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Eplerenone 1,4-dioxane solvate

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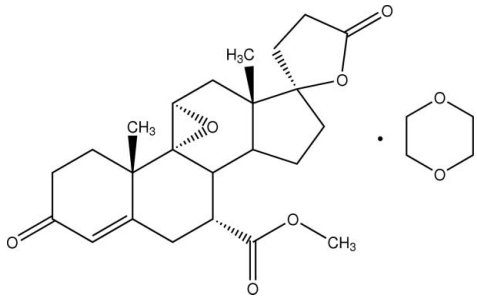
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.006 Å; disorder in solvent or counterion; R factor = 0.065; wR factor = 0.209; data-to-parameter ratio = 10.3.

Eplerenone is an aldosterone receptor antagonist. Single crystals of the title compound [systematic name: 9α,11-epoxy-7α-(methoxycarbonyl)-3-oxo-17α-pregn-4-ene-21,17-carbolactone dioxane solvate], $C_{24}H_{30}O_6\cdot C_4H_8O_2$, were obtained from a dioxane solution. Within the eplerenone molecule, both five-membered rings display an envelope conformation. The three six-membered rings exhibit different conformations: chair, half-chair and envelope. In the crystal structure, adjacent eplerenone molecules are linked together *via* weak intermolecular C—H···O hydrogen bonding, forming a cavity of about 310 Å^3, and the dioxane solvent molecules occupy the cavity. The solvent molecule is disordered equally over two positions.

Related literature

For general background, see: Grob & Kalvoda (1985). For related structures, see: Grob *et al.* (1997); Yang *et al.* (2007). For cavity analysis, see: Spek (2003).



Experimental

Crystal data

$C_{24}H_{30}O_6\cdot C_4H_8O_2$
M_r = 502.58
Orthorhombic, $P2_12_12_1$
a = 8.2308 (8) Å
b = 13.5580 (12) Å
c = 23.2279 (18) Å
V = 2592.1 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 291 (2) K
0.48 × 0.45 × 0.27 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
Absorption correction: none
24758 measured reflections
3340 independent reflections
2244 reflections with $I > 2\sigma(I)$
R_{int} = 0.060

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.209
S = 1.07
3340 reflections
324 parameters
12 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7A···O72B^{i}	0.97	2.59	3.370 (15)	137
C12—H12B···O1^{ii}	0.97	2.55	3.500 (6)	167
C14—H14···O5	0.98	2.55	3.138 (5)	119
C18—H18A···O71B^{iii}	0.97	2.55	3.335 (18)	138
C19—H19B···O1^{ii}	0.97	2.60	3.252 (6)	125
C21—H21C···O5^{iv}	0.96	2.50	3.326 (7)	144

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

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2379).

References

Altomare, A., Cascarano, G., Giacovazzo, C. & Guagliardi, A. (1993). *J. Appl. Cryst.* **26**, 343–350.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Grob, J., Boillaz, M., Schmidlin, J., Wehrli, H., Wieland, P., Fuhrer, H., Rihs, G., Joss, U., de Gasparo, M., Haenni, H., Ramjoue, H. P., Whitebread, S. E. & Kalvoda, J. (1997). *Helv. Chim. Acta*, **80**, 566–585.
Grob, J. & Kalvoda, J. (1985). US Patent 4 559 332.
Rigaku (1998). *PROCESS-AUTO*. Rigaku Corporation, Tokyo, Japan.
Rigaku/MSC (2002). *CrystalStructure*. Version 3.00. Rigaku/MSC, The Woodlands, Texas, USA.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Yang, Q., Ye, W.-D., Yuan, J.-Y. & Nie, J.-J. (2007). *Acta Cryst.* E**63**, o2068–o2070.

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supplementary materials

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Eplerenone 1,4-dioxane solvate

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Comment

The eplerenone is known as an aldosterone receptor antagonist and can be administered in a therapeutically effective amount where use of an aldosterone receptor antagonist (Grob & Kalvoda, 1985). The crystal structure of the eplerenone with dioxane solvent is reported here.

The crystal of the title compound consists of eplerenone molecules and lattice dioxane moleuces as shown in Fig. 1. The molecule of eplerenone contains three fused six-membered rings, two five-membered rings and one three-membered ring. Both five-membered rings display the envelope configuration, similar to that found in the structure of eplerenone tetrahydrofuran solvate (Yang *et al.*, 2007). The O5-carbonyl group of the C23-ester group forms an intra-molecular hydrogen bond with the adjacent C14-methine group (Fig. 1 and Table 1). This structural features is identical with that found in the crystal structure of eplerenone dichloromethane solvate (Grob *et al.*, 1997).

