C8A—N9A—C13A—C14A	−14.8 (9)
C8A—N9A—C10A—N18A	17.3 (9)
N9A—C10A—N18A—C19A	−156.9 (5)
C10A—N18A—C19A—C20A	−136.9 (6)
C10A—N18A—C19A—C24A	99.7 (6)
C21A—N22A—C25A—C26A	−66.7 (6)
C23A—N22A—C25A—C26A	170.5 (5)
N22A—C25A—C26A—C27A	−168.6 (5)
C25A—C26A—C27A—C28A	64.3 (7)
C29A—C30A—O33A—C34A	177.7 (7)
C4B—C5B—C8B—N9B	−48.9 (8)
C5B—C8B—N9B—C10B	122.3 (6)
C5B—C8B—N9B—C13B	−74.3 (7)
C8B—N9B—C13B—C14B	13 (1)
C8B—N9B—C10B—N18B	−15.4 (9)
N9B—C10B—N18B—C19B	161.9 (5)
C10B—N18B—C19B—C20B	129.0 (6)
C10B—N18B—C19B—C24B	−109.2 (6)
C21B—N22B—C25B—C26B	160.0 (6)
C23B—N22B—C25B—C26B	−77.4 (7)
N22B—C25B—C26B—C27B	−175.8 (5)
C25B—C26B—C27B—C28B	−177.8 (6)
C29B—C30B—O33B—C34B	−4 (1)

The data were collected with a variable scan speed between 1.96 and 29.30° min^{-1}. The scan width was 1° below $K\alpha_1$ and 1° above $K\alpha_2$ with a ratio of total background time to scan time of 1. The intensity data were corrected for the 5% decay. Systematic absences indicated *C2/c* or *Cc* as space group. Although intensity statistics indicated the centrosymmetric space group, structure solution and refinement with full-matrix least squares on F^2 for all reflections resulted in an R value not lower than 0.15. Structure solution and refinement in space group *Cc* converged to $R = 0.0396$. H atoms were calculated at geometrical positions and were allowed to ride on their parent atom.

Data collection: *P2₁ Diffractometer Program* (Syntex, 1975). Cell refinement: *P2₁ Diffractometer Program*. Data reduction: *REDU4* (Stoe & Cie, 1992). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEX2.1* (McArdle, 1994). Software used to prepare material for publication: *PARST* (Nardelli, 1983).

The authors thank Dr J. Tollenaere of Janssen Research Foundation, Beerse, Belgium, for providing a sample of the title compound.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1156). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

McArdle, P. (1994). *J. Appl. Cryst.* **27**, 438–439.
Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.
Richards, D. M., Brogden, R. N., Heel, R. C., Speight, T. M. & Avery, G. S. (1984). *Drugs*, **28**, 38–61.
Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures*. Univ. of Göttingen, Germany.
Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. Univ. of Göttingen, Germany.
Stoe & Cie (1992). *REDU4. Data Reduction Program*. Version 7.03. Stoe & Cie, Darmstadt, Germany.
Syntex (1975). *P2₁ Diffractometer Program*. Version 1. Syntex Analytical Instruments, Cupertino, California, USA.

Acta Cryst. (1995). C**51**, 2135–2137

Methyl 2α,3β,23-Triacetoxyurs-12,18-dien-28-oate

PHILIP J. COX, DAVID G. DURHAM, XIAOJUN LIU AND R. MICHAEL E. RICHARDS

School of Pharmacy, The Robert Gordon University, Schoolhill, Aberdeen AB9 1FR, Scotland

(*Received* 22 *February* 1995; *accepted* 28 *April* 1995)

Abstract

The crystal and molecular structure of the novel triterpenoid methyl 2α,3β,23-triacetoxyurs-12,18-dien-28-oate, $C_{37}H_{54}O_8$, has been determined. There are two molecules in the asymmetric unit, each having a long bond due to steric effects and considerable out-of-plane bending at the C12═C13—C18═C19 chromophores.

