Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
4426 measured reflections
4179 independent reflections
1929 observed reflections [$F > 3\sigma(F)$]
$R_{\rm int} = 0.007$
$\theta_{\rm max} = 30°$
$h = -11 \rightarrow 11$
$k = 0 \rightarrow 12$
$l = -15 \rightarrow 15$
3 standard reflections frequency: 100 min intensity variation: 4%

Refinement

Refinement on F
$R = 0.057$
$wR = 0.057$
$S = 1.56$
1929 reflections
232 parameters
Only coordinates of H atoms refined
$w = 1/[\sigma^2(F) + (0.02F_o^2)^2]$
$(\Delta/\sigma)_{\rm max} = 0.003$
$\Delta\rho_{\rm max} = 0.251$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.51$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

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

	x	y	z	$U_{\rm eq}$
S	0.8820 (2)	0.2169 (2)	0.8417 (1)	0.046 (1)
Cl	1.3380 (2)	0.0960 (2)	0.2376 (1)	0.072 (1)
O1	0.7451 (5)	0.3181 (5)	0.7497 (3)	0.056 (2)
O2	0.8218 (5)	0.1647 (4)	0.9615 (3)	0.058 (2)
N1	1.2440 (6)	−0.1234 (6)	0.6902 (4)	0.042 (2)
C2	1.1075 (8)	0.0202 (7)	0.6893 (5)	0.040 (3)
C2α	1.3390 (8)	−0.1488 (7)	0.5827 (5)	0.039 (3)
N2	1.0112 (7)	0.0425 (5)	0.7985 (4)	0.048 (2)
C3	1.0544 (8)	0.1435 (7)	0.5802 (6)	0.047 (3)
C3α	1.2894 (8)	−0.0233 (7)	0.4760 (5)	0.038 (3)
N4	1.1427 (7)	0.1234 (6)	0.4767 (4)	0.051 (2)
C5	1.3932 (8)	−0.0533 (7)	0.3672 (5)	0.045 (3)
C6	1.5372 (9)	−0.2024 (9)	0.3671 (6)	0.055 (3)
C7	1.5831 (9)	−0.3279 (8)	0.4711 (7)	0.058 (3)
C8	1.4886 (9)	−0.3021 (8)	0.5768 (6)	0.057 (3)
C9	1.0297 (8)	0.3075 (7)	0.8524 (5)	0.049 (3)
C10	1.1863 (9)	0.2284 (8)	0.9262 (5)	0.051 (3)
C11	1.2996 (9)	0.3013 (8)	0.9362 (5)	0.052 (3)
C12	1.2582 (8)	0.4607 (7)	0.8726 (5)	0.043 (3)
N12	1.3696 (9)	0.5354 (7)	0.8849 (5)	0.063 (3)
C13	1.1011 (9)	0.5398 (7)	0.7986 (6)	0.057 (3)
C14	0.9893 (9)	0.4656 (8)	0.7878 (6)	0.057 (3)

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

S—O1	1.416 (4)	C2α—C8	1.409 (9)
S—O2	1.436 (4)	C3α—C5	1.418 (8)
S—N2	1.643 (5)	C5—C6	1.355 (8)
S—C9	1.729 (6)	C6—C7	1.395 (9)
Cl—C5	1.704 (6)	C7—C8	1.353 (9)
N1—C2	1.297 (7)	C9—C10	1.374 (9)
N1—C2α	1.372 (7)	C9—C14	1.386 (9)
C2—C3	1.423 (7)	C10—C11	1.357 (9)
C2—N2	1.390 (7)	C11—C12	1.392 (9)
C3—N4	1.307 (8)	C12—C13	1.377 (8)
N4—C3α	1.351 (8)	C12—N12	1.363 (8)
C2α—C3α	1.414 (8)	C13—C14	1.358 (9)
O1—S—O2	118.8 (2)	C3α—C5—C6	119.3 (5)
O1—S—N2	108.3 (2)	C6—C5—Cl	120.0 (5)
O2—S—N2	102.9 (2)	C5—C6—C7	121.3 (6)
O1—S—C9	109.4 (2)	C6—C7—C8	120.7 (6)
O2—S—C9	110.1 (2)	C7—C8—C2α	120.5 (6)
C2—N1—C2α	117.0 (5)	S—C9—C10	121.7 (5)
N1—C2—C3	121.3 (5)	S—C9—C14	120.2 (5)
N1—C2—N2	116.1 (5)	C10—C9—C14	118.1 (6)
N2—C2—C3	122.5 (5)	S—N2—C2	127.6 (4)
C2—C3—N4	123.2 (6)	C9—C10—C11	121.5 (6)
C3—N4—C3α	116.8 (5)	C10—C11—C12	120.5 (6)
N4—C3α—C2α	120.5 (5)	C11—C12—C13	117.9 (6)
N4—C3α—C5	120.0 (5)	C11—C12—N12	120.6 (6)
N1—C2α—C3α	121.2 (5)	C13—C12—N12	121.6 (6)
N1—C2α—C8	120.1 (5)	C12—C13—C14	121.4 (6)
C3α—C2α—C8	118.7 (5)	C13—C14—C9	120.6 (6)
C2α—C3α—C5	119.5 (5)	N2—S—C9	106.6 (3)
C3α—C5—Cl	120.6 (5)		

