Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

3,7,17-Tri-*O*-acetyl-5-*O*-butanoyl-13,15-dihydroxymyrisinol

Masood Parvez,[a]* Viqar Uddin Ahmad,[b] Javid Hussain,[b] Hidayat Hussain[b] and Umar Farooq[b]

[a]Department of Chemistry, The University of Calgary, 2500 University Drive NW, Calgary, Alberta, Canada T2N 1N4, and [b]HEJ Research Institute of Chemistry, International Center for Chemical Sciences, University of Karachi, Karachi 75270, Pakistan

Correspondence e-mail: parvez@ucalgary.ca

Received 2 December 2003
Accepted 15 December 2003
Online 10 January 2004

The structure of the tricyclic diterpenoid, $C_{30}H_{42}O_{11}$, is composed of a five-membered ring in a *C*4-envelope conformation, a seven-membered ring in a chair conformation and a six-membered ring that adopts a twisted half-chair conformation. The structure is stabilized by intramolecular and intermolecular hydrogen-bond interactions, with O···O separations of 2.815 (4) and 2.812 (4) Å.

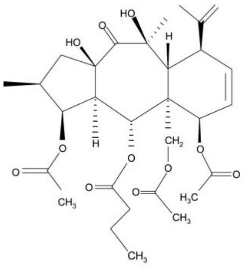
Key indicators

Single-crystal X-ray study
T = 295 K
Mean *σ*(C–C) = 0.005 Å
Disorder in main residue
R factor = 0.048
wR factor = 0.139
Data-to-parameter ratio = 8.0

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

Comment

Euphorbia decipiens grows wild in different parts of Iran at high altitudes (Rechinger & Schiman-Czeika, 1964). Some of the plants belonging to the genus *Euphorbia* are used in folk medicine, for instance *E. kansui* is considered a herbal remedy for edema, ascites and cancer in China and investigation of this plant showed two antileukemic diterpene esters with an ingenane carbon skeleton (Wu *et al.*, 1991). The macrocyclic and polycyclic diterpenes isolated from different species of *Euphorbia* plants with ingenane, tigliane and daphnane skeletons have skin-irritant, tumour-promoting and anti-tumour activities (Shi *et al.*, 1995; Seip & Hecker, 1983; Mbwambo *et al.*, 1996). Some esters of myrsinol isolated from *E. myrsinites* showed anti-HIV-1 reverse transcriptase (RT) inhibition (Öksüs *et al.*, 1995). In this article, we report the crystal structure of the title compound, (I), an interesting diterpenoid isolated from the whole plant material of *E. decipiens*. The crystal structure of decipinone, closely related to the structure of (I), has already been reported from our laboratory (Ahmad *et al.*, 1998).



(I)

The structure of (I), a tricyclic diterpenoid, is shown in Fig. 1. The five-membered ring is in a *C*4-envelope conformation, with C4 0.657 (5) Å out of the plane of the remaining ring atoms, for which the maximum deviation is 0.016 (2) Å. The seven-membered ring is in a chair conformation wherein C1,C5,C7,C12 form an essentially planar base [maximum deviation = 0.005 (2) Å], with C6 lying 0.633 (5) Å below and C13 and C14 0.998 (5) and 1.182 (5) Å, respectively, above the base. The six-membered ring adopts a somewhat twisted half-chair conformation, with C8 0.564 (5) Å out of the plane of the

DOI: 10.1107/S1600536803028770

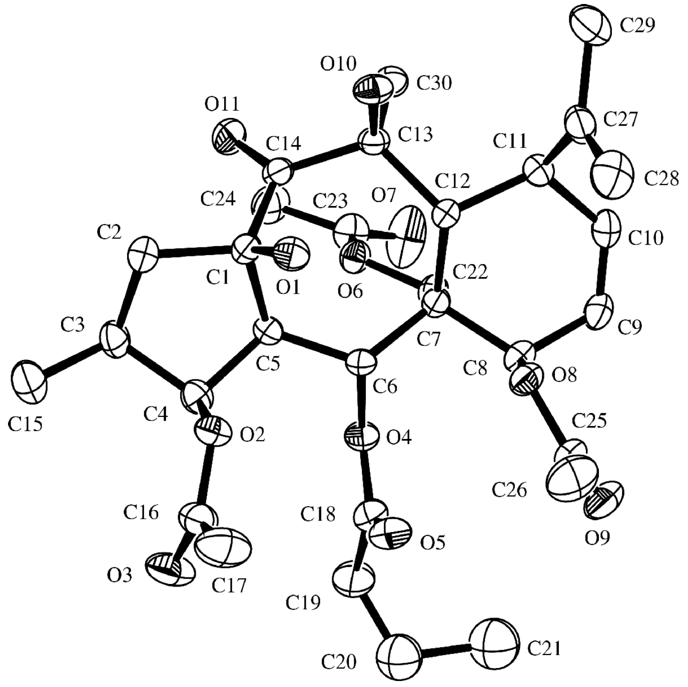


Figure 1
*ORTEP*II (Johnson, 1976) drawing of (I), with displacement ellipsoids drawn at the 30% probability level. The minor disorder components and H atoms have been omitted.

rest of the ring atoms, which deviate from the mean plane by 0.041 (2)–0.152 (2) Å. The molecular dimensions in the molecule, with mean bond lengths including O—Csp^3 = 1.454 (19) Å, O—Csp^2 = 1.337 (8) Å, C=*O* = 1.203 (9) Å, Csp^3—Csp^3 = 1.55 (2) Å, Csp^3—Csp^2 = 1.50 (2) Å and C=*C* = 1.33 (2) Å, are in agreement with the values previously reported (Orpen *et al.*, 1994). The structure is stabilized by intramolecular and intermolecular hydrogen-bonding interactions, with dimensions O1···O10 = 2.815 (4) Å and O1—H1*A*···O10 = 127°, and O10···O3^{i} = 2.812 (4) Å and O10—H10*A*···O3^{i} = 159° (symmetry code as in Table 2).