Comment

A number of compounds have been isolated from *Rubus pinfaensis* Levl. et Vant, a herb used in Chinese medicine to promote wound healing (Liu, 1994). We have reported previously the structures of two triterpenoids from this source (Cox, Durham, Liu & Richards, 1993, 1994) and now report on a further novel triterpenoid. Overall, the molecule adopts a slightly bow-shaped conformation (Fig. 2) with the β-face concave; the stereochemistry has been established as 2α-OAc, 3β-OAc, 4β-Me, 4α-COAc, 8β-Me, 10β-Me, 14α-Me, 17β-COOMe, 20α-Me. Ring conformations are: *A* chair, *B* chair, *C* distorted C9 sofa, *D* chair, *E* C21 sofa. The C12═C13—C18═C19 torsion angles for the two molecules in the asymmetric unit are −54.2 (6) and −55.1 (6)°, departing considerably from an ideal strain-free *cis* torsion angle of 0°. After 1000 block-diagonal Newton–Raphson iterations (Hypercube Inc, 1994) starting with the atom coordinates of molecule 1, this torsion

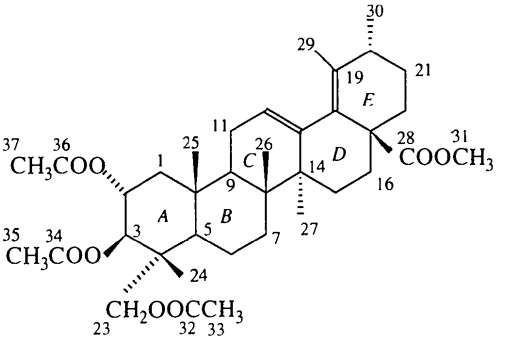

Fig. 1. A schematic view of the molecule showing the numbering scheme used.

angle was lowered to −31.0°. The longest bond is C8—C14, which is 1.598 (5) in molecule 1 and 1.596 (5) Å in molecule 2; the long bond is attributed to steric effects as these two bonded C_{sp^3} atoms have no attached H atoms.

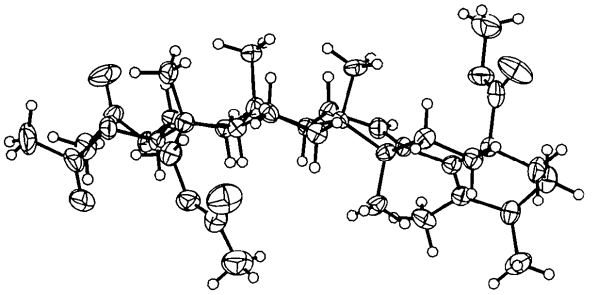

Fig. 2. A side view of the molecule with the displacement ellipsoids shown at the 50% electron probablilty level and H atoms drawn as spheres of arbitrary radii.

Experimental

Glucosyl 2α,3β,23-trihydroxyurs-12,18-dien-28-oate was extracted from the roots of the herb *Rubus pinfaensis* Levl. et Vant. The title compound was obtained by acylation and methylation of the acid obtained by hydrolysis of the natural product.

Crystal data

$C_{37}H_{54}O_8$	Mo $K\alpha$ radiation
M_r = 626.80	λ = 0.71069 Å
Orthorhombic	Cell parameters from 250 reflections
$P2_12_12_1$	θ = 1.88–25.12°
a = 15.243 (6) Å	μ = 0.083 mm^{-1}
b = 15.384 (6) Å	T = 120 (2) K
c = 29.716 (9) Å	Tablet
V = 6968 (4) Å^3	0.30 × 0.28 × 0.22 mm
Z = 8	Colourless
D_x = 1.195 Mg m^{-3}	

Data collection

Delft Instruments FAST diffractometer with Oxford Cryosystem low-temperature device (Cosier & Glazer, 1986)	10439 independent reflections
	4696 observed reflections [$I > 2\sigma(I)$]
	R_{int} = 0.1076
Area detector	θ_{max} = 25.12°
Absorption correction: none	$h = -15 \rightarrow 17$
	$k = -16 \rightarrow 17$
24211 measured reflections	$l = -25 \rightarrow 34$

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max}$ = 1.429
$R[F^2 > 2\sigma(F^2)]$ = 0.0452	$\Delta\rho_{max}$ = 0.211 e Å^{-3}
$wR(F^2)$ = 0.0747	$\Delta\rho_{min}$ = −0.147 e Å^{-3}
S = 0.898	Extinction correction: none
10435 reflections	Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
825 parameters	
H-atom treatment: riding mode, common temperature factors for methyl and non-methyl H atoms	Absolute configuration: Flack (1983) parameter = 1.4 (11)
$w = 1/\sigma^2(F_o^2)$	