Data were collected with the diffractometer mounted on a Rigaku 100 PL with a molybdenum anode operating at 57.5 kV and 90 mA using a 0.5 × 5 mm filament and an Nb filter. The structure was solved by direct methods using the program *SHELXS*86 (Sheldrick, 1985) and refined using *SHELX*76 (Sheldrick, 1976).

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: HA1087). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Eupenifeldin

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Abstract

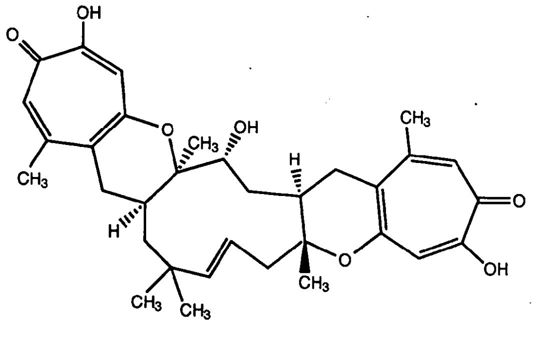
Eupenifeldin, 4,16,19-trihydroxy-1,6a,10,10,13,18a-hexamethyl-3,6a,7,10,11,11a,12,15,18a,19,20,20a,21-dodecahydro-1*H*-dicyclohepta[*e*:*e*′]cycloundeca[1,2-*b*:5,6-*b*′]dipyran-3,15-dione, $C_{33}H_{40}O_7$, is a novel cytotoxic

bistropolone, whose structure has been determined by single-crystal analysis. Intramolecular hydrogen bonds are observed within the tropolones. One dihydropyran ring is *trans* fused to the central eleven-membered ring, the other is *cis* fused. The two crystallographically independent molecules adopt similar conformations. A double-ribbon structure exists in the crystal.

Comment

Tropolone and its derivatives have been extensively studied for their interesting aromatic character and intramolecular hydrogen bonds (Shimanouchi & Sasada, 1973; Sekiya, Nishimura, Mori, Takeshita & Nishiyama, 1990). However, only three other naturally occurring bistropolones have been reported (Baggaley & Norin, 1968; Görler, Moll, Siehl, Strähle & Westphal, 1982; Itô, 1979). X-ray structures of these molecules have not been determined, or were determined with limited precision, because of difficulties in growing crystals suitable for X-ray diffraction experiments (Itô, Arai, Ohashi & Sasada, 1981; Görler, Moll, Siehl, Strähle & Westphal, 1982).

The title compound, (I), was isolated from cultures of *Eupenicillium brefeldianum* using extraction and recrystallization (Mayerl *et al.*, 1993). It is cytotoxic against the HCT-116 cell line and has *in vivo* antitumor activity in the P388 leukemia model. Spectroscopic studies (FAB, UV, IR, COSY, HMBC, COLOC) identified the compound as bistropolone, but only yielded a partial structure. The locations of two ether linkages and the configurations at the stereocenters were not established. Finally, single-crystal X-ray analysis was performed to determine the complete structure, including relative stereochemistry, but not the absolute configuration.



(I)

The two crystallographically independent molecules (hereafter molecules *A* and *B*) in the asymmetric unit adopt similar conformations. However, statistically significant differences in corresponding torsion angles between the two molecules, particularly the torsion angles involving the atoms of the tropolones, were observed. The dihedral angle between the two tropolone rings is 125.4 (1)° for molecule *A* and 118.4 (2)° for molecule *B*. The junctions between each of the two dihydropyran rings and the eleven-membered ring are different. One of the dihydropyran rings is *trans* fused and the other is *cis*, but both adopt twist conformations. In the eleven-membered ring, the configuration at the C=C double bond is *trans*.

