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Betamethasone-21-pentanoate methanol solvate

Viktor Suitchmezian, Inke Jess and Christian Näther*

Institut für Anorganische Chemie, Christian-Albrechts-Universität Kiel, Olshausenstr. 40, D-24098 Kiel, Germany
Correspondence e-mail: cnaether@ac.uni-kiel.de

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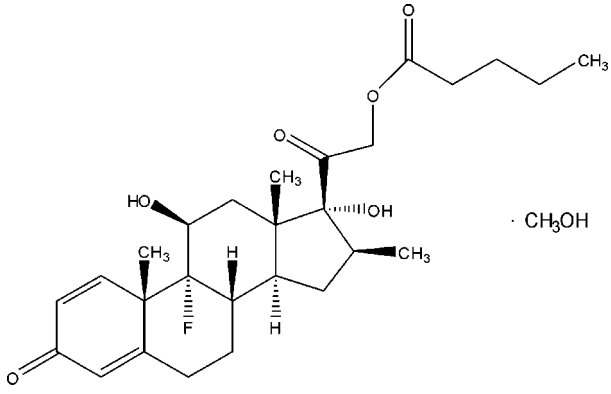
Key indicators: single-crystal X-ray study; T = 166 K; mean σ(C–C) = 0.004 Å; R factor = 0.042; wR factor = 0.114; data-to-parameter ratio = 10.9.

In the crystal structure of the title compound {systematic name: 2-[(8*S*,9*R*,10*S*,11*S*,13*S*,14*S*,16*S*,17*R*)-9-fluoro-11,17-dihydroxy-10,13,16-trimethyl-3-oxo-6,7,8,9,10,11,12,13,14,15,16,17-dodecahydro-3*H*-cyclopenta[*a*]phenanthren-17-yl]-2-oxoethyl pentanoate methanol solvate}, $C_{27}H_{37}FO_6\cdot CH_3OH$ molecules are connected *via* O—H···O hydrogen bonding between the hydroxyl H atom and the carbonyl O atom. The molecules are additionally connected *via* the methanol molecules, which act as hydrogen-bond donors and acceptors.

Related literature

For related literature, see: Auphan (1995); Beato (1995); Besedovsky (1986); Näther & Jess (2006); Suitchmezian *et al.* (2006*a*,*b*,*c*); Winiski *et al.* (2007).



Experimental

Crystal data

$C_{27}H_{37}FO_6\cdot CH_4O$
M_r = 508.61
Orthorhombic, $P2_12_12_1$
a = 9.9980 (5) Å
b = 15.7885 (9) Å
c = 16.9696 (12) Å
V = 2678.7 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 166 (2) K
0.15 × 0.10 × 0.04 mm

Data collection

STOE IPDS-1 diffractometer
Absorption correction: none
16982 measured reflections
3595 independent reflections
3009 reflections with $I > 2\sigma(I)$
R_{int} = 0.049

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.114
S = 1.02
3595 reflections
330 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H1*O1*···O7^{i}	0.84	2.01	2.845 (3)	176
O5—H1*O5*···O1^{ii}	0.84	1.95	2.773 (2)	165
O7—H1*O7*···O6^{iii}	0.84	1.94	2.771 (3)	171

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

Data collection: *IPDS* (Stoe & Cie, 1998); cell refinement: *IPDS*; data reduction: *IPDS*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *CIFTAB* in *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2615).

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supplementary materials

Acta Cryst. (2007). E**63**, o4839 [**doi:10.1107/S1600536807059545**]

Betamhethasone-21-pentanoate methanol solvate

V. Suitchmezian, I. Jess and C. Näther

Comment

Glucocorticoids belong to the most effective drugs against inflammatory and auto immune deseases (Auphan, 1995; Besedovsky, 1986; Beato, 1995; Winiski *et al.*, 2007). Surprisingly, their polymorphism is only minor investigated. Therefore, we started systematic investigations on the polymorphism and pseudopolymorphism of these drugs (Suitchmezian *et al.*, 2006*a*; Suitchmezian *et al.*, 2006*b*; Suitchmezian *et al.*, 2006*c*; Näther *et al.*, 2006).