In the crystal the adjacent eplerenone molecules are linked together *via* weak intermolecular C—H···O hydrogen bonding (Table 1), forming the 310 $Å^3$ cavity (Spek, 2003), where the disordered dioxane solvent molecules occupied (Fig. 2).

Experimental

The eplerenone dichloromethane was prepared according to the procedure reported by Grob *et al.* (1997). The dichloromethane solvent was removed by heating at 363 K to get a powder crystalline sample. Single crystals of the title compound were crystallized from a dioxane solution of eplerenone at room temperature.

Refinement

H atoms were placed in calculated positions with C—H = 0.93 to 0.98 Å and refined in riding mode with $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl groups and $1.2U_{eq}(C)$ for others. The solvent dioxane molecule is disordered in the crystal structure. Two sites model was used in the refinement with each component 0.5 occupancy factor. The C—C and C—O distances of disordered dioxane molecule were restrained to 1.54±0.01 and 1.40±0.01 Å, respectively. The thermal parameters for oxygen atoms of dioxane were constrained to be the same, so do for the carbon atoms of dioxane. In the absence of significant anomalous scattering effects, Friedel pairs were merged; the absolute configuration was not determined.

Figures

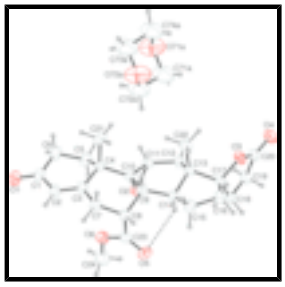

Fig. 1. The molecular structure of the title compound with 30% probability displacement (arbitrary spheres for H atoms). One of disordered dioxane components has been omitted for clarity. The dashed line indicates intramolecular hydrogen bongding.

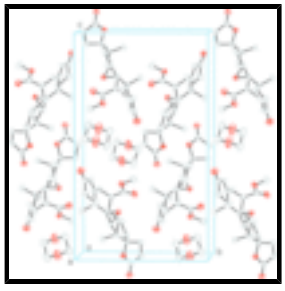

Fig. 2. The unit cell packing of the title compound. H atoms and one of disordered components have been omitted for clarity.

9α,11-Epoxy-7α-(methoxycarbonyl)-3-oxo-17α-pregn-4-ene-21,17-carbolactone dioxane solvate

Crystal data

$C_{24}H_{30}O_6 \cdot C_4H_8O_2$	F_{000} = 1080
M_r = 502.58	D_x = 1.288 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 8986 reflections
a = 8.2308 (8) Å	θ = 3.0–25.5º
b = 13.5580 (12) Å	μ = 0.09 mm^{-1}
c = 23.2279 (18) Å	T = 291 (2) K
V = 2592.1 (4) Å^3	Prism, colorless
Z = 4	0.48 × 0.45 × 0.27 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer	3340 independent reflections
Radiation source: fine-focus sealed tube	2244 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.060
Detector resolution: 10.0 pixels mm^{-1}	θ_{max} = 27.4º
T = 291(2) K	θ_{min} = 3.0º
ω scans	h = −10→9
Absorption correction: none	k = −17→17
24758 measured reflections	l = −29→30

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.065$

$wR(F^2) = 0.209$

$S = 1.07$

3340 reflections

324 parameters

12 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1356P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.36$ e Å^{-3}

$\Delta\rho_{min} = -0.42$ e Å^{-3}

Extinction correction: SHELXL97, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.016 (4)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
O1	−0.0712 (5)	0.5542 (3)	0.10266 (16)	0.0857 (12)	
O2	0.1407 (3)	0.82048 (19)	0.29115 (11)	0.0458 (6)	
O3	0.4915 (4)	0.8356 (2)	0.49706 (11)	0.0573 (8)	
O4	0.3797 (5)	0.8881 (3)	0.57887 (13)	0.0739 (10)	
O5	0.4814 (6)	0.9205 (3)	0.22206 (16)	0.0855 (12)	
O6	0.2938 (4)	0.8363 (2)	0.17302 (13)	0.0638 (9)	
C1	−0.0097 (6)	0.5814 (3)	0.1479 (2)	0.0596 (11)	
C2	0.1593 (6)	0.6089 (3)	0.15113 (19)	0.0548 (10)	
H2	0.2196	0.6078	0.1173	0.066*	
C3	0.2352 (5)	0.6361 (3)	0.19966 (18)	0.0494 (9)	
C4	0.1465 (4)	0.6421 (3)	0.25689 (16)	0.0430 (8)	
C5	−0.0358 (5)	0.6585 (4)	0.2456 (2)	0.0594 (11)	
H5A	−0.0947	0.6521	0.2815	0.071*	
H5B	−0.0521	0.7250	0.2314	0.071*	