Experimental

The plants of *Euphorbia decipiens* Boiss. & Buhse (Euphorbiaceae) were collected at the mountain of Kandovan, north of Karaj, Iran, in 1998, and identified by Mr Bahram Zehzad (plant taxonomist) at the Department of Biological Sciences, Shahid Beheshti University, Eveen, Tehran. A voucher specimen (No. 98112) has been deposited at the herbarium of the Biology Department of Shahid Beheshti University.

The air-dried ground plant material (4 kg) was exhaustively extracted with acetone at room temperature. The extract was evaporated and the residue (62 g) defatted by extraction with hexane. The defatted extract (51 g) was then extracted with chloroform. The chloroform extract (44 g) was subjected to chromatography on a silica gel column (880 g), using hexane with a gradient of $CHCl_3$ up to 100%, followed by methanol. 20 fractions were collected. Fraction 14 of the first column was loaded on silica gel and purified by column chromatography, using hexane–EtOAc (50:50) [(8.1 mg, R_F 0.63; $CHCl_3$-acetone (93:7)].

Crystal data

$C_{30}H_{42}O_{11}$
M_r = 578.64
Orthorhombic, $P2_12_12_1$
a = 10.389 (3) Å
b = 14.745 (4) Å
c = 19.531 (7) Å
V = 2991.9 (16) Å^3
Z = 4
D_x = 1.285 Mg m^{-3}

Mo *K*α radiation
Cell parameters from 5134 reflections
θ = 2.1–25.0°
μ = 0.10 mm^{-1}
T = 295 (2) K
Block, colourless
0.10 × 0.09 × 0.08 mm

Data collection

Nonius KappaCCD diffractometer
ω and φ scans
Absorption correction: none
5134 measured reflections
2943 independent reflections
2416 reflections with *I* > 2σ(*I*)

$R_{\rm int}$ = 0.024
$\theta_{\rm max}$ = 25.0°
h = −12 → 12
k = −17 → 17
l = −23 → 22

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.139
S = 1.07
2943 reflections
369 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0808P)^2 + 0.577P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max}$ = 0.42 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.33 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.019 (4)

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.421 (4)	O6—C22	1.443 (5)
O2—C16	1.328 (5)	O7—C23	1.186 (5)
O2—C4	1.453 (4)	O8—C25	1.344 (5)
O3—C16	1.210 (5)	O8—C8	1.463 (4)
O4—C18	1.330 (6)	O9—C25	1.200 (5)
O4—C6	1.469 (4)	O10—C13	1.444 (4)
O5—C18	1.205 (6)	O11—C14	1.212 (4)
O6—C23	1.346 (5)		
C16—O2—C4	118.8 (3)	C23—O6—C22	116.4 (3)
C18—O4—C6	118.9 (3)	C25—O8—C8	116.0 (3)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···O10	0.82	2.24	2.815 (4)	127
O10—H10*A*···O3^{i}	0.82	2.03	2.812 (4)	159

Symmetry code: (i) $\frac{1}{2} - x, 1 - y, \frac{1}{2} + z$.

The absolute configuration could not be established in this analysis, and is arbitrarily assigned. In the absence of significant anomalous scattering effects, the Friedel pairs were merged. Atoms C20 and C21 were disordered over two sites with equal site-occupancy factors and were allowed to refine with isotropic displacement parameters. H atoms were positioned geometrically and refined as riding, with C—H = 0.93–0.98 Å and O—H = 0.82 Å; $U_{\rm iso}$(H) was set at 1.2 (1.5 for methyl and hydroxy groups) times $U_{\rm eq}$ of the parent atom.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *HKL DENZO* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SAPI*91 (Fan, 1991); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II

(Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

References

Ahmad, V. U., Jassbi, A. R. & Parvez, M. (1998). *Tetrahedron*, **54**, 1573–1584.
Fan, H.-F. (1991). *SAPI*91. Rigaku Corporation, Tokyo, Japan.
Hooft, R. (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Mbwambo, Z. H., Lee, S. K., Mshiu, E. N., Pezzuto, J. M. & Kinghorn, A. D. (1996). *J. Nat. Prod*. **59**, 1051–1055.
Öksüs, S., Gürek, F., Gil, R. R., Pengsuparp, T., Pezzuto, J. M. & Cordell, G. A. (1995). *Phytochemistry*, **38**, 1457–1462.
Orpen, A. G., Brammer, L., Allen, F. H., Kennard, O., Watson, D. G., & Taylor, R. (1994). *Structure Correlation*, Vol. 2, edited by H.-B. Bürgi & J. D. Dunitz, Appendix A. Weinheim: VCH Publishers.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Rechinger, K. H. & Schiman-Czeika, H. (1964). *Flora Iranica*, **6**, 33–34.
Seip, E. H. & Hecker, E. (1983). *Phytochemistry*, **22**, 1791–1795.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Shi, Y. P., Jia, Z. J., Jamil, L. & Sadiq, S. (1995). *Phytochemistry*, **40**, 1219–1221.
Wu, T. S., Lin, Y. M., Haruna, M., Pan, D. J., Shingu, T., Chen, Y. P., Hsu, H. Y., Nakano, T. & Lee, K. H. (1991). *J. Nat. Prod*. **54**, 823–829.