Within this project we also investigated betamethasone valerate, also known as 9-fluoro-11β,21-dihydroxy-16β-methyl-3,20 -dioxypregna-1,4-dien-17-ylpentanoat, (II) (see Fig 3). For this compound we found an methanol solvate, which is still unknown. During our attempts to crystallize this solvate at higher temperatures, we obtained single crystals of the title compound, (I), which formed by a movement of the pentanoat group to position 21. To identify this product in all further investigations by X-ray powder diffraction, the single-crystal structure of this solvate was determined.

In the crystal structure of the title compound, (I), (Fig. 1) the molecules are connected *via* O—H ··· O hydrogen bonding between the hydroxyl hydrogen atom at O5 and the carbonyl oxygen atom O1 (Tab.1). The molecules are additionally connected by O—H···O hydrogen bonding between the hydroxyl hydrogen atom at O2 and the hydroxyl oxygen atom O7 of the solvent molecules and between the hydroxyl hydrogen atom attached to O7 and the carbonyl oxygen atom O6 (Fig. 2). This, the methanol molecules act as acceptors and donors. In the direction of the *a* axis channels are formed in which the methanol molecules are located (Fig. 2).

Experimental

Betamethasone valerate (9-Fluor-11β,21-dihydroxy-16β-methyl-3,20 -dioxypregna-1,4-dien-17-ylpentanoat) was obtained from Symbiotec Pharmalab (India) as an enantiomeric pure compound. This compound was recrystallized at 70°C in an teflon lined steel autoclave. On cooling, single crystals of the title compound were obtained. The product is obtained as a pure phase, which was proven by comparison of the experimental X-ray powder pattern with that, calculated from single-crystal data.

Refinement

The C—H hydrogen atoms were positioned with idealized geometry (methyl H atoms allowed to rotate but not to tip) and were refined with fixed isotropic displacement parameters [U_{iso}(H)= 1.2*U_{eq}(C) or 1.5*U_{eq}(C) for methyl groups] using a riding model with d(C—H) = 0.95 Å for olefin, 1.00 Å for methin, 0.99 Å for methylen and 0.98 Å for methyl H atoms. The position of the hydroxyl hydrogen atoms were located in difference map but they were positioned with idealized geometry allowed to rotate but not to tip with d(O—H) = 0.84 Å and refined with fixed isotropic displacement parameters [U_{iso}(H)= 1.5*U_{eq}(O)] using a riding model. Because no strong anomalous scattering atoms are present, the absolute structure and absolute configuration cannot be determined. Therefore, Friedel opposites were merged prior to refinement and the absolute configuration was assigned base on the known absolute configuration of the starting material.

Figures

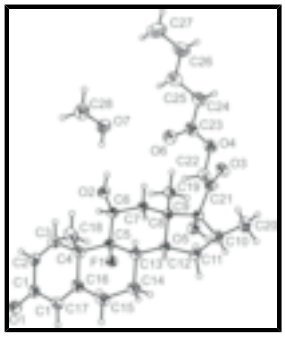

Fig. 1: View of the asymmetric unit of the title compound with labelling and displacement ellipsoids drawn at the 50% probability level.

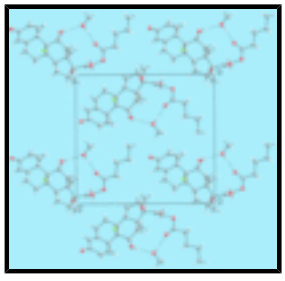

Fig. 2 Crystal structure of the title compound with view along the *a* axis (hydrogen bonding is shown as dashed lines).