C6	−0.1048 (5)	0.5854 (4)	0.2021 (2)	0.0669 (13)	
H6A	−0.1070	0.5202	0.2193	0.080*	
H6B	−0.2159	0.6038	0.1931	0.080*	
C7	0.4150 (5)	0.6551 (4)	0.19859 (17)	0.0526 (10)	
H7A	0.4490	0.6648	0.1590	0.063*	
H7B	0.4709	0.5973	0.2132	0.063*	
C8	0.4663 (4)	0.7454 (3)	0.23433 (17)	0.0493 (10)	
H8	0.5852	0.7445	0.2364	0.059*	
C9	0.4029 (4)	0.7308 (3)	0.29600 (16)	0.0433 (8)	
H9	0.4423	0.6663	0.3091	0.052*	
C10	0.2189 (4)	0.7252 (3)	0.29489 (16)	0.0396 (8)	
C11	0.1279 (5)	0.7654 (3)	0.34464 (16)	0.0446 (9)	
H11	0.0218	0.7348	0.3514	0.054*	
C12	0.2060 (5)	0.8094 (3)	0.39728 (17)	0.0472 (9)	
H12A	0.1550	0.7820	0.4314	0.057*	
H12B	0.1876	0.8800	0.3974	0.057*	
C13	0.3889 (5)	0.7893 (3)	0.39967 (16)	0.0436 (9)	
C14	0.4620 (4)	0.8068 (3)	0.33951 (16)	0.0439 (9)	
H14	0.4279	0.8723	0.3263	0.053*	
C15	0.6476 (5)	0.8095 (4)	0.3502 (2)	0.0625 (12)	
H15A	0.6946	0.7443	0.3455	0.075*	
H15B	0.7003	0.8545	0.3237	0.075*	
C16	0.6669 (5)	0.8450 (4)	0.4121 (2)	0.0596 (11)	
H16A	0.7288	0.9058	0.4133	0.072*	
H16B	0.7229	0.7958	0.4350	0.072*	
C17	0.4952 (5)	0.8617 (3)	0.43499 (15)	0.0481 (9)	
C18	0.4392 (6)	0.9696 (3)	0.43506 (18)	0.0530 (10)	
H18A	0.5318	1.0139	0.4349	0.064*	
H18B	0.3724	0.9834	0.4016	0.064*	
C19	0.3419 (6)	0.9810 (3)	0.48991 (17)	0.0593 (11)	
H19A	0.3603	1.0453	0.5071	0.071*	
H19B	0.2267	0.9731	0.4825	0.071*	
C20	0.4042 (6)	0.9003 (3)	0.52813 (18)	0.0551 (10)	
C21	0.1706 (6)	0.5435 (3)	0.2897 (2)	0.0604 (11)	
H21A	0.1290	0.4901	0.2670	0.091*	
H21B	0.1136	0.5462	0.3258	0.091*	
H21C	0.2843	0.5333	0.2968	0.091*	
C22	0.4163 (6)	0.6834 (3)	0.42091 (18)	0.0571 (11)	
H22A	0.3896	0.6793	0.4611	0.086*	
H22B	0.5282	0.6657	0.4155	0.086*	
H22C	0.3484	0.6391	0.3995	0.086*	
C23	0.4185 (6)	0.8438 (4)	0.20998 (18)	0.0558 (11)	
C24	0.2338 (9)	0.9278 (4)	0.1488 (3)	0.0927 (19)	
H24A	0.2996	0.9460	0.1164	0.139*	
H24B	0.2388	0.9787	0.1775	0.139*	
H24C	0.1233	0.9191	0.1366	0.139*	
O71A	0.193 (2)	0.3566 (12)	0.4854 (9)	0.166 (4)	0.50
O72A	0.431 (2)	0.3198 (9)	0.4216 (7)	0.166 (4)	0.50
C71A	0.294 (3)	0.4369 (11)	0.4700 (7)	0.154 (3)	0.50