The four seven-membered rings are essentially planar with minor distortions. The largest deviation from each of the least-squares planes is about 0.1 Å. Three of the exocyclic O and C atoms are significantly displaced from their respective least-squares planes: O8(*A*) 0.303 (6), C30(*A*) 0.220 (7) and O9(*B*) 0.183 (5) Å. The bond lengths in each seven-membered ring alternate, as was also found in previous studies (Barrow & Mills, 1973; Schaefer & Reed, 1971; Watkin & Hamor, 1971), and follow the same pattern. The mean bond length is 1.38 (2) Å for formal C=C bonds and 1.43 (2) Å for formal C—C bonds. The lengths of the four C=O bonds, 1.257 (8)–1.262 (8) Å, are marginally longer than the 1.225 (5) Å found in X-ray diffraction studies of molecules containing tropolones (Shaefer & Reed, 1971) but are in good agreement with the value of 1.257 Å in the crystal structure of tropone at 213 K (Barrow & Mills, 1973). The C—OH bonds of the tropolones range from 1.320 (8) to 1.366 (8) Å, compared to 1.342 (4) Å in 5-isopropyltropolone (Sekiya, Nishimura, Mori, Takeshita & Nishiyama, 1990). Hydrogen bonds were observed within each troplone of molecule *A* between the carbonyl O atom and the hydroxyl group. The hydroxyl H atoms of molecule *B* were not observed in difference Fourier maps.

The crystal structure is constructed from a double-ribbon network (Fig. 2). Each ribbon is infinite along the *b* axis and consists of molecules *A* and *B* linked by three independent hydrogen bonds, O9(*A*)···O24(*B*), O24(*A*)···O1(*B*) and O25(*A*)···O1(*B*). A weak hydrogen bond between two *A* molecules, O1(*A*)···O8(*A*), then holds two neighboring ribbons to form a double ribbon, with the polar sides of molecules face-to-face. *B* molecules are along the two edges of the double ribbon, while *A* molecules, polymerized *via* the O1···O8 hydrogen bonds, are in the middle.

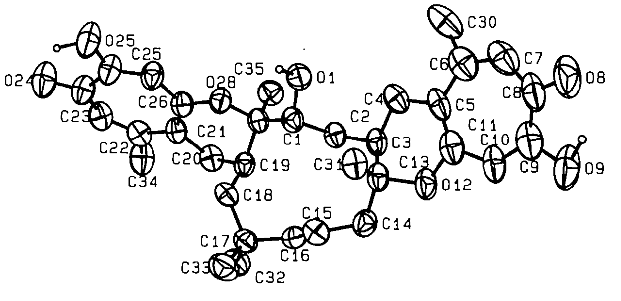
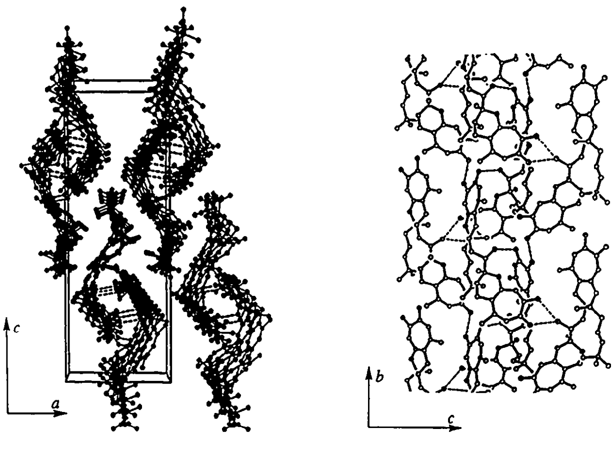


Fig. 1. The molecular structure of (I), showing the atom numbering, with displacement ellipsoids drawn at the 50% probability level.



(a) (b)

Fig. 2. View of the molecular packing in the crystal (*a*) down the *b* axis and (*b*) down the *a* axis, detailing hydrogen-bond interactions.

Experimental

Pale yellow thin plates were first obtained by recrystallization from $CHCl_3$/MeOH. Only unit-cell constants were determined from these crystals since they were unstable in the air, even in the solvent, and became useless within a few of hours. In order to obtain crystals suitable for X-ray analysis, many different solvents were tested for recrystallization. Orange prismatic crystals were finally grown in clusters from $CHCl_3$/EtOH, which were in different crystal forms and stable at room temperature.

