C5—H5*B*···O2^{iv}	0.97	2.62	3.585 (6)	173
C5′—H5*B*′···O1^{v}	0.97	2.45	3.368 (7)	157

Symmetry codes: (i) $x-\frac{1}{2}, \frac{1}{2}-y, z-\frac{1}{2}$; (ii) $1-x, -y, 1-z$; (iii) $1-x, 1-y, 1-z$; (iv) $\frac{3}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$; (v) $x, 1+y, z$.

The initial solution of the structure located C4 and C5 in positions approximately coplanar with the other C atoms and at a distance from each other much shorter than for a known C—C single bond. Initial cycles of refinement showed areas of high residual electron density above and below both C4 and C5, and subsequent anisotropic refinement of these C atoms caused them to show extremely large and prolated displacement ellipsoids. Atoms C4 and C5 were then refined isotropically with their displacement parameters held at 0.05 Å^2. The positions of maximum residual electron density above and below C4 and C5 were then chosen as the sites for the disordered C atoms. Occupancies for C4, C4′, C5 and C5′ were each initially set to 50%. Subsequent isotropic and anisotropic refinement continued smoothly. Occupancies for C4, C4′, C5 and C5′ were then allowed to refine but remained at 50% within error and so were again fixed at 50%. Several non-carboxyl H atoms were found in electron-density difference maps, but were replaced in calculated positions and allowed to refine as riding models on their appropriate C atoms. Disordered H atoms were added in calculated positions to C4, C4′, C5 and C5′, and also to C6, with occupancies set at 50%. The displacement parameters for these methylene H atoms were refined as two groups relating to the flexional disorder. The carboxyl H3 atom was found in an electron-density difference map and was allowed to refine both positionally and isotropically.

Data collection: *XSCANS* (Fait, 1991). Cell refinement: *XSCANS* (Siemens, 1996). Data reduction: *XSCANS* (Siemens, 1996). Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1994). Program(s) used to refine structure: *SHELXTL/PC*. Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXTL/PC*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1448). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1998). C**54**, 1285–1287

10,16-Dioxatetracyclo[7.6.1.0^{1,11}.0^{4,9}]hexadecan-11-ol

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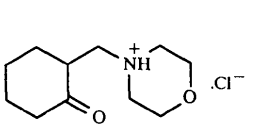
Abstract

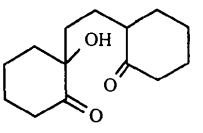
The crystal structure of the title compound, $C_{14}H_{22}O_3$, consists of independent molecules in each of which three six-membered rings and a five-membered ring are fused together. All the six-membered rings display chair conformations, while the five-membered ring has an envelope conformation. The molecular dimensions are normal with mean bond distances C_{sp^3}—C_{sp^3} 1.523 (10) and C_{sp^3}—O 1.44 (2) Å. Pairs of molecules lying about inversion centers are linked by O—H···O hydrogen bonds [O···O 2.806 (3) Å].

Comment

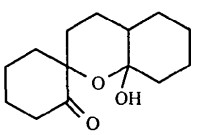
The usefulness of Mannich bases as intermediates for the synthesis of biologically active compounds is well known. Some of the Mannich bases have been found to be active against EM T_6 mammary carcinoma cells (Dimmock, Erciyas, Raghavan & Kirkpatrick, 1990). The Mannich bases containing morpholino groups have been reported to exhibit a high degree of cytotoxicity (Dimmock, Erciyas, Bigam *et al.*, 1990). It was considered worthwhile to study the alkylation reaction of 2-(morpholinomethyl)cyclohexanone, (1), in an attempt to synthesise intermediates which could be transformed into biologically active compounds. It has been reported that the reaction of (1).HCl with CH_3CN or CH_3NO_2 in aqueous medium yielded a compound formulated as (2) in solution, but which was suggested to be either (3) or (4) in the solid state, depending on the conditions (Mannich, 1941; Roth & Dvorak, 1963). We have repeated the original synthesis and have now shown that the structure of the crystalline material that we obtained is correctly formulated as (4). As the crystals of (4) are composed of a racemic mixture, the stereochemistry presented in the scheme is for one of the two enantiomers.



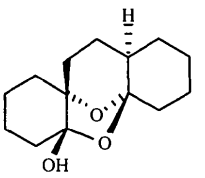
(1).HCl



(2)



(3)



(4)

The crystal of (4) (Fig. 1) contains discrete molecules separated by normal van der Waals distances. Each molecule is composed of four fused rings of which three are six-membered (C1–C6, O1/C1/C6–C9 and C9–C14) and one is five-membered (O1/O2/C1/C9/C10). The six-membered rings display chair conformations. The C1–C6 and C9–C14 rings display torsion angles in the ranges ±[49.9 (3)–57.1 (3)] and ±[39.5 (3)–60.1 (3)]°, respectively; the latter shows a larger degree of flattening due to the constraints imposed by the fused ring system and the hydroxy group on C10. The torsion angles in the six-membered heterocyclic ring are in the range ±[41.7 (2)–76.6 (2)]°, reflecting the constraint exerted by the five-membered ring, which itself adopts a C9-envelope conformation, with C9 0.656 (3) Å out of the plane of the remaining ring atoms. The molecular dimensions in (4) are normal and lie within expected ranges for corresponding bond distances and angles (Orpen *et al.*, 1994), with mean bond distances C_{sp^3}—C_{sp^3} 1.523 (10) and C_{sp^3}—O 1.44 (2) Å.