Fig. 3 Reaction scheme

2-((8*S*,9*R*,10*S*,11S,13*S*,14S,16*S*,17*R*)-9-fluoro-11,17-dihydroxy- 10,13,16-trimethyl-3-oxo-6,7,8,9,10,11,12,13,14,15,16,17-dodecahydro-3H -cyclopenta[*a*]phenanthren- 17-yl)-2-oxoethyl pentanoate methanol solvate

Crystal data

$C_{27}H_{37}FO_6 \cdot CH_4O$	D_x = 1.261 Mg m^{-3} D_m = N Mg m^{-3} D_m measured by not measured
M_r = 508.61	Melting point: not measured K
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 9.9980 (5) Å	Cell parameters from 16982 reflections
b = 15.7885 (9) Å	θ = 2.4–28.0º
c = 16.9696 (12) Å	μ = 0.09 mm^{-1}
V = 2678.7 (3) Å^3	T = 166 (2) K
Z = 4	Needles, colorless
F_{000} = 1096	0.15 × 0.10 × 0.04 mm

Data collection

STOE IPDS-1 diffractometer	3009 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.049
Monochromator: graphite	θ_{max} = 28.0º
T = 166(2) K	θ_{min} = 2.4º
Phi scans	h = −13→12

Absorption correction: none
16982 measured reflections
3595 independent reflections

$k = -19 \rightarrow 20$
$l = -22 \rightarrow 22$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.042$

$wR(F^2) = 0.114$

$S = 1.03$

3595 reflections

330 parameters

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

$w = 1/[\sigma^2(F_o^2) + (0.0728P)^2 + 0.2844P]$ where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max} = 0.37$ e Å^{-3}

$\Delta\rho_{min} = -0.17$ e Å^{-3}

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

Extinction coefficient: 0.015 (3)

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}
O1	0.4547 (2)	0.35867 (12)	0.46094 (11)	0.0447 (5)
O2	0.7591 (2)	0.33239 (10)	0.10538 (10)	0.0341 (4)
H1O1	0.7493	0.3492	0.0588	0.051*
O3	0.86752 (19)	0.07486 (12)	−0.10976 (11)	0.0373 (4)
O4	0.6562 (2)	0.11246 (12)	−0.19919 (10)	0.0395 (4)
O5	0.60803 (17)	0.02899 (11)	0.02422 (11)	0.0339 (4)
H1O5	0.6045	−0.0240	0.0281	0.051*
O6	0.6920 (3)	0.23945 (14)	−0.14560 (12)	0.0545 (6)
O7	0.2372 (3)	0.11731 (13)	0.05510 (12)	0.0538 (6)
H1O7	0.2171	0.1575	0.0853	0.081*
F1	0.56997 (13)	0.19207 (9)	0.22638 (8)	0.0293 (3)
C1	0.5279 (3)	0.34132 (15)	0.40401 (14)	0.0321 (5)

