Discussion. Fig. 1 shows the molecule and numbering scheme and Fig. 2 a stereoscopic view of the unit cell. Table 1* lists atom parameters; Table 2 gives bond distances and angles.

The fused five-membered rings, C(2)–C(3)–O(3)–C(7)–O(2) and C(2)–C(3)–C(4)–O(4)–C(1), both have the envelope conformation with maximum distances from the best least-squares plane for each ring of −0·205 (4) Å for C(7) and 0·247 (2) Å for O(4), while the largest deviation from the best plane in the third ring, C(5)–C(6)–O(6)–C(10)–O(5), is 0·210 (4) Å for C(6). The obtuse angle between the fused rings is 115·9 (4)°, calculated as the angle between the best planes formed by C(2),C(3) and the atoms bonded directly to them [*cf.* 5-hydroxy-1,2-*O*-isopropylidene-α-D-*xylo*-hexofuranurono-6,3-lactone (Sheldrick *et al.*, 1983)]. The single hydroxyl group forms a weak hydrogen bond, O(1) to O(5)($x-1, y, z$) with an O···O distance of 3·014 (7) Å, linking the molecules in the *a*-axis direction only.

C—O bond distances in the isopropylidene rings are nearly equal, only O(2)–C(7), at 1·439 (4) Å, having a value greater than 3σ from the average.

We wish to thank the University Computing Service of the University of Leeds for the provision of facilities.

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[*] Lists of structure amplitudes, anisotropic thermal parameters, H-atom parameters and least-squares-planes' details have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39870 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Acta Cryst. (1985). C**41**, 433–436

Structure of a Diterpene from the *Austroeupatorium inulaefolia* (HBK) K et R: 2,18-Diacetoxy-15,16-epoxy-3-hydroxy-12-oxo-19-norlabdane-8(17),13(16),14-triene Monohydrate, $C_{23}H_{30}O_7.H_2O$

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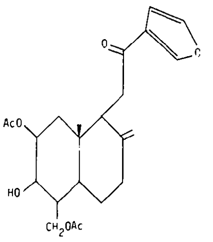
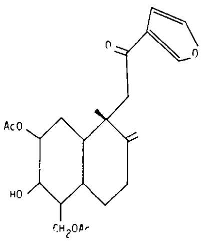
Abstract. $M_r = 436{\cdot}50$, orthorhombic, $P2_12_12_1$, $a = 14{\cdot}076$ (3), $b = 21{\cdot}088$ (5), $c = 7{\cdot}822$ (2) Å, $V = 2321{\cdot}8$ Å^3, $Z = 4$, $D_x = 1{\cdot}249$, $D_m = 1{\cdot}246$ (5) g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}54$ cm^{-1}, $F(000) = 936$, $T = 296$ K. Final $R = 0{\cdot}067$ for 1235 unique observed reflections. The crystalline structure consists of discrete molecules situated almost parallel to the *xz* plane forming layers at $y = 0$ and $\frac{1}{2}$, and channels running parallel to **c** where the water molecules are trapped by hydrogen bonds with three different diterpene molecules. The bicyclic system has the chair conformation with all substituents in *cis* positions.

Introduction. *Austroeupatorium inulaefolia* (HBK) K et R was found to be rich in a new series of diterpenes with norlabdane skeletons (Bahsas, 1979). Chemical and spectroscopic studies suggested two possible structures (*A* and *B*) for one of these compounds. The present crystallographic study was undertaken to provide information on its structure and to determine if structure *B* was present.



(*A*)



(*B*)

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Experimental. Density determined by flotation in a solution of chlorobenzene and bromobenzene. Plate-like crystal, $0{\cdot}25 \times 0{\cdot}20 \times 0{\cdot}15$ mm, automated four-circle Philips PW 1100 diffractometer, lattice parameters determined by a least-squares procedure applied to setting angles of 25 strong reflections in range $6{\cdot}02 < \theta < 9{\cdot}11°$. Intensity data up to $(\sin\theta)/\lambda = 0{\cdot}605$ Å^{-1} measured with graphite-monochromated Mo $K\alpha$ radiation. After Lp corrections, initial 2349 reflections reduced to 1235 unique data with $I > 3\sigma(I)$ and 1114 unobserved reflections. Ranges of h, k and l $0 \le h \le 16$, $0 \le k \le 25$ and $\underline{0} \le l \le 9$. No absorption correction applied. 3 standard reflections, 1% intensity variation.

