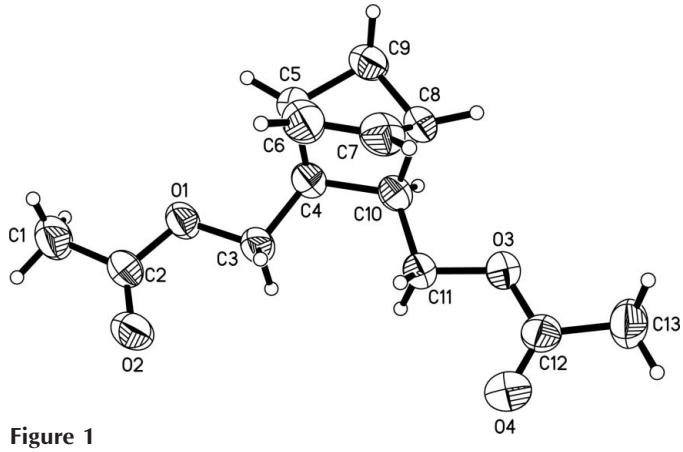


Figure 1
View of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

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