C2	0.5175 (3)	0.38628 (15)	0.32881 (14)	0.0299 (5)
H2	0.4524	0.4296	0.3228	0.036*
C3	0.5977 (3)	0.36759 (14)	0.26878 (14)	0.0286 (5)
H3	0.5844	0.3969	0.2205	0.034*
C4	0.7082 (2)	0.30293 (16)	0.27228 (13)	0.0296 (5)
C5	0.6905 (2)	0.23474 (14)	0.20552 (12)	0.0244 (4)
C6	0.6600 (2)	0.27067 (14)	0.12304 (12)	0.0252 (4)
H6	0.5718	0.3003	0.1259	0.030*
C7	0.6487 (2)	0.20086 (14)	0.06002 (13)	0.0247 (4)
H7A	0.5648	0.1689	0.0687	0.030*
H7B	0.6431	0.2277	0.0074	0.030*
C8	0.7664 (2)	0.13873 (13)	0.06056 (12)	0.0233 (4)
C9	0.7443 (2)	0.05480 (14)	0.01248 (13)	0.0264 (5)
C10	0.8411 (3)	−0.01119 (15)	0.05322 (15)	0.0328 (5)
H10	0.7855	−0.0621	0.0656	0.039*
C11	0.8794 (3)	0.02912 (16)	0.13355 (14)	0.0331 (5)
H11A	0.8719	−0.0128	0.1767	0.040*
H11B	0.9719	0.0514	0.1323	0.040*
C12	0.7776 (2)	0.10131 (14)	0.14435 (13)	0.0265 (5)
H12	0.6896	0.0742	0.1565	0.032*
C13	0.8021 (2)	0.16737 (15)	0.20797 (13)	0.0288 (5)
H13	0.8897	0.1957	0.1973	0.035*
C14	0.8090 (3)	0.12488 (18)	0.28931 (14)	0.0397 (6)
H14A	0.8886	0.0876	0.2912	0.048*
H14B	0.7288	0.0889	0.2965	0.048*
C15	0.8168 (3)	0.1888 (2)	0.35744 (15)	0.0459 (7)
H15A	0.8055	0.1585	0.4081	0.055*
H15B	0.9062	0.2158	0.3576	0.055*
C16	0.7115 (3)	0.25614 (17)	0.35066 (14)	0.0331 (5)
C17	0.6280 (3)	0.27433 (17)	0.40949 (14)	0.0347 (6)
H17	0.6339	0.2424	0.4568	0.042*
C18	0.8407 (3)	0.35432 (19)	0.26525 (16)	0.0401 (6)
H18A	0.9170	0.3155	0.2676	0.060*
H18B	0.8464	0.3950	0.3087	0.060*
H18C	0.8419	0.3848	0.2150	0.060*
C19	0.8963 (2)	0.18166 (14)	0.03324 (13)	0.0267 (4)
H19A	0.9695	0.1404	0.0341	0.040*
H19B	0.9176	0.2288	0.0687	0.040*
H19C	0.8847	0.2032	−0.0205	0.040*
C20	0.9629 (3)	−0.04336 (18)	0.00755 (17)	0.0410 (6)
H20A	0.9336	−0.0683	−0.0424	0.062*
H20B	1.0095	−0.0863	0.0389	0.062*
H20C	1.0237	0.0040	−0.0031	0.062*
C21	0.7607 (3)	0.06993 (14)	−0.07560 (13)	0.0293 (5)
C22	0.6305 (3)	0.08233 (18)	−0.12067 (15)	0.0359 (6)
H22A	0.5816	0.0279	−0.1233	0.043*
H22B	0.5735	0.1236	−0.0924	0.043*
C23	0.6882 (3)	0.19431 (19)	−0.20325 (16)	0.0395 (6)
C24	0.7145 (3)	0.2230 (2)	−0.28574 (17)	0.0492 (8)

