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$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O3—H1$\cdots$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.

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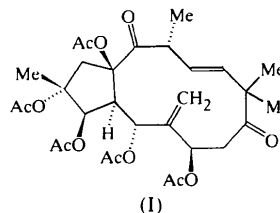
The bicyclic structure of (I) consists of a five-membered ring fused with a twelve-membered ring. It has five acetyl groups, four methyl groups, one methyl-

ene group and six quaternary C atoms, including two keto groups. This compound is a new member of the small group of jatrophane diterpenoids, which are considered to be the most important taxonomic members in this family.

The two rings are not planar and are inclined at an angle of 28.89 (15)° with respect to one another along the C4—C15 bond. The five-membered ring adopts an envelope conformation, with atoms C1, C2, C4 and C15 coplanar to within 0.035 Å, and atom C3 0.626 Å out of this least-squares plane.

The methylene group at C6 is 1.567 (9) Å below the plane defined by atoms C4–C7. The keto group at C9 is disordered. One of the disordered O atoms (O42B) lies nearly in the plane defined by atom C8–C11, while the other (O42A) lies 0.75 (2) Å out of the plane. The methyl group at C10 lies slightly below the same plane. The other keto group (at C14) lies 1.403 (4) Å above the plane defined by atoms C12–C15. No abnormally short intermolecular contacts have been found.
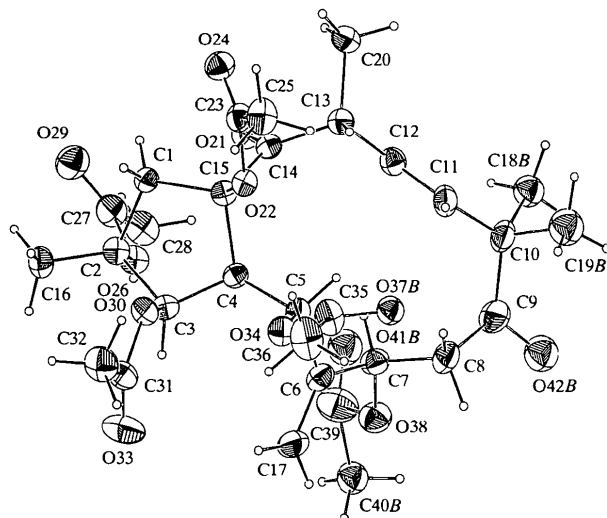

Fig. 1. Structure of the title compound, showing 30% probability displacement ellipsoids and the atom numbering (H atoms are shown as spheres of arbitrary size). For clarity, only one component of each of the disordered atoms has been shown.

Experimental

The dichloromethane extract of a methanol extraction of the whole undried plant of *E. esula* was fractionated by column chromatography on polyamide, then on silica gel, and further purified by preparative TLC and HPLC to obtain esulatin B. Crystals were obtained from a solution of the compound in methanol by slow evaporation at room temperature.

Crystal data

$C_{30}H_{40}O_{12}$
$M_r = 592.62$
Orthorhombic
$P2_12_12$
$a = 16.031$ (2) Å
$b = 17.942$ (3) Å
$c = 11.250$ (3) Å
$V = 3235.8$ (11) Å^3
$Z = 4$
$D_x = 1.216$ Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 29 reflections
$\theta = 20.1–37.9°$
$\mu = 0.788$ mm^{-1}
$T = 293$ (2) K
Plate
$0.25 \times 0.22 \times 0.11$ mm
Colourless

Data collection

Siemens AED four-circle diffractometer
ω–2θ scans
Absorption correction:
  ψ scan (North *et al.*, 1968)
  $T_{min} = 0.85$, $T_{max} = 0.92$
3444 measured reflections
3444 independent reflections

1935 reflections with $I > 2\sigma(I)$
$\theta_{max} = 69.90°$
$h = -18 \rightarrow 19$
$k = 0 \rightarrow 21$
$l = 0 \rightarrow 13$
1 standard reflection
  every 100 reflections
  intensity decay: negligible

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.077$
$wR(F^2) = 0.280$
$S = 1.037$
3377 reflections
386 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.1573P)^2]$
  where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = -0.002$

$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}
Extinction correction: SHELXL93 (Sheldrick, 1993)
Extinction coefficient: 0.0006 (3)
Scattering factors from *International Tables for Crystallography* (Vol. C)