H71A	0.3886	0.4419	0.4946	0.185*	0.50
H71B	0.2350	0.4989	0.4703	0.185*	0.50
C72A	0.337 (3)	0.4045 (12)	0.4096 (6)	0.154 (3)	0.50
H72A	0.2413	0.3884	0.3872	0.185*	0.50
H72B	0.4000	0.4543	0.3896	0.185*	0.50
C73A	0.315 (3)	0.2469 (13)	0.4373 (7)	0.154 (3)	0.50
H73A	0.3591	0.1811	0.4325	0.185*	0.50
H73B	0.2172	0.2530	0.4142	0.185*	0.50
C74A	0.280 (3)	0.2688 (12)	0.5006 (7)	0.154 (3)	0.50
H74A	0.2126	0.2194	0.5190	0.185*	0.50
H74B	0.3772	0.2813	0.5231	0.185*	0.50
O71B	0.187 (2)	0.3779 (12)	0.4944 (8)	0.166 (4)	0.50
O72B	0.368 (2)	0.2869 (10)	0.3974 (6)	0.166 (4)	0.50
C71B	0.190 (4)	0.4141 (11)	0.4372 (7)	0.154 (3)	0.50
H71C	0.2779	0.4609	0.4330	0.185*	0.50
H71D	0.0886	0.4486	0.4295	0.185*	0.50
C72B	0.211 (2)	0.3308 (12)	0.3932 (7)	0.154 (3)	0.50
H72C	0.1285	0.2807	0.3998	0.185*	0.50
H72D	0.1953	0.3568	0.3547	0.185*	0.50
C73B	0.365 (4)	0.2373 (12)	0.4509 (6)	0.154 (3)	0.50
H73C	0.4686	0.2056	0.4589	0.185*	0.50
H73D	0.2796	0.1883	0.4521	0.185*	0.50
C74B	0.333 (3)	0.3201 (11)	0.4930 (7)	0.154 (3)	0.50
H74C	0.3443	0.2917	0.5311	0.185*	0.50
H74D	0.4216	0.3665	0.4885	0.185*	0.50

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.087 (3)	0.097 (3)	0.073 (2)	−0.032 (2)	−0.019 (2)	−0.012 (2)
O2	0.0459 (13)	0.0406 (14)	0.0510 (14)	0.0051 (11)	−0.0005 (12)	0.0003 (12)
O3	0.0726 (18)	0.0561 (17)	0.0431 (14)	0.0134 (15)	−0.0107 (14)	0.0028 (13)
O4	0.108 (3)	0.069 (2)	0.0451 (17)	0.009 (2)	−0.0006 (18)	−0.0004 (15)
O5	0.106 (3)	0.070 (2)	0.081 (2)	−0.041 (2)	−0.002 (2)	0.0095 (18)
O6	0.072 (2)	0.0581 (18)	0.0613 (18)	−0.0009 (16)	−0.0077 (16)	0.0094 (15)
C1	0.063 (3)	0.046 (2)	0.069 (3)	−0.007 (2)	−0.005 (2)	−0.011 (2)
C2	0.057 (2)	0.058 (2)	0.050 (2)	−0.005 (2)	−0.004 (2)	−0.0102 (19)
C3	0.050 (2)	0.048 (2)	0.050 (2)	0.0030 (18)	0.0030 (19)	−0.0074 (19)
C4	0.0394 (18)	0.0406 (19)	0.049 (2)	0.0015 (15)	0.0054 (17)	−0.0026 (16)
C5	0.041 (2)	0.067 (3)	0.070 (3)	−0.001 (2)	−0.002 (2)	−0.016 (2)
C6	0.048 (2)	0.071 (3)	0.082 (3)	−0.008 (2)	0.003 (2)	−0.020 (3)
C7	0.0431 (19)	0.065 (3)	0.049 (2)	0.0012 (19)	0.0061 (18)	−0.009 (2)
C8	0.0374 (19)	0.062 (3)	0.049 (2)	−0.0047 (19)	0.0070 (17)	−0.0024 (18)
C9	0.0391 (17)	0.046 (2)	0.0448 (19)	0.0065 (16)	0.0011 (17)	−0.0005 (17)
C10	0.0363 (16)	0.0373 (18)	0.0450 (19)	0.0005 (14)	0.0055 (16)	0.0027 (16)
C11	0.0427 (19)	0.045 (2)	0.046 (2)	0.0004 (16)	0.0059 (17)	0.0015 (17)
C12	0.047 (2)	0.048 (2)	0.046 (2)	0.0015 (17)	0.0009 (18)	−0.0014 (18)
C13	0.047 (2)	0.045 (2)	0.0391 (18)	0.0044 (16)	−0.0018 (16)	0.0009 (16)