H24A	0.6439	0.1999	−0.3206	0.059*
H24B	0.8012	0.1994	−0.3034	0.059*
C25	0.7181 (3)	0.3179 (2)	−0.29465 (18)	0.0492 (7)
H25A	0.6345	0.3417	−0.2726	0.059*
H25B	0.7935	0.3403	−0.2631	0.059*
C26	0.7331 (5)	0.3483 (2)	−0.37817 (19)	0.0616 (9)
H26A	0.8181	0.3262	−0.3999	0.074*
H26B	0.6591	0.3249	−0.4103	0.074*
C27	0.7325 (6)	0.4429 (3)	−0.3852 (3)	0.0842 (14)
H27A	0.7415	0.4589	−0.4408	0.126*
H27B	0.8075	0.4664	−0.3551	0.126*
H27C	0.6482	0.4652	−0.3644	0.126*
C28	0.3002 (3)	0.0530 (2)	0.09857 (19)	0.0468 (7)
H28A	0.3839	0.0746	0.1209	0.070*
H28B	0.3195	0.0049	0.0639	0.070*
H28C	0.2410	0.0345	0.1413	0.070*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0559 (13)	0.0369 (9)	0.0414 (10)	0.0089 (9)	0.0246 (9)	0.0001 (8)
O2	0.0466 (11)	0.0262 (8)	0.0294 (8)	−0.0058 (8)	0.0022 (8)	0.0026 (6)
O3	0.0330 (10)	0.0468 (11)	0.0322 (9)	0.0017 (8)	0.0091 (7)	−0.0023 (8)
O4	0.0453 (11)	0.0422 (10)	0.0309 (9)	0.0044 (9)	−0.0036 (8)	−0.0048 (8)
O5	0.0265 (9)	0.0286 (8)	0.0467 (10)	−0.0063 (7)	0.0078 (7)	−0.0001 (8)
O6	0.0805 (17)	0.0417 (11)	0.0412 (11)	−0.0009 (11)	0.0090 (11)	−0.0043 (9)
O7	0.0847 (17)	0.0376 (10)	0.0392 (10)	0.0193 (11)	−0.0032 (11)	0.0084 (8)
F1	0.0257 (7)	0.0326 (7)	0.0295 (7)	0.0000 (6)	0.0054 (5)	0.0035 (5)
C1	0.0343 (13)	0.0287 (11)	0.0333 (12)	0.0015 (10)	0.0064 (10)	−0.0052 (9)
C2	0.0299 (13)	0.0234 (10)	0.0366 (12)	0.0041 (9)	0.0015 (10)	−0.0032 (9)
C3	0.0311 (12)	0.0261 (10)	0.0284 (11)	−0.0004 (9)	−0.0020 (9)	−0.0015 (9)
C4	0.0273 (12)	0.0370 (12)	0.0245 (10)	0.0042 (10)	0.0009 (8)	−0.0014 (9)
C5	0.0221 (11)	0.0283 (10)	0.0228 (9)	0.0003 (9)	0.0029 (8)	0.0023 (8)
C6	0.0254 (11)	0.0259 (10)	0.0243 (10)	0.0048 (9)	0.0002 (8)	0.0023 (8)
C7	0.0240 (11)	0.0271 (10)	0.0230 (9)	0.0030 (9)	−0.0003 (8)	0.0023 (8)
C8	0.0221 (11)	0.0241 (9)	0.0237 (9)	0.0023 (8)	0.0027 (8)	0.0023 (8)
C9	0.0233 (11)	0.0246 (10)	0.0314 (11)	−0.0010 (9)	0.0057 (9)	0.0001 (8)
C10	0.0320 (13)	0.0263 (11)	0.0402 (13)	0.0047 (10)	0.0076 (10)	0.0027 (9)
C11	0.0348 (14)	0.0309 (11)	0.0336 (12)	0.0128 (10)	0.0039 (10)	0.0059 (9)
C12	0.0269 (12)	0.0263 (10)	0.0263 (10)	0.0058 (9)	0.0042 (9)	0.0048 (8)
C13	0.0268 (12)	0.0354 (12)	0.0243 (10)	0.0094 (9)	0.0004 (9)	0.0033 (9)
C14	0.0453 (16)	0.0471 (15)	0.0267 (11)	0.0214 (13)	−0.0002 (11)	0.0070 (10)
C15	0.0485 (17)	0.0626 (18)	0.0266 (11)	0.0278 (15)	−0.0038 (11)	0.0038 (12)
C16	0.0343 (14)	0.0396 (13)	0.0255 (11)	0.0092 (11)	−0.0037 (9)	−0.0015 (10)
C17	0.0448 (15)	0.0361 (13)	0.0233 (10)	0.0066 (11)	0.0021 (10)	−0.0013 (9)
C18	0.0303 (14)	0.0520 (16)	0.0381 (13)	−0.0047 (12)	−0.0011 (11)	−0.0109 (12)
C19	0.0248 (11)	0.0281 (11)	0.0272 (10)	−0.0024 (9)	0.0022 (8)	0.0015 (9)
C20	0.0403 (15)	0.0383 (14)	0.0444 (14)	0.0138 (12)	0.0049 (12)	−0.0032 (11)

C21	0.0321 (13)	0.0254 (10)	0.0306 (11)	−0.0016 (9)	0.0042 (9)	−0.0048 (8)
C22	0.0342 (14)	0.0407 (13)	0.0328 (12)	0.0001 (11)	0.0010 (10)	−0.0056 (10)
C23	0.0354 (14)	0.0465 (15)	0.0365 (13)	0.0094 (12)	0.0024 (11)	−0.0023 (11)
C24	0.0498 (19)	0.0592 (18)	0.0386 (14)	0.0138 (15)	0.0078 (13)	0.0033 (13)
C25	0.0459 (18)	0.0598 (18)	0.0419 (15)	−0.0004 (15)	0.0050 (13)	−0.0012 (13)
C26	0.069 (2)	0.071 (2)	0.0439 (16)	0.002 (2)	0.0097 (17)	0.0088 (15)
C27	0.093 (3)	0.070 (2)	0.089 (3)	0.008 (3)	0.026 (3)	0.034 (2)
C28	0.0436 (17)	0.0467 (15)	0.0502 (16)	0.0134 (13)	0.0040 (13)	0.0122 (13)