Structure solved by direct methods. The phase solution with the highest 'combined figure of merit' obtained from *MULTAN*77 (Main, Lessinger, Woolfson, Germain & Declercq, 1977) for 300 reflections with $E > 1{\cdot}42$ gave an E map from which positions of all ring atoms were obtained. Terminal atoms found by successive Fourier syntheses. Structure refined by full-matrix least squares. H atoms placed in geometrically calculated positions with exceptions of H_2O—, $=CH_2$— and OH— H atoms which were located from a difference map. In final cycles only five terminal atoms assigned anisotropic thermal parameters, H atoms refined as rigid groups with the constraint C—H = 1·00 Å, with one overall isotropic temperature factor. The isotropic temperature factors for the H atoms of the water molecule were set equal to that of O(7). Final convergence gave $R = 0{\cdot}067$, $R_w = 0{\cdot}068$, $(\Delta/\sigma)_{\max}$ 0·019. The absolute configuration could not be determined since another run with two additional cycles having all positional parameters inverted converged to the same final R. Max. and min. heights in final difference Fourier synthesis 0·30 and $-0{\cdot}26$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallogrpahy* (1968). Function minimized $\sum w(|F_o| - |F_c|)^2$ where $w = k/\sigma^2(F)$; k converged to 2·354. All calculations performed with *SHELX*77 (Sheldrick, 1977) on a Burroughs B 5900 computer at the Universidad de Los Andes.

Discussion. Positional and isotropic thermal parameters and the resulting bond lengths and angles are given in Tables 1 and 2 respectively.*

The structure consists of discrete molecules of formula A situated almost parallel to the xz plane forming layers at $y = 0$ and $\frac{1}{2}$, and channels running parallel to **c** at $(x = \frac{1}{4}, y = 0)$ and $(x = \frac{3}{4}, y = \frac{1}{2})$ where the water molecules are trapped by hydrogen bonds.

Table 1. *Atom coordinates* ($\times 10^4$) *and isotropic temperature factors* (Å$^2 \times 10^3$)

	x	y	z	U
C(1)	5989 (5)	681 (4)	5352 (11)	43 (2)
C(2)	4916 (5)	670 (4)	5041 (11)	43 (2)
C(3)	4357 (5)	556 (3)	6678 (10)	38 (2)
C(4)	4655 (5)	996 (3)	8152 (11)	40 (2)
C(5)	5734 (5)	943 (3)	8421 (10)	35 (2)
C(6)	6086 (6)	1279 (4)	10046 (11)	50 (2)
C(7)	7116 (6)	1109 (4)	10427 (11)	51 (2)
C(8)	7744 (5)	1207 (4)	8919 (11)	42 (2)
C(9)	7404 (5)	897 (4)	7268 (10)	38 (2)
C(10)	6359 (5)	1095 (3)	6822 (10)	37 (2)
C(11)	8098 (6)	978 (4)	5785 (11)	50 (2)
C(12)	9038 (5)	657 (4)	5988 (11)	45 (2)
C(13)	9853 (5)	903 (4)	5108 (12)	46 (2)
C(14)	10795 (7)	662 (4)	5155 (14)	63 (3)
C(15)	11314 (6)	1002 (6)	4095 (17)	87 *
C(16)	9887 (6)	1395 (5)	4007 (15)	80*
C(17)	8516 (6)	1550 (4)	9045 (12)	64 (3)
C(18)	4275 (5)	1678 (4)	7898 (11)	49 (2)
C(19)	6354 (5)	1808 (4)	6351 (11)	48 (2)
C(20)	3956 (6)	1288 (4)	3130 (12)	49 (2)
C(21)	3686 (7)	1952 (5)	2709 (13)	72*
C(22)	2690 (6)	2111 (4)	7759 (13)	55 (3)
C(23)	1707 (7)	2014 (5)	8302 (17)	89*
O(2)	4637 (3)	1274 (2)	4310 (7)	47 (2)
O(3)	3369 (4)	596 (2)	6337 (7)	49 (2)
O(12)	9117 (4)	186 (3)	6905 (8)	60 (2)
O(15)	10773 (5)	1478 (4)	3330 (12)	109*
O(18)	3274 (4)	1675 (3)	8510 (7)	52 (2)
O(20)	3607 (4)	828 (3)	2466 (8)	62 (2)
O(22)	3002 (5)	2520 (3)	6845 (9)	82 (2)
O(7)	2695 (4)	−444 (3)	4481 (9)	74 (2)

* Equivalent isotropic temperature factors; $U_{eq} = (U_{11} + U_{22} + U_{33})/3$.