Crystal data

$C_{33}H_{40}O_7$
$M_r = 548.676$
Orthorhombic
$P2_12_12_1$
$a = 11.519\ (1)$ Å
$b = 15.600\ (1)$ Å
$c = 32.258\ (2)$ Å
$V = 5796.8\ (8)$ Å^3
$Z = 8$
$D_x = 1.257$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 20.49$–$34.18°$
$\mu = 0.67$ mm^{-1}
$T = 290$ K
Prismatic
0.50 × 0.25 × 0.25 mm
Orange

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical (ψ scans)
$T_{\min} = 0.9725$, $T_{\max} = 0.9993$
6326 measured reflections
4840 independent reflections
3811 observed reflections [$I \geq 3\sigma(I)$]

$R_{\rm int} = 0.012$
$\theta_{\max} = 60°$
$h = 0 \rightarrow 12$
$k = -17 \rightarrow 12$
$l = -36 \rightarrow 0$
3 standard reflections
frequency: 120 min
intensity variation: -0.0011% h^{-1}

Refinement

Refinement on F
$R = 0.054$
$wR = 0.055$
$S = 1.852$
3811 reflections
721 parameters
H-atom parameters not refined

Unit weights applied
$(\Delta/\sigma)_{\max} = 0.08$
$\Delta\rho_{\max} = 0.46$ e Å^{-3}
$\Delta\rho_{\min} = -0.15$ e Å^{-3}
Atomic scattering factors from *MolEN* (Fair, 1990)