Geometric parameters (Å, °)

O1—C1	1.243 (3)	C12—H12	1.0000
O2—C6	1.421 (3)	C13—C14	1.536 (3)
O2—H1O1	0.8400	C13—H13	1.0000
O3—C21	1.217 (3)	C14—C15	1.537 (4)
O4—C23	1.333 (4)	C14—H14A	0.9900
O4—C22	1.438 (3)	C14—H14B	0.9900
O5—C9	1.436 (3)	C15—C16	1.499 (4)
O5—H1O5	0.8400	C15—H15A	0.9900
O6—C23	1.211 (3)	C15—H15B	0.9900
O7—C28	1.404 (3)	C16—C17	1.333 (3)
O7—H1O7	0.8400	C17—H17	0.9500
F1—C5	1.426 (3)	C18—H18A	0.9800
C1—C17	1.459 (4)	C18—H18B	0.9800
C1—C2	1.464 (3)	C18—H18C	0.9800
C2—C3	1.329 (3)	C19—H19A	0.9800
C2—H2	0.9500	C19—H19B	0.9800
C3—C4	1.505 (3)	C19—H19C	0.9800
C3—H3	0.9500	C20—H20A	0.9800
C4—C16	1.522 (3)	C20—H20B	0.9800
C4—C18	1.558 (4)	C20—H20C	0.9800
C4—C5	1.573 (3)	C21—C22	1.523 (4)
C5—C6	1.541 (3)	C22—H22A	0.9900
C5—C13	1.542 (3)	C22—H22B	0.9900
C6—C7	1.540 (3)	C23—C24	1.495 (4)
C6—H6	1.0000	C24—C25	1.506 (5)
C7—C8	1.532 (3)	C24—H24A	0.9900
C7—H7A	0.9900	C24—H24B	0.9900
C7—H7B	0.9900	C25—C26	1.504 (4)
C8—C19	1.537 (3)	C25—H25A	0.9900
C8—C12	1.544 (3)	C25—H25B	0.9900
C8—C9	1.572 (3)	C26—C27	1.498 (6)
C9—C21	1.523 (3)	C26—H26A	0.9900
C9—C10	1.581 (3)	C26—H26B	0.9900
C10—C20	1.531 (4)	C27—H27A	0.9800
C10—C11	1.552 (4)	C27—H27B	0.9800
C10—H10	1.0000	C27—H27C	0.9800
C11—C12	1.539 (3)	C28—H28A	0.9800
C11—H11A	0.9900	C28—H28B	0.9800