In the molecule, three C atoms and three O atoms were found to be disordered. The disordered atoms and their site occupation factors are: C18A 0.47 (5), C18B 0.53 (5); C19A 0.22 (4), C19B 0.78 (4); O37A 0.60 (3), O37B 0.40 (3); C40A 0.69 (7), C40B 0.31 (7); O41A 0.67 (3), O41B 0.33 (3); O42A 0.52 (4), O42B 0.48 (4). The distances between these isotropic components and the nearest C or O atoms were restrained to be 1.52 (1) Å for C—C bonds and 1.25 (1) Å for C=O double bonds. Limitations in the modelling of this disorder are thought to be the main reason for the high R factors. The H atoms were included at geometrically calculated positions, except for the methyl H atoms on atoms C16, C18A, C18B, C19A, C19B, C20, C25, C28, C32, C36, C40A and C40B, which were found from circular difference Fourier synthesis. They were then allowed to ride on their parent atoms with $U_{iso} = xU_{eq}$(parent), where $x = 1.5$ for methyl H atoms and $x = 1.2$ for all other atoms.

Data collection: local program (Belletti *et al.*, 1993). Cell refinement: local program. Data reduction: local program. Program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ZORTEP* (Zsolnai & Huttner, 1994). Software used to prepare material for publication: *SHELXL93*. Geometric calculations: *PARST* (Nardelli, 1983).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1208). Services for accessing these data are described at the back of the journal.

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2-*O*-Benzoyl-*myo*-inositol-1,3,5-ortho-formate†

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Abstract

Protected *myo*-inositol derivatives are important precursors in the synthesis of phosphorylated *myo*-inositol derivatives, which play a significant role in cellular signal transduction. The structure of the title compound, $C_{14}H_{14}O_7$, which was prepared from *myo*-inositol, has been determined by X-ray crystallography. Several types of hydrogen-bonding interactions are involved in the packing of the molecule in the crystal.

Comment

myo-Inositol and its phosphorylated derivatives play an important role in the cellular signalling process

(Potter & Lampe, 1995) and have been the subject of theoretical studies (Liang *et al.*, 1994). *myo*-Inositol-1,3,5-orthoformate and its derivatives are important intermediates for the synthesis of several *myo*-inositol phosphates (Das & Shashidhar, 1997, and references therein). The title compound, (I), is a key intermediate for the synthesis of *myo*-inositol pentaphosphates (Ozaki *et al.*, 1994; Chung & Chang, 1996). We present here the crystal structure of (I), which was prepared in a one-pot procedure from *myo*-inositol.

(I)
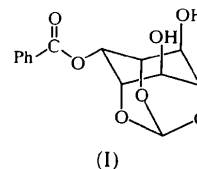
The structure of (I) (Fig. 1) resembles that of *myo*-inositol-1,3,5-orthoformate (Uhlmann & Vasella, 1992). The O1—C1, O3—C3 and O5—C5 bonds are longer than the corresponding lengths from the respective O atoms to C7 by 0.03, 0.05 and 0.05 Å, respectively (Table 1). There are a few potential hydrogen-bond interactions, of both the O—H···O and C—H···O types (Table 2). The crystal density (1.513 Mg m⁻³) is relatively high, indicating a tight packing of molecules in the lattice. The hydroxyl group at O6 is involved in three-centre hydrogen bonding (Taylor *et al.*, 1984; Jeffrey & Maluszynska, 1982) with two acceptor O atoms, one intra- and the other intermolecular, resulting in a large deviation from linearity of the *D*—H···*A* angles. The O6 atom also acts as the acceptor for two more C—H···O interactions.
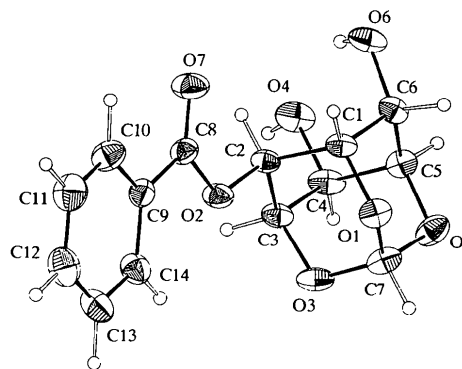

Fig. 1. *ZORTEP* (Zsolnai, 1995) diagram of (I) showing the labelling of the non-H atoms. Displacement ellipsoids are plotted at the 50% probability level.

Experimental

myo-Inositol (2.7 g, 0.015 mol), trimethylorthoformate (2.39 g, 0.0225 mol), *p*-toluenesulfonic acid monohydrate (0.25 g, 1.31 mmol) and dry DMF (20 ml) were mixed and heated at

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