C14	0.0387 (18)	0.048 (2)	0.045 (2)	−0.0007 (16)	0.0033 (16)	−0.0016 (17)
C15	0.041 (2)	0.085 (3)	0.061 (3)	−0.003 (2)	−0.001 (2)	−0.008 (2)
C16	0.047 (2)	0.068 (3)	0.064 (3)	0.002 (2)	−0.008 (2)	−0.008 (2)
C17	0.058 (2)	0.048 (2)	0.0390 (19)	0.0053 (18)	−0.0067 (17)	0.0005 (16)
C18	0.060 (2)	0.045 (2)	0.054 (2)	−0.0058 (19)	−0.007 (2)	0.0038 (18)
C19	0.075 (3)	0.049 (2)	0.054 (2)	0.008 (2)	−0.009 (2)	−0.0017 (19)
C20	0.071 (3)	0.047 (2)	0.047 (2)	0.000 (2)	−0.002 (2)	−0.0025 (18)
C21	0.063 (3)	0.047 (2)	0.071 (3)	−0.006 (2)	0.001 (2)	0.004 (2)
C22	0.072 (3)	0.046 (2)	0.053 (2)	0.010 (2)	−0.001 (2)	0.0045 (19)
C23	0.056 (2)	0.065 (3)	0.046 (2)	−0.012 (2)	0.007 (2)	0.006 (2)
C24	0.124 (5)	0.076 (4)	0.078 (3)	0.023 (4)	−0.018 (4)	0.015 (3)
O71A	0.207 (6)	0.082 (6)	0.208 (8)	0.044 (4)	0.120 (5)	0.001 (5)
O72A	0.207 (6)	0.082 (6)	0.208 (8)	0.044 (4)	0.120 (5)	0.001 (5)
C71A	0.252 (10)	0.078 (4)	0.133 (5)	0.035 (6)	0.038 (6)	0.020 (4)
C72A	0.252 (10)	0.078 (4)	0.133 (5)	0.035 (6)	0.038 (6)	0.020 (4)
C73A	0.252 (10)	0.078 (4)	0.133 (5)	0.035 (6)	0.038 (6)	0.020 (4)
C74A	0.252 (10)	0.078 (4)	0.133 (5)	0.035 (6)	0.038 (6)	0.020 (4)
O71B	0.207 (6)	0.082 (6)	0.208 (8)	0.044 (4)	0.120 (5)	0.001 (5)
O72B	0.207 (6)	0.082 (6)	0.208 (8)	0.044 (4)	0.120 (5)	0.001 (5)
C71B	0.252 (10)	0.078 (4)	0.133 (5)	0.035 (6)	0.038 (6)	0.020 (4)
C72B	0.252 (10)	0.078 (4)	0.133 (5)	0.035 (6)	0.038 (6)	0.020 (4)
C73B	0.252 (10)	0.078 (4)	0.133 (5)	0.035 (6)	0.038 (6)	0.020 (4)
C74B	0.252 (10)	0.078 (4)	0.133 (5)	0.035 (6)	0.038 (6)	0.020 (4)

Geometric parameters (Å, °)

O1—C1	1.224 (6)	C16—H16A	0.9700
O2—C10	1.445 (4)	C16—H16B	0.9700
O2—C11	1.453 (5)	C17—C18	1.534 (6)
O3—C20	1.344 (5)	C18—C19	1.513 (6)
O3—C17	1.485 (4)	C18—H18A	0.9700
O4—C20	1.207 (5)	C18—H18B	0.9700
O5—C23	1.195 (6)	C19—C20	1.500 (6)
O6—C23	1.342 (6)	C19—H19A	0.9700
O6—C24	1.448 (6)	C19—H19B	0.9700
C1—C2	1.442 (7)	C21—H21A	0.9600
C1—C6	1.483 (7)	C21—H21B	0.9600
C2—C3	1.341 (6)	C21—H21C	0.9600
C2—H2	0.9300	C22—H22A	0.9600
C3—C7	1.502 (6)	C22—H22B	0.9600
C3—C4	1.519 (5)	C22—H22C	0.9600
C4—C5	1.539 (5)	C24—H24A	0.9600
C4—C10	1.550 (5)	C24—H24B	0.9600
C4—C21	1.552 (6)	C24—H24C	0.9600
C5—C6	1.525 (6)	O71A—C71A	1.412 (10)
C5—H5A	0.9700	O71A—C74A	1.431 (10)
C5—H5B	0.9700	O72A—C72A	1.412 (10)
C6—H6A	0.9700	O72A—C73A	1.422 (10)
C6—H6B	0.9700	C71A—C72A	1.513 (10)