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
Molecule 1				
O1	−0.3698 (2)	0.4004 (2)	0.59072 (10)	0.0360 (8)
O2	−0.3478 (2)	0.3159 (3)	0.65169 (12)	0.0770 (13)
O3	−0.2797 (2)	0.2607 (2)	0.55011 (10)	0.0401 (9)
O4	−0.4083 (2)	0.2633 (2)	0.51242 (13)	0.0626 (11)
O5	−0.2024 (2)	0.3631 (2)	0.44376 (11)	0.0453 (9)
O6	−0.0847 (2)	0.3657 (3)	0.39829 (13)	0.0833 (13)
O7	0.0281 (2)	0.8997 (2)	0.58660 (11)	0.0525 (10)
O8	0.1502 (2)	0.8969 (2)	0.54424 (12)	0.0740 (13)
C1	−0.2520 (2)	0.4998 (3)	0.57422 (14)	0.0296 (11)
C2	−0.2759 (2)	0.4033 (3)	0.58139 (15)	0.0316 (12)
C3	−0.2593 (2)	0.3513 (3)	0.5401 (2)	0.0335 (12)
C4	−0.1627 (2)	0.3546 (3)	0.52355 (15)	0.0328 (12)
C5	−0.1355 (2)	0.4508 (3)	0.52062 (14)	0.0268 (11)
C6	−0.0416 (2)	0.4645 (3)	0.50358 (14)	0.0332 (12)
C7	−0.0301 (2)	0.5562 (3)	0.48696 (14)	0.0308 (12)
C8	−0.0501 (2)	0.6262 (3)	0.52211 (14)	0.0264 (11)
C9	−0.1389 (2)	0.6053 (2)	0.54660 (13)	0.0259 (11)
C10	−0.1534 (3)	0.5093 (3)	0.5618 (2)	0.0341 (12)
C11	−0.1567 (3)	0.6725 (3)	0.58254 (15)	0.0340 (12)
C12	−0.1372 (3)	0.7628 (3)	0.56766 (15)	0.0333 (12)
C13	−0.0952 (3)	0.7857 (3)	0.53099 (15)	0.0304 (12)
C14	−0.0569 (3)	0.7193 (3)	0.49855 (14)	0.0298 (12)
C15	0.0354 (2)	0.7493 (3)	0.48337 (15)	0.0354 (12)
C16	0.0407 (3)	0.8446 (3)	0.4697 (2)	0.0372 (13)
C17	0.0082 (3)	0.9061 (3)	0.50699 (15)	0.0319 (12)
C18	−0.0861 (3)	0.8798 (3)	0.51891 (14)	0.0306 (11)
C19	−0.1529 (3)	0.9340 (3)	0.51524 (14)	0.0310 (11)
C20	−0.1424 (3)	1.0301 (3)	0.50503 (15)	0.0377 (12)
C21	−0.0493 (3)	1.0613 (3)	0.5128 (2)	0.0438 (13)
C22	0.0144 (3)	0.9998 (3)	0.4896 (2)	0.0458 (14)
C23	−0.1585 (3)	0.3108 (3)	0.4765 (2)	0.0444 (13)
C24	−0.1034 (2)	0.2975 (3)	0.5535 (2)	0.0404 (13)
C25	−0.0993 (2)	0.4838 (3)	0.60393 (14)	0.0359 (13)
C26	0.0262 (2)	0.6259 (3)	0.55636 (13)	0.0385 (12)
C27	−0.1172 (3)	0.7191 (3)	0.45681 (14)	0.0383 (12)
C28	0.0716 (3)	0.8974 (3)	0.5463 (2)	0.0454 (14)
C29	−0.2481 (2)	0.9061 (3)	0.52104 (15)	0.0439 (13)
C30	−0.1749 (3)	1.0518 (3)	0.4574 (2)	0.055 (2)
C31	0.0831 (3)	0.8922 (4)	0.6260 (2)	0.080 (2)
C32	−0.1588 (4)	0.3897 (3)	0.4075 (2)	0.053 (2)
C33	−0.2080 (3)	0.4541 (4)	0.3819 (2)	0.081 (2)
C34	−0.3572 (3)	0.2259 (3)	0.5360 (2)	0.0444 (14)
C35	−0.3649 (3)	0.1360 (3)	0.5527 (2)	0.068 (2)
C36	−0.3950 (3)	0.3516 (3)	0.6258 (2)	0.0405 (13)
C37	−0.4954 (2)	0.3466 (3)	0.6271 (2)	0.0472 (14)
Molecule 2				
O1′	−0.3518 (2)	1.1308 (2)	0.34287 (10)	0.0370 (8)
O2′	−0.3787 (2)	1.1117 (2)	0.41734 (11)	0.0505 (9)
O3′	−0.4909 (2)	1.0453 (2)	0.30202 (10)	0.0373 (8)
O4′	−0.4934 (2)	1.1311 (2)	0.24012 (12)	0.0708 (13)
O5′	−0.3833 (2)	0.9535 (2)	0.20207 (11)	0.0462 (9)
O6′	−0.4715 (3)	0.9136 (3)	0.14792 (12)	0.133 (2)
O7′	0.0952 (2)	0.5775 (2)	0.32273 (11)	0.0523 (9)
O8′	0.1322 (2)	0.6900 (2)	0.36718 (12)	0.0631 (10)
C1′	−0.2549 (2)	1.0073 (3)	0.33276 (14)	0.0284 (11)
C2′	−0.3494 (2)	1.0365 (3)	0.33669 (14)	0.0286 (11)
C3′	−0.4001 (2)	1.0187 (3)	0.29406 (15)	0.0328 (12)
C4′	−0.4010 (2)	0.9206 (3)	0.28217 (14)	0.0293 (12)
C5′	−0.3036 (2)	0.8882 (3)	0.28185 (14)	0.0310 (12)