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

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

	x	y	z	$B_{\rm eq}$
Molecule *A*				
O1	0.7699 (4)	0.0938 (2)	0.2905 (1)	5.04 (9)
O8	0.9614 (6)	0.5840 (3)	0.1927 (2)	9.3 (2)
O9	1.0035 (5)	0.5085 (3)	0.1252 (1)	8.8 (1)
O12	0.8957 (4)	0.2361 (2)	0.1546 (1)	5.19 (9)
O24	0.8024 (4)	−0.3718 (3)	0.4088 (1)	7.1 (1)
O25	0.8664 (4)	−0.2163 (3)	0.4225 (1)	6.9 (1)
O28	0.7675 (3)	−0.0767 (2)	0.3055 (1)	4.17 (8)
C1	0.7729 (5)	0.0361 (3)	0.2565 (2)	4.1 (1)
C2	0.7151 (5)	0.0788 (3)	0.2191 (2)	3.9 (1)
C3	0.7668 (5)	0.1605 (3)	0.1990 (2)	4.1 (1)
C4	0.7571 (5)	0.2396 (4)	0.2271 (2)	5.3 (1)
C5	0.8117 (5)	0.3204 (3)	0.2086 (2)	4.9 (1)
C6	0.7949 (5)	0.3984 (4)	0.2305 (2)	5.8 (2)
C7	0.8420 (6)	0.4803 (4)	0.2225 (2)	6.4 (2)
C8	0.9192 (7)	0.5100 (4)	0.1897 (2)	6.8 (2)
C9	0.9444 (6)	0.4625 (4)	0.1543 (2)	6.7 (2)
C10	0.9241 (6)	0.3783 (4)	0.1470 (2)	5.9 (2)
C11	0.8741 (5)	0.3132 (4)	0.1730 (2)	5.2 (1)
C13	0.8870 (5)	0.1581 (3)	0.1796 (2)	4.3 (1)
C14	0.9028 (5)	0.0892 (4)	0.1468 (2)	4.8 (1)
C15	0.9337 (5)	0.0019 (4)	0.1630 (2)	4.8 (1)
C16	0.8686 (5)	−0.0660 (4)	0.1607 (2)	4.3 (1)
C17	0.8917 (5)	−0.1519 (3)	0.1813 (2)	4.3 (1)
C18	0.8544 (5)	−0.1458 (3)	0.2273 (2)	4.1 (1)
C19	0.7265 (5)	−0.1215 (3)	0.2353 (2)	3.9 (1)
C20	0.6626 (5)	−0.2002 (4)	0.2514 (2)	4.5 (1)
C21	0.6987 (4)	−0.2215 (3)	0.2959 (2)	3.9 (1)
C22	0.6747 (5)	−0.3065 (4)	0.3098 (2)	4.5 (1)
C23	0.7094 (5)	−0.3468 (4)	0.3461 (2)	5.0 (1)
C24	0.7780 (6)	−0.3202 (4)	0.3804 (2)	5.3 (1)
C25	0.8116 (5)	−0.2313 (4)	0.3862 (2)	5.3 (1)
C26	0.7970 (5)	−0.1638 (4)	0.3603 (2)	4.8 (1)
C27	0.7509 (5)	−0.1588 (3)	0.3194 (2)	4.1 (1)
C29	0.7100 (5)	−0.0482 (3)	0.2669 (2)	3.7 (1)
C30	0.7170 (6)	0.3937 (4)	0.2672 (3)	7.6 (2)
C31	0.9875 (5)	0.1605 (4)	0.2106 (2)	4.7 (1)
C32	0.8230 (6)	−0.2215 (4)	0.1591 (2)	5.9 (2)
C33	1.0187 (5)	−0.1749 (4)	0.1809 (2)	6.1 (2)
C34	0.6038 (6)	−0.3614 (4)	0.2815 (2)	6.4 (2)
C35	0.5832 (5)	−0.0324 (4)	0.2768 (2)	4.9 (1)
Molecule *B*				
O1	0.5660 (5)	0.3533 (2)	−0.0221 (1)	6.6 (1)
O8	0.5483 (6)	−0.1822 (3)	0.0688 (2)	9.4 (2)
O9	0.5514 (5)	−0.1120 (3)	0.1383 (1)	7.9 (1)
O12	0.5649 (4)	0.1716 (2)	0.1074 (1)	4.87 (9)
O24	0.6370 (5)	0.8382 (3)	−0.1110 (2)	9.3 (1)
O25	0.7562 (4)	0.7031 (3)	−0.1102 (2)	8.0 (1)
O28	0.5568 (4)	0.5229 (2)	−0.0264 (1)	5.14 (9)
C1	0.5202 (5)	0.3991 (3)	0.0130 (2)	4.7 (1)
C2	0.4334 (5)	0.3420 (4)	0.0357 (2)	4.6 (1)
C3	0.4727 (5)	0.2558 (3)	0.0554 (2)	4.8 (1)
C4	0.4935 (7)	0.1866 (4)	0.0236 (2)	6.6 (2)
C5	0.5203 (6)	0.1001 (3)	0.0434 (2)	5.6 (2)
C6	0.5202 (8)	0.0261 (4)	0.0178 (2)	8.6 (2)
C7	0.5349 (8)	−0.0595 (4)	0.0289 (2)	8.4 (2)
C8	0.5443 (7)	−0.1017 (4)	0.0670 (2)	6.8 (2)
C9	0.5485 (6)	−0.0579 (4)	0.1068 (2)	6.0 (2)
C10	0.5547 (6)	0.0281 (3)	0.1131 (2)	5.1 (1)
C11	0.5479 (6)	0.0991 (3)	0.0855 (2)	5.1 (1)

C13	0.5742 (5)	0.2559 (3)	0.0869 (2)	4.3 (1)
C14	0.5558 (6)	0.3167 (3)	0.1232 (2)	4.6 (1)
C15	0.5874 (5)	0.4078 (4)	0.1166 (2)	4.6 (1)
C16	0.5140 (5)	0.4722 (3)	0.1170 (2)	4.3 (1)
C17	0.5412 (6)	0.5648 (3)	0.1065 (2)	4.9 (1)
C18	0.5483 (5)	0.5732 (3)	0.0581 (2)	4.6 (1)
C19	0.4387 (5)	0.5472 (3)	0.0338 (2)	4.2 (1)
C20	0.3844 (5)	0.6266 (4)	0.0137 (2)	4.9 (1)
C21	0.4586 (5)	0.6600 (4)	−0.0221 (2)	4.6 (1)
C22	0.4317 (6)	0.7444 (4)	−0.0369 (2)	5.1 (1)
C23	0.4912 (6)	0.7928 (4)	−0.0652 (2)	6.0 (2)
C24	0.5948 (7)	0.7818 (4)	−0.0874 (2)	6.4 (2)
C25	0.6620 (6)	0.7028 (4)	−0.0857 (2)	5.8 (2)
C26	0.6370 (6)	0.6261 (4)	−0.0658 (2)	5.2 (1)
C27	0.5459 (5)	0.6075 (4)	−0.0383 (2)	4.7 (1)
C29	0.4647 (5)	0.4835 (3)	−0.0013 (2)	4.7 (1)
C30	0.498 (2)	0.0413 (5)	−0.0279 (2)	19.3 (6)
C31	0.6957 (6)	0.2606 (4)	0.0684 (2)	5.7 (2)
C32	0.4446 (7)	0.6225 (4)	0.1229 (2)	7.2 (2)
C33	0.6577 (7)	0.5924 (4)	0.1239 (2)	8.0 (2)
C34	0.3237 (6)	0.7848 (4)	−0.0198 (2)	7.0 (2)
C35	0.3578 (6)	0.4648 (4)	−0.0292 (2)	6.0 (2)