C7—C8	1.539 (6)	C71A—H71A	0.9700
C7—H7A	0.9700	C71A—H71B	0.9700
C7—H7B	0.9700	C72A—H72A	0.9700
C8—C23	1.501 (7)	C72A—H72B	0.9700
C8—C9	1.538 (5)	C73A—C74A	1.527 (10)
C8—H8	0.9800	C73A—H73A	0.9700
C9—C10	1.517 (5)	C73A—H73B	0.9700
C9—C14	1.524 (5)	C74A—H74A	0.9700
C9—H9	0.9800	C74A—H74B	0.9700
C10—C11	1.481 (5)	O71B—C71B	1.416 (10)
C11—C12	1.504 (5)	O71B—C74B	1.430 (10)
C11—H11	0.9800	O72B—C73B	1.414 (10)
C12—C13	1.531 (5)	O72B—C72B	1.431 (10)
C12—H12A	0.9700	C71B—C72B	1.532 (10)
C12—H12B	0.9700	C71B—H71C	0.9700
C13—C22	1.535 (6)	C71B—H71D	0.9700
C13—C14	1.540 (5)	C72B—H72C	0.9700
C13—C17	1.550 (6)	C72B—H72D	0.9700
C14—C15	1.548 (5)	C73B—C74B	1.512 (10)
C14—H14	0.9800	C73B—H73C	0.9700
C15—C16	1.524 (6)	C73B—H73D	0.9700
C15—H15A	0.9700	C74B—H74C	0.9700
C15—H15B	0.9700	C74B—H74D	0.9700
C16—C17	1.527 (6)		
C10—O2—C11	61.4 (2)	C16—C17—C18	114.8 (4)
C20—O3—C17	112.1 (3)	O3—C17—C13	110.6 (3)
C23—O6—C24	116.4 (4)	C16—C17—C13	104.1 (3)
O1—C1—C2	121.4 (5)	C18—C17—C13	115.8 (3)
O1—C1—C6	121.4 (4)	C19—C18—C17	105.0 (3)
C2—C1—C6	117.1 (4)	C19—C18—H18A	110.8
C3—C2—C1	124.3 (4)	C17—C18—H18A	110.8
C3—C2—H2	117.8	C19—C18—H18B	110.8
C1—C2—H2	117.8	C17—C18—H18B	110.8
C2—C3—C7	119.5 (4)	H18A—C18—H18B	108.8
C2—C3—C4	121.8 (4)	C20—C19—C18	104.0 (4)
C7—C3—C4	118.6 (3)	C20—C19—H19A	111.0
C3—C4—C5	109.1 (3)	C18—C19—H19A	111.0
C3—C4—C10	110.7 (3)	C20—C19—H19B	111.0
C5—C4—C10	111.5 (3)	C18—C19—H19B	111.0
C3—C4—C21	108.8 (3)	H19A—C19—H19B	109.0
C5—C4—C21	109.4 (4)	O4—C20—O3	121.6 (4)
C10—C4—C21	107.3 (3)	O4—C20—C19	128.4 (4)
C6—C5—C4	112.5 (4)	O3—C20—C19	110.0 (3)
C6—C5—H5A	109.1	C4—C21—H21A	109.5
C4—C5—H5A	109.1	C4—C21—H21B	109.5
C6—C5—H5B	109.1	H21A—C21—H21B	109.5
C4—C5—H5B	109.1	C4—C21—H21C	109.5
H5A—C5—H5B	107.8	H21A—C21—H21C	109.5
C1—C6—C5	112.9 (4)	H21B—C21—H21C	109.5