C6′	−0.2950 (2)	0.7931 (3)	0.2687 (2)	0.0392 (13)
C7′	−0.2019 (2)	0.7731 (3)	0.25413 (14)	0.0376 (13)
C8′	−0.1350 (2)	0.7929 (3)	0.29075 (14)	0.0276 (11)
C9′	−0.1506 (2)	0.8839 (3)	0.31147 (13)	0.0279 (11)
C10′	−0.2484 (2)	0.9073 (3)	0.32456 (14)	0.0307 (11)
C11′	−0.0845 (2)	0.9008 (3)	0.34968 (15)	0.0383 (13)
C12′	0.0071 (2)	0.8745 (3)	0.33793 (14)	0.0334 (12)
C13′	0.0283 (2)	0.8263 (3)	0.30278 (14)	0.0276 (11)
C14′	−0.0389 (2)	0.7891 (3)	0.26950 (14)	0.0293 (11)
C15′	−0.0145 (2)	0.6941 (3)	0.25739 (15)	0.0352 (12)
C16′	0.0821 (2)	0.6781 (3)	0.24660 (15)	0.0339 (12)
C17′	0.1408 (2)	0.7080 (3)	0.28619 (15)	0.0332 (12)
C18′	0.1241 (3)	0.8056 (3)	0.29322 (15)	0.0330 (12)
C19′	0.1851 (3)	0.8670 (3)	0.2887 (2)	0.0365 (13)
C20′	0.2810 (3)	0.8451 (3)	0.2826 (2)	0.0457 (14)
C21′	0.3000 (3)	0.7508 (3)	0.2967 (2)	0.0496 (15)
C22′	0.2384 (2)	0.6890 (3)	0.2745 (2)	0.0493 (15)
C23′	−0.4402 (2)	0.9133 (3)	0.23454 (14)	0.0374 (13)
C24′	−0.4632 (2)	0.8684 (3)	0.31232 (14)	0.0378 (13)
C25′	−0.2815 (2)	0.8606 (3)	0.36764 (14)	0.0354 (12)
C26′	−0.1413 (2)	0.7221 (3)	0.32800 (14)	0.0386 (12)
C27′	−0.0308 (2)	0.8457 (3)	0.22630 (14)	0.0384 (13)
C28′	0.1213 (3)	0.6599 (3)	0.3297 (2)	0.0398 (13)
C29′	0.1659 (3)	0.9643 (3)	0.2888 (2)	0.0509 (15)
C30′	0.3118 (3)	0.8619 (4)	0.2338 (2)	0.070 (2)
C31′	0.0755 (3)	0.5298 (3)	0.3624 (2)	0.065 (2)
C32′	−0.4046 (4)	0.9468 (4)	0.1601 (2)	0.077 (2)
C33′	−0.3429 (3)	0.9929 (4)	0.1307 (2)	0.094 (2)
C34′	−0.5292 (3)	1.0985 (3)	0.2709 (2)	0.0473 (14)
C35′	−0.6248 (2)	1.1081 (3)	0.2821 (2)	0.055 (2)
C36′	−0.3691 (3)	1.1595 (3)	0.3853 (2)	0.0371 (13)
C37′	−0.3754 (3)	1.2567 (3)	0.3868 (2)	0.0533 (15)

Table 2. *Selected geometric parameters* (Å, °)