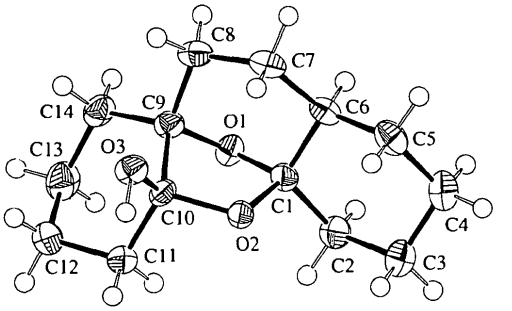


Fig. 1. *ORTEP*II (Johnson, 1976) drawing of (4) with the atomic numbering scheme. Displacement ellipsoids are plotted at the 50% probability level and H atoms have been assigned arbitrary radii.

There is a short intermolecular interaction of the O—H···O type with O3···O2 and H1···O2 distances of 2.806 (3) and 1.88 Å, respectively, and an O3—H1···O2 angle of 165°. Pairs of molecules lying about inversion centers are hydrogen bonded, resulting in eight-membered rings and the formation of dimers.

Experimental

A mixture of (1).HCl (0.466 g, 0.02 mol) and acetonitrile (0.80 g, 0.02 mol) or nitromethane (1.25 g, 0.02 mol) in distilled water (50 ml) was heated on a steam bath for 24 h. The sticky solid obtained on cooling was filtered off (suction) and crystallized from ethyl acetate to give large colorless needles of (4) (m.p. 325 K).

Crystal data

$C_{14}H_{22}O_3$
M_r = 238.32
Monoclinic
$C2/c$
a = 21.558 (4) Å
b = 6.5897 (12) Å
c = 18.861 (2) Å
β = 111.891 (10)°
V = 2486.3 (7) Å^3
Z = 8
D_x = 1.273 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.7107 Å
Cell parameters from 25 reflections
θ = 18.0–25.0°
μ = 0.088 mm^{-1}
T = 150 (1) K
Block, cut from a large needle
0.43 × 0.37 × 0.28 mm
Colorless

Data collection

Rigaku AFC-6*S* diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2258 measured reflections
2199 independent reflections
1358 reflections with $I > 2\sigma(I)$

R_{int} = 0.018
θ_{max} = 25°
h = 0 → 25
k = 0 → 7
l = −22 → 20
3 standard reflections
every 200 reflections
intensity decay: 3.3%

Refinement

Refinement on F^2
$R(F) = 0.040$
$wR(F^2) = 0.096$
$S = 1.055$
2195 reflections
157 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0487P)^2 + 2.4104P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.237$ e Å^{-3}
$\Delta\rho_{min} = -0.194$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H1···O2^i	0.95	1.88	2.806 (3)	165

Symmetry code: (i) $1 - x, -y, -z$.

The H atoms were included at geometrically idealized positions with C—H and O—H distances of 0.95 Å.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1994). Program(s) used to solve structure: *SAPI*91 (Fan, 1991). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976) in *TEXSAN*. Software used to prepare material for publication: *SHELXL*93.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1423). Services for accessing these data are described at the back of the journal.

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trans-2,3,5,7,15-Pentaacetoxyjatropha-6(17),11-diene-9,14-dione, a Diterpene Oligoester

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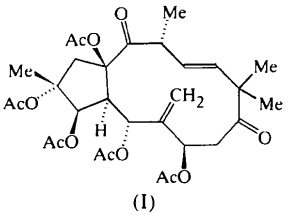
Abstract

The title compound (systematic name: 3,6,6,14-tetramethyl-10-methylene-2,7-dioxobicyclo[10.3.0]pentadeca-4-ene-1,9,11,13,14-pentayl pentaacetate; $C_{30}H_{40}O_{12}$) is a new diterpene oligoester. The structure consists of a five-membered and a twelve-membered ring, both of which are non-planar. They are inclined at an angle of 28.89 (15)°. The five-membered ring has an envelope conformation.

Comment

The title compound has been isolated from the whole undried plant of *Euphorbia esula* (Euphorbiaceae). *Euphorbia esula* L or leafy spurge is a plant distributed all over the world and contains a toxic milky latex, which is a skin irritant. Extracts of the plant have been widely used in folk medicine to treat various cancers, swellings and warts (Hartwell, 1969). As part of studies on biologically active compounds from the family Euphorbiaceae, Hohmann *et al.* (1997) examined a Hungarian population of *E. esula* for its diterpene constituents. This paper deals with the structure elucidation of jatrophane ester (I), named esulatin B.



(I)

The bicyclic structure of (I) consists of a five-membered ring fused with a twelve-membered ring. It has five acetyl groups, four methyl groups, one methyl-