C1—C6—H6A	109.0	C13—C22—H22A	109.5
C5—C6—H6A	109.0	C13—C22—H22B	109.5
C1—C6—H6B	109.0	H22A—C22—H22B	109.5
C5—C6—H6B	109.0	C13—C22—H22C	109.5
H6A—C6—H6B	107.8	H22A—C22—H22C	109.5
C3—C7—C8	113.5 (3)	H22B—C22—H22C	109.5
C3—C7—H7A	108.9	O5—C23—O6	123.2 (4)
C8—C7—H7A	108.9	O5—C23—C8	124.9 (4)
C3—C7—H7B	108.9	O6—C23—C8	112.0 (4)
C8—C7—H7B	108.9	O6—C24—H24A	109.5
H7A—C7—H7B	107.7	O6—C24—H24B	109.5
C23—C8—C9	112.2 (3)	H24A—C24—H24B	109.5
C23—C8—C7	115.6 (3)	O6—C24—H24C	109.5
C9—C8—C7	107.8 (3)	H24A—C24—H24C	109.5
C23—C8—H8	106.9	H24B—C24—H24C	109.5
C9—C8—H8	106.9	C71A—O71A—C74A	114.3 (16)
C7—C8—H8	106.9	C72A—O72A—C73A	104.3 (15)
C10—C9—C14	111.3 (3)	O71A—C71A—C72A	98.6 (15)
C10—C9—C8	109.2 (3)	O71A—C71A—H71A	112.1
C14—C9—C8	115.0 (3)	C72A—C71A—H71A	112.1
C10—C9—H9	107.0	O71A—C71A—H71B	112.1
C14—C9—H9	107.0	C72A—C71A—H71B	112.1
C8—C9—H9	107.0	H71A—C71A—H71B	109.7
O2—C10—C11	59.5 (2)	O72A—C72A—C71A	100.5 (12)
O2—C10—C9	113.7 (3)	O72A—C72A—H72A	111.7
C11—C10—C9	118.2 (3)	C71A—C72A—H72A	111.7
O2—C10—C4	116.4 (3)	O72A—C72A—H72B	111.7
C11—C10—C4	121.2 (3)	C71A—C72A—H72B	111.7
C9—C10—C4	115.4 (3)	H72A—C72A—H72B	109.4
O2—C11—C10	59.0 (2)	O72A—C73A—C74A	103.7 (15)
O2—C11—C12	117.4 (3)	O72A—C73A—H73A	111.0
C10—C11—C12	124.4 (3)	C74A—C73A—H73A	111.0
O2—C11—H11	114.8	O72A—C73A—H73B	111.0
C10—C11—H11	114.8	C74A—C73A—H73B	111.0
C12—C11—H11	114.8	H73A—C73A—H73B	109.0
C11—C12—C13	112.3 (3)	O71A—C74A—C73A	91.1 (14)
C11—C12—H12A	109.1	O71A—C74A—H74A	113.5
C13—C12—H12A	109.1	C73A—C74A—H74A	113.5
C11—C12—H12B	109.1	O71A—C74A—H74B	113.5
C13—C12—H12B	109.1	C73A—C74A—H74B	113.5
H12A—C12—H12B	107.9	H74A—C74A—H74B	110.8
C12—C13—C22	108.8 (4)	C71B—O71B—C74B	99.1 (16)
C12—C13—C14	108.9 (3)	C73B—O72B—C72B	103.9 (15)
C22—C13—C14	112.2 (3)	O71B—C71B—C72B	111.8 (15)
C12—C13—C17	117.5 (3)	O71B—C71B—H71C	109.3
C22—C13—C17	109.8 (3)	C72B—C71B—H71C	109.3
C14—C13—C17	99.3 (3)	O71B—C71B—H71D	109.3
C9—C14—C13	111.9 (3)	C72B—C71B—H71D	109.3
C9—C14—C15	115.9 (3)	H71C—C71B—H71D	107.9

C13—C14—C15	104.1 (3)	O72B—C72B—C71B	111.4 (16)
C9—C14—H14	108.2	O72B—C72B—H72C	109.3
C13—C14—H14	108.2	C71B—C72B—H72C	109.3
C15—C14—H14	108.2	O72B—C72B—H72D	109.3
C16—C15—C14	105.2 (4)	C71B—C72B—H72D	109.3
C16—C15—H15A	110.7	H72C—C72B—H72D	108.0
C14—C15—H15A	110.7	O72B—C73B—C74B	102.6 (13)
C16—C15—H15B	110.7	O72B—C73B—H73C	111.2
C14—C15—H15B	110.7	C74B—C73B—H73C	111.2
H15A—C15—H15B	108.8	O72B—C73B—H73D	111.2
C15—C16—C17	106.2 (3)	C74B—C73B—H73D	111.2
C15—C16—H16A	110.5	H73C—C73B—H73D	109.2
C17—C16—H16A	110.5	O71B—C74B—C73B	124.7 (19)
C15—C16—H16B	110.5	O71B—C74B—H74C	106.1
C17—C16—H16B	110.5	C73B—C74B—H74C	106.1
H16A—C16—H16B	108.7	O71B—C74B—H74D	106.1
O3—C17—C16	108.8 (3)	C73B—C74B—H74D	106.1
O3—C17—C18	102.7 (3)	H74C—C74B—H74D	106.3