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

	Molecule *A*	Molecule *B*
O1—C1	1.419 (6)	1.442 (7)
O8—C8	1.257 (8)	1.258 (7)
O9—C9	1.366 (8)	1.320 (8)
O12—C11	1.364 (7)	1.348 (6)
O12—C13	1.463 (6)	1.478 (6)
O24—C24	1.252 (7)	1.262 (8)
O25—C25	1.352 (7)	1.343 (8)
O28—C27	1.369 (6)	1.380 (7)
O28—C29	1.481 (6)	1.470 (7)
C1—C2	1.532 (7)	1.526 (8)
C1—C29	1.538 (7)	1.535 (8)
C2—C3	1.549 (7)	1.555 (8)
C3—C4	1.535 (8)	1.507 (8)
C3—C13	1.521 (8)	1.549 (8)
C4—C5	1.530 (8)	1.525 (8)
C5—C6	1.422 (8)	1.421 (8)
C5—C11	1.361 (8)	1.395 (8)
C6—C7	1.412 (9)	1.393 (9)
C6—C30	1.49 (1)	1.52 (1)
C7—C8	1.46 (1)	1.40 (1)
C8—C9	1.392 (9)	1.458 (9)
C9—C10	1.355 (8)	1.359 (8)
C10—C11	1.437 (8)	1.425 (8)
C13—C14	1.518 (8)	1.524 (7)
C13—C31	1.532 (8)	1.524 (9)
C14—C15	1.501 (8)	1.483 (7)
C15—C16	1.300 (8)	1.312 (8)
C16—C17	1.520 (8)	1.517 (7)
C17—C18	1.550 (8)	1.570 (7)
C17—C32	1.523 (8)	1.53 (1)
C17—C33	1.506 (8)	1.52 (1)
C18—C19	1.543 (8)	1.541 (8)
C19—C20	1.524 (8)	1.531 (8)
C19—C29	1.545 (7)	1.538 (7)
C20—C21	1.532 (8)	1.532 (8)
C21—C22	1.426 (8)	1.435 (8)
C21—C27	1.376 (7)	1.398 (8)
C22—C23	1.388 (8)	1.368 (9)
C22—C34	1.495 (9)	1.50 (1)
C23—C24	1.422 (9)	1.40 (1)
C24—C25	1.453 (8)	1.457 (9)
C25—C26	1.356 (8)	1.388 (9)
C26—C27	1.426 (8)	1.404 (8)
C29—C35	1.515 (8)	1.552 (9)

D—H···*A*	*D*···*A*	H···*A*	*D*—H···*A*
O9*A*—H9*A*···O8*A*	2.526	1.778	114.8
O25*A*—H25*A*···O24*A*	2.574	2.042	112.7
O9*A*—H9*A*···O24*B*^{i}	2.879	2.202	112.5
O24*A*—H24*A*···O1*B*^{ii}	2.710	–	–
O25*A*—H25*A*···O1*B*^{ii}	2.893	2.017	149.6
O1*A*—HO1*A*···O8*A*^{iii}	3.143	2.362	138.4

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

The Lorentz and polarization effects were corrected. The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1985) and was refined by full-matrix least-squares techniques using *MolEN* (Fair, 1990). The positions of all the H atoms, except the hydroxyl H atoms, were calculated from an idealized geometry with standard bond lengths and angles. The three hydroxyl H atoms of molecule *A* were located from difference Fourier maps. All the non-hydroxyl H atoms were included in structure-factor calculations with isotropic displacement factors and fixed parameters. For determination of the absolute configuration, Friedel pairs were collected up to $\sin\theta/\lambda = 0.42$ Å^{-1}. Refinements on both enantiomers converged with the same *R* values; the absolute configuration was not determined.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, bond distances, angles and torsion angles have been deposited with the IUCr (Reference: HH1073). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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