Table 2. *Bond lengths* (Å) *and angles* (°)

C(1)–C(2)	1·521 (11)	C(10)–C(19)	1·550 (12)
C(1)–C(10)	1·538 (12)	C(11)–C(12)	1·500 (12)
C(2)–C(3)	1·529 (13)	C(12)–O(12)	1·229 (9)
C(2)–O(2)	1·450 (9)	C(12)–C(13)	1·438 (11)
C(3)–C(4)	1·536 (12)	C(13)–C(14)	1·424 (11)
C(3)–O(3)	1·424 (9)	C(13)–C(16)	1·339 (12)
C(4)–C(5)	1·538 (11)	C(14)–C(15)	1·312 (13)
C(4)–C(18)	1·545 (12)	C(15)–O(15)	1·395 (12)
C(5)–C(6)	1·533 (12)	C(16)–O(15)	1·361 (11)
C(5)–C(10)	1·557 (12)	C(18)–O(18)	1·468 (9)
C(6)–C(7)	1·519 (11)	C(22)–O(18)	1·362 (10)
C(7)–C(8)	1·490 (11)	C(22)–C(23)	1·466 (12)
C(8)–C(9)	1·522 (12)	C(22)–O(22)	1·207 (10)
C(8)–C(17)	1·312 (11)	C(20)–O(2)	1·335 (10)
C(9)–C(10)	1·571 (11)	C(20)–O(20)	1·205 (10)
C(9)–C(11)	1·531 (12)	C(20)–C(21)	1·487 (12)

C(2)–C(1)–C(10)	117·7 (8)	C(1)–C(10)–C(9)	109·6 (7)
C(1)–C(2)–C(3)	111·9 (8)	C(9)–C(10)–C(19)	108·6 (7)
C(3)–C(2)–O(2)	109·2 (7)	C(1)–C(10)–C(19)	111·4 (7)
C(1)–C(2)–O(2)	108·6 (7)	C(9)–C(11)–C(12)	115·3 (8)
C(2)–C(3)–O(3)	109·5 (7)	C(11)–C(12)–C(13)	119·2 (8)
C(2)–C(3)–C(4)	113·5 (7)	C(11)–C(12)–O(12)	120·6 (8)
C(4)–C(3)–O(3)	112·0 (7)	C(13)–C(12)–O(12)	120·2 (8)
C(3)–C(4)–C(5)	109·0 (7)	C(12)–C(13)–C(14)	126·8 (9)
C(3)–C(4)–C(18)	113·6 (7)	C(14)–C(13)–C(16)	105·0 (9)
C(5)–C(4)–C(18)	114·8 (7)	C(12)–C(13)–C(16)	128·2 (9)
C(4)–C(5)–C(6)	113·5 (7)	C(13)–C(14)–C(15)	107·7 (10)
C(6)–C(5)–C(10)	112·7 (6)	C(14)–C(15)–O(15)	111·0 (9)
C(4)–C(5)–C(10)	115·8 (7)	C(13)–C(16)–O(15)	112·5 (9)
C(5)–C(6)–C(7)	111·2 (8)	C(4)–C(18)–O(18)	107·5 (7)
C(6)–C(7)–C(8)	112·1 (8)	C(18)–O(18)–C(22)	117·1 (7)
C(9)–C(8)–C(17)	124·1 (9)	O(2)–C(20)–O(20)	124·9 (9)
C(7)–C(8)–C(9)	114·8 (7)	C(21)–C(20)–O(20)	123·9 (9)
C(7)–C(8)–C(17)	121·1 (10)	C(21)–C(20)–O(2)	111·2 (8)
C(8)–C(9)–C(11)	113·6 (7)	O(18)–C(22)–O(22)	121·1 (9)
C(10)–C(9)–C(11)	113·3 (7)	C(23)–C(22)–O(22)	128·3 (10)
C(8)–C(9)–C(10)	111·6 (7)	C(23)–C(22)–O(18)	110·5 (9)
C(5)–C(10)–C(9)	107·2 (7)	C(2)–O(2)–C(20)	119·2 (7)
C(1)–C(10)–C(5)	107·1 (6)	C(15)–O(15)–C(16)	103·8 (9)
C(5)–C(10)–C(19)	112·9 (7)		