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7A···O72B^{i}	0.97	2.59	3.370 (15)	137
C12—H12B···O1^{ii}	0.97	2.55	3.500 (6)	167
C14—H14···O5	0.98	2.55	3.138 (5)	119
C18—H18A···O71B^{iii}	0.97	2.55	3.335 (18)	138
C19—H19B···O1^{ii}	0.97	2.60	3.252 (6)	125
C21—H21C···O5^{iv}	0.96	2.50	3.326 (7)	144

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+1/2; (ii) −*x*, *y*+1/2, −*z*+1/2; (iii) *x*+1/2, −*y*+3/2, −*z*+1; (iv) −*x*+1, *y*−1/2, −*z*+1/2.

Fig. 1

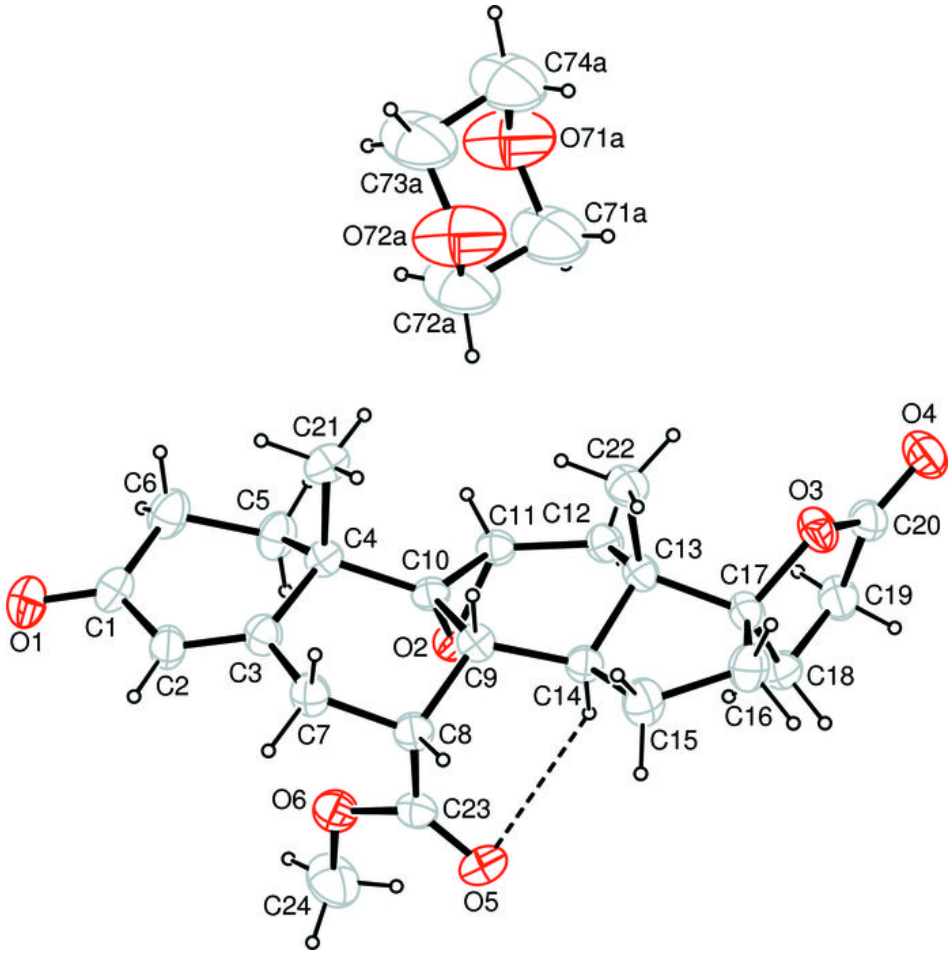
C74a
O71a
C73a
C71a
O72a
C72a
C21
C22
O4
C6
C5
C11
C12
O3
C20
C4
C10
C13
C17
C19
O1
C1
O2
C9
C18
C2
C3
C14
C16
C7
C8
C15
O6
C23
C24
O5

Fig. 2