C11—H11B	0.9900	C28—H28C	0.9800
C12—C13	1.521 (3)		
C6—O2—H1O1	109.5	C13—C14—H14B	109.0
C23—O4—C22	114.3 (2)	C15—C14—H14B	109.0
C9—O5—H1O5	109.5	H14A—C14—H14B	107.8
C28—O7—H1O7	109.5	C16—C15—C14	111.9 (2)
O1—C1—C17	120.9 (2)	C16—C15—H15A	109.2
O1—C1—C2	121.9 (2)	C14—C15—H15A	109.2
C17—C1—C2	117.1 (2)	C16—C15—H15B	109.2
C3—C2—C1	121.2 (2)	C14—C15—H15B	109.2
C3—C2—H2	119.4	H15A—C15—H15B	107.9
C1—C2—H2	119.4	C17—C16—C15	122.3 (2)
C2—C3—C4	124.3 (2)	C17—C16—C4	122.4 (2)
C2—C3—H3	117.9	C15—C16—C4	115.2 (2)
C4—C3—H3	117.9	C16—C17—C1	122.6 (2)
C3—C4—C16	112.32 (19)	C16—C17—H17	118.7
C3—C4—C18	105.5 (2)	C1—C17—H17	118.7
C16—C4—C18	107.5 (2)	C4—C18—H18A	109.5
C3—C4—C5	110.70 (19)	C4—C18—H18B	109.5
C16—C4—C5	107.44 (19)	H18A—C18—H18B	109.5
C18—C4—C5	113.38 (19)	C4—C18—H18C	109.5
F1—C5—C6	103.42 (17)	H18A—C18—H18C	109.5
F1—C5—C13	106.20 (17)	H18B—C18—H18C	109.5
C6—C5—C13	114.95 (18)	C8—C19—H19A	109.5
F1—C5—C4	103.86 (16)	C8—C19—H19B	109.5
C6—C5—C4	115.12 (18)	H19A—C19—H19B	109.5
C13—C5—C4	111.82 (18)	C8—C19—H19C	109.5
O2—C6—C7	113.29 (18)	H19A—C19—H19C	109.5
O2—C6—C5	107.81 (18)	H19B—C19—H19C	109.5
C7—C6—C5	112.45 (18)	C10—C20—H20A	109.5
O2—C6—H6	107.7	C10—C20—H20B	109.5
C7—C6—H6	107.7	H20A—C20—H20B	109.5
C5—C6—H6	107.7	C10—C20—H20C	109.5
C8—C7—C6	113.44 (18)	H20A—C20—H20C	109.5
C8—C7—H7A	108.9	H20B—C20—H20C	109.5
C6—C7—H7A	108.9	O3—C21—C9	124.9 (2)
C8—C7—H7B	108.9	O3—C21—C22	120.2 (2)
C6—C7—H7B	108.9	C9—C21—C22	114.9 (2)
H7A—C7—H7B	107.7	O4—C22—C21	110.8 (2)
C7—C8—C19	111.41 (17)	O4—C22—H22A	109.5
C7—C8—C12	107.84 (17)	C21—C22—H22A	109.5
C19—C8—C12	112.66 (18)	O4—C22—H22B	109.5
C7—C8—C9	115.37 (19)	C21—C22—H22B	109.5
C19—C8—C9	109.53 (17)	H22A—C22—H22B	108.1
C12—C8—C9	99.53 (16)	O6—C23—O4	122.4 (3)
O5—C9—C21	106.5 (2)	O6—C23—C24	124.9 (3)
O5—C9—C8	107.51 (18)	O4—C23—C24	112.6 (2)
C21—C9—C8	111.22 (17)	C23—C24—C25	113.6 (3)
O5—C9—C10	109.46 (18)	C23—C24—H24A	108.9

C21—C9—C10	117.8 (2)	C25—C24—H24A	108.9
C8—C9—C10	104.02 (19)	C23—C24—H24B	108.9
C20—C10—C11	112.6 (2)	C25—C24—H24B	108.9
C20—C10—C9	119.0 (2)	H24A—C24—H24B	107.7
C11—C10—C9	105.36 (18)	C26—C25—C24	114.5 (3)
C20—C10—H10	106.4	C26—C25—H25A	108.6
C11—C10—H10	106.4	C24—C25—H25A	108.6
C9—C10—H10	106.4	C26—C25—H25B	108.6
C12—C11—C10	104.20 (19)	C24—C25—H25B	108.6
C12—C11—H11A	110.9	H25A—C25—H25B	107.6
C10—C11—H11A	110.9	C27—C26—C25	113.1 (3)
C12—C11—H11B	110.9	C27—C26—H26A	109.0
C10—C11—H11B	110.9	C25—C26—H26A	109.0
H11A—C11—H11B	108.9	C27—C26—H26B	109.0
C13—C12—C11	119.1 (2)	C25—C26—H26B	109.0
C13—C12—C8	113.76 (18)	H26A—C26—H26B	107.8
C11—C12—C8	102.81 (17)	C26—C27—H27A	109.5
C13—C12—H12	106.8	C26—C27—H27B	109.5
C11—C12—H12	106.8	H27A—C27—H27B	109.5
C8—C12—H12	106.8	C26—C27—H27C	109.5
C12—C13—C14	110.2 (2)	H27A—C27—H27C	109.5
C12—C13—C5	109.73 (19)	H27B—C27—H27C	109.5
C14—C13—C5	110.97 (19)	O7—C28—H28A	109.5
C12—C13—H13	108.6	O7—C28—H28B	109.5
C14—C13—H13	108.6	H28A—C28—H28B	109.5
C5—C13—H13	108.6	O7—C28—H28C	109.5
C13—C14—C15	113.0 (2)	H28A—C28—H28C	109.5
C13—C14—H14A	109.0	H28B—C28—H28C	109.5
C15—C14—H14A	109.0		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H1O1···O7^{i}	0.84	2.01	2.845 (3)	176
O5—H1O5···O1^{ii}	0.84	1.95	2.773 (2)	165
O7—H1O7···O6^{iii}	0.84	1.94	2.771 (3)	171