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* Lists of anisotropic thermal parameters, H-atom coordinates and their isotropic temperature factors, mean-plane calculations and structure factors have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39680 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The molecule has a bicyclic system with the chair conformation (Fig. 1) with all substituents in *cis* positions. The furan ring is planar ($\chi^2 = 2{\cdot}7$), similar to what has been observed earlier (Eguren, Fayos & Perales, 1981; Jefferies, Payne, Raston & White, 1981; Kazlauskas, Murphy, Wells, Noack, Oberhänsli & Schönholzer, 1979; Savona, Passannanti, Paternostro, Piozzi, Hanson, Hitchcock & Siverns, 1978; McEachan, McPhail & Sim, 1966). The shortest $C(sp^3)$—$C(sp^3)$ distance, C(6)—C(7) = 1·519 (11) Å, could be a consequence of the C(8)=C(17) double bond, while the longest ones, 1·57 (1) and 1·56 (1) Å, for C(9)—C(10) and C(5)—C(10) are characteristic of all diterpenes so far reported (Eguren *et al.*, 1981; Jefferies *et al.*, 1981; Kazlauskas *et al.*, 1979; Savona *et al.*, 1978; McEachan *et al.*, 1966). The bond angles in the bicyclic system are in the range 107·1–117·7° with mean value 111·7°. The ones that deviate most from the tetrahedral value are C(2)—C(1)—C(10) and C(9)—C(8)—C(7) which are 117·7 and 114·8° respectively.

Comparison of the hydroxy diterpene reported here (I) and the diterpene acid with a similar skeleton, isolated from *Dodonaea Petiolaris* (II) (Jefferies *et al.*, 1981), shows that the C(9) and C(10) substituents are *trans* to those at C(3) in (II), while the substituents in (I) are all in *cis* positions. The —CO—C_4H_3O moieties present in both molecules are differently oriented along the C(9)—C(11) bond. The intermolecular hydrogen bond present in the diterpene acid (II) crystal structure, involving the ketone oxygen of the —CO—C_4H_3O fragment, could be the main factor for the observed difference. All bond distances and angles are similar within 3σ, except for the angles C(2)—C(1)—C(10) of 117·7 (8) *vs* 109·5 (7)°, C(8)—C(9)—C(10) of 111·6 (7) *vs* 107·4 (6)° and C(7)—C(8)—C(9) of 114·8 (7) *vs* 111·7 (7)°, in compounds (I) and (II) respectively. The wider angles reported for (I) can be ascribed to steric effects due to the *cis* position of all bicyclic system substituents. This effect can also be responsible for the more relaxed chair conformation of structure (I) which has torsion angles, systematically smaller than those present in (II), with mean absolute values of 51·6 and 54·3° *vs* 56·6 and 59·0° for *A* and *B* ring torsion angles respectively.

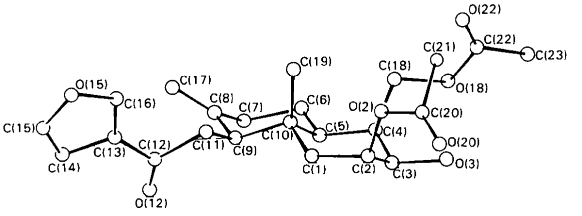

Fig. 1. A perspective view of the molecule showing the labeling of the atoms and the chair conformation of the bicyclic system.

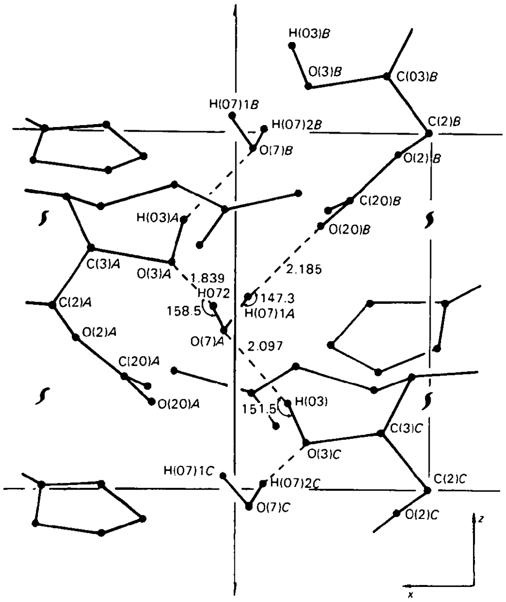

Fig. 2. (010) projection of the layer content of one half unit cell at $y = 0$, showing the symmetry elements that are on the plane. The numbered atoms are those which belong to the 13-membered rings that hold together the molecules and are responsible for the layer formation. Molecules *A*, *B* and *C* are related to the coordinates of Table 1 by the following operations: $A(x, y, z)$, $B(0{\cdot}5 - x, -y, 0{\cdot}5 + z)$ and $C(0{\cdot}5 - x, -y, z - 0{\cdot}5)$. Hydrogen-bond distances in Å, angles in °. E.s.d.'s: for distances 0·020 Å, for angles 0·2°.