Molecule 1		Molecule 2	
O1—C36	1.341 (5)	O1′—C36′	1.362 (5)
O1—C2	1.458 (4)	O1′—C2′	1.463 (5)
O2—C36	1.187 (5)	O2′—C36′	1.211 (5)
O3—C34	1.364 (5)	O3′—C34′	1.365 (5)
O3—C3	1.458 (5)	O3′—C3′	1.463 (4)
O4—C34	1.196 (5)	O4′—C34′	1.177 (5)
O5—C32	1.331 (5)	O5′—C32′	1.294 (5)
O5—C23	1.429 (5)	O5′—C23′	1.438 (5)
O6—C32	1.220 (5)	O6′—C32′	1.196 (5)
O7—C28	1.370 (5)	O7′—C28′	1.345 (5)
O7—C31	1.445 (5)	O7′—C31′	1.420 (5)
O8—C28	1.199 (5)	O8′—C28′	1.217 (5)
C12—C13	1.312 (5)	C12′—C13′	1.321 (5)
C18—C19	1.321 (5)	C18′—C19′	1.332 (5)
C13—C12—C11	126.5 (4)	C13′—C12′—C11′	124.4 (4)
C12—C13—C18	120.2 (4)	C12′—C13′—C18′	120.0 (4)
C12—C13—C14	122.3 (4)	C12′—C13′—C14′	123.8 (4)
C18—C13—C14	117.5 (4)	C18′—C13′—C14′	116.3 (4)
C19—C18—C13	124.0 (4)	C19′—C18′—C13′	122.7 (4)
C19—C18—C17	122.4 (4)	C19′—C18′—C17′	124.3 (4)
C13—C18—C17	113.4 (3)	C13′—C18′—C17′	112.9 (4)
C18—C19—C20	123.4 (4)	C18′—C19′—C20′	121.9 (5)
C18—C19—C29	123.2 (4)	C18′—C19′—C29′	124.2 (4)
C20—C19—C29	113.4 (4)	C20′—C19′—C29′	113.8 (4)

Absence of crystal decay in the X-ray beam was confirmed by checking equivalent reflections at the beginning and end of the data collection, which lasted about 8 h. The two largest (Δ/σ) values of 1.429 and 0.382 are associated with the methyl H atoms and indicate that these atoms, attached to C33′ and C31, may be in free rotation. All non-H atoms were given anisotropic displacement parameters; the methylene and methyl H atoms were given common isotropic displacement parameters and were allowed to ride on their attached C atoms.

The structure was solved using *SIR*92 (Altomare *et al.*, 1994) in the *CRYSTAN* (Edwards, Gilmore, Mackay & Stewart, 1995) system. Refinement was completed with *SHELXL*93 (Sheldrick, 1993) and the molecular plot was obtained using *SNOOPI* (Davies, 1983).

We thank the SERC X-ray Crystallographic Service at The University of Wales, Cardiff, for collecting the data, and Dr C. J. Gilmore for assistance in solving the structure.

Lists of structure factors, torsion angles, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BM1010). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435–436.
Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–108.
Cox, P. J., Durham, D. G., Liu, X. & Richards, R. M. E. (1993). *J. Crystallogr. Spectrosc. Res.* **23**, 779–783.
Cox, P. J., Durham, D. G., Liu, X. & Richards, R. M. E. (1994). *J. Chem. Res.* (*S*), p. 122; *J. Chem. Res.* (*M*), pp. 843–860.
Davies, K. (1983). *SNOOPI. Molecular Plotting Program.* Chemical Crystallography Laboratory, Univ. of Oxford, England.
Edwards, C., Gilmore, C. J., Mackay, S. & Stewart, N. (1995). *CRYSTAN* 6.3. *A Computer Program for the Solution and Refinement of Crystal Structures.* MacScience, Japan.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Hypercube Inc. (1994). *HYPERCHEM.* Release 4 for *Windows.* Hypercube Inc., Waterloo, Ontario, Canada.
Liu, I. X. (1994). PhD thesis, The Robert Gordon Univ., Aberdeen, Scotland.
Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures.* Univ. of Göttingen, Germany.

Acta Cryst. (1995). C**51**, 2137–2139

2-(Phenylmethylthio)benzaldehyde

WING-TAK WONG AND SIU-MING LEE

Department of Chemistry, The University of Hong Kong, Pokfulam Road, Hong Kong

(*Received* 4 *January* 1995; *accepted* 9 *May* 1995)

Abstract

In the title compound, $C_{14}H_{12}OS$, the two planar benzaldehyde and benzyl groups are inclined at 77.7 (1)°. The torsion angle about the central C—S bond is 174.9 (2)°. The molecules are held together in the crystal by van der Waals interactions.

Comment

As part of our program to design and study chelating ligands containing both O and S donors (Wong, Lee & Cheung, 1995), we were interested in the coordination properties of the title compound, (I).