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

Fig. 1

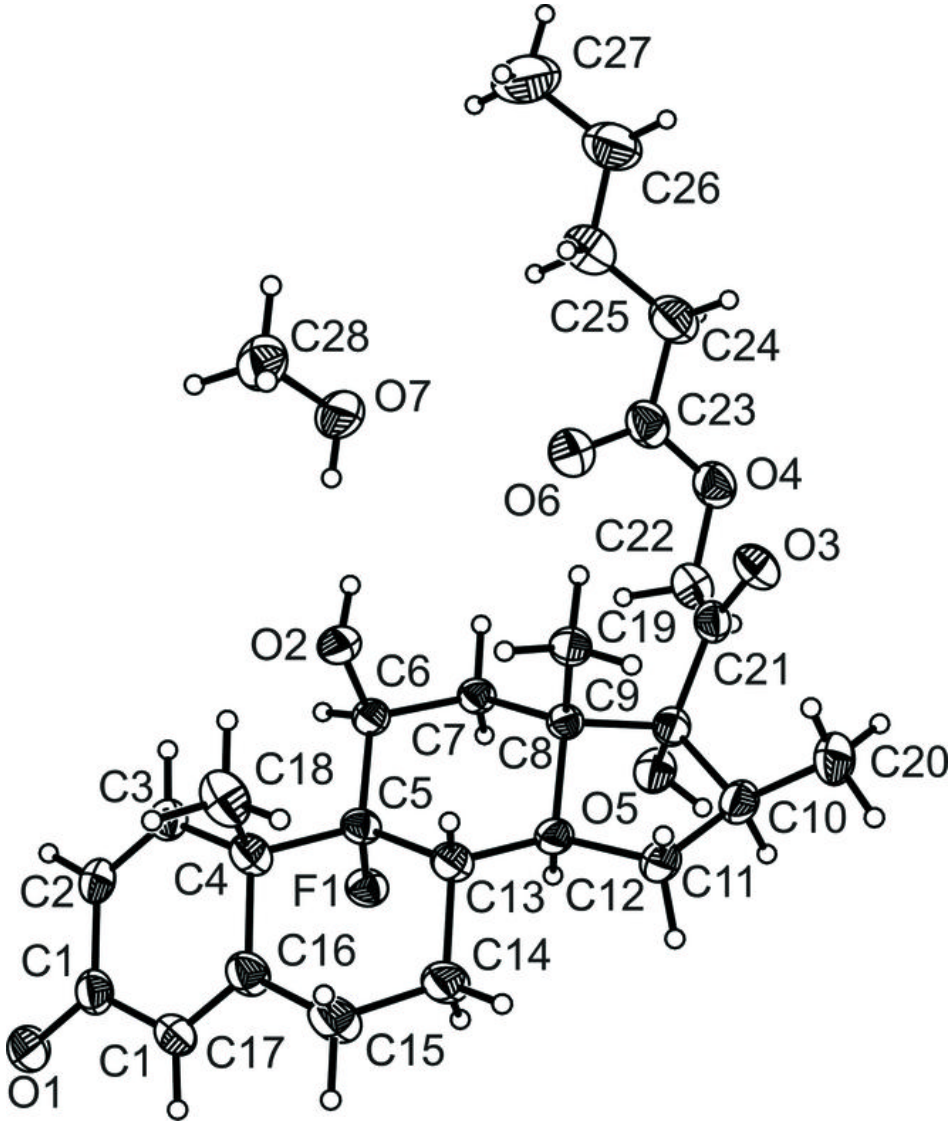
C27
C26
C25
C24
C28
O7
C23
O6
O4
C22
O3
C19
O2
C6
C9
C21
C7
C8
C18
C20
C3
C5
O5
C10
C4
F1
C13
C12
C11
C2
C16
C14
C1
C17
C15
C1
O1

Fig. 2