The layers at $y = 0$ and $\frac{1}{2}$ consist of molecules related by a 2_1 (*c*) axis held together by hydrogen bonds with water molecules situated alongside the screw axis. Fig. 2 displays the (010) projection of the layer content at $y = 0$. Each water molecule (Fig. 2) forms three different hydrogen bonds with three diterpene molecules (*A*, *B* and *C*).

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Acta Cryst. (1985). C**41**, 436–439

(*E*,*E*)-8-Acetoxygermacra-1(10),4-dien-6,12-olide, $C_{17}H_{22}O_4$

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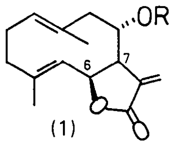
Abstract. $M_r = 290\cdot36$, monoclinic, $P2_1$, $a = 7\cdot151$ (2), $b = 16\cdot421$ (3), $c = 6\cdot772$ (1) Å, $\beta = 98\cdot18$ (1)°, $V = 787\cdot4$ (2) Å^3, $Z = 2$, $D_x = 1\cdot225$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1\cdot54178$ Å, $\mu = 7\cdot11$ cm^{-1}, $F(000) = 312$, $T = 298$ K, $R = 0\cdot047$ for 986 independent reflections. The title compound, isolated from *Gochnatia foliolosa* (Compositae), contains a lactone ring with unusual *cis* fusion to the 6,7 positions of the ten-membered germacradiene ring. The *cis* fusion results in the ten-membered ring adopting a chair–boat conformation with the methyl groups *syn* and lying below the plane of the ten-membered ring. Molecular mechanics calculations are reported for the four known *cis*-fused germacranolides.

Introduction. The tribe Mutiseae is one of the least known tribes in the family Compositae, and its small area of distribution implies the tribe is of relatively recent evolutionary origin. The tribe is predominately American with fewer than half of the genera and less than a quarter of the species found elsewhere. Gochnatiinae is the most fragmented and widely distributed subtribe with 21 genera in the Americas and 15 in the Old World. The genus *Gochnatia* is composed of 68 species of which only five (Bohlmann, Zdero, Robinson & King, 1981; Bohlmann, Schmeda-Hirschman, Jakupovic, King & Robinson, 1984; Bohlmann & Zdero, 1979; Dominguez, Franco, Cano & Wolzak, 1978; Bohlmann, Jakupovic, Robinson & King, 1981) have been investigated chemically.

The discovery in *Gochnatia foliolosa* (D. Don) D. Don ex. et A. var. *foliolosa* of the germacranolide (1) (R = acetyl), in which the lactone ring has closed *cis* at the 6 position of the germacradiene ring may be of taxonomic significance. This molecular configuration was reported for compounds (2) and (3) isolated from the genus *Montanoa* (Herz, Govindan & Blount, 1980; Bohlmann & Le Van, 1978) of the tribe Heliantheae and compound (4) isolated from *Ursinia anthemoides* (Samek, Holub, Rychlewska, Grabarczyk & Drozdz, 1979; Rychlewska, 1981). The classification of *Ursinia* has been of some dispute (Heywood & Humphries, 1977). The distribution of the *cis*-fused germacranolides within the genus, the subtribe and the tribe may assist in clarifying taxonomic problems. The germacranolide structures assigned in earlier studies of the *Gochnatia* will be reexamined.



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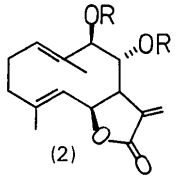

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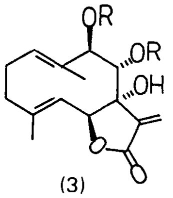

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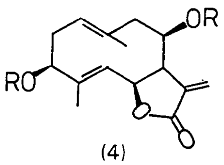

(4)

Experimental. Extracts of *Gochnatia foliolosa* collected in Florida near Concepción, Chile, yielded a crystalline solid, m.p. 388 K, $\lambda(\mathrm{MeOH})_{\max} = 215$ nm (ε 19·72), $\nu(\mathrm{Nujol})_{\max} = 3360$, 2950, 2860, 1760, 1715, 1650,

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