Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(9*R*,15*R*)-Methyl 3,15-diacetyl-4-(2-cyanoethyl)-4,9,10-trimethyl-perhydrocyclopenta[*a*]phenanthrene-13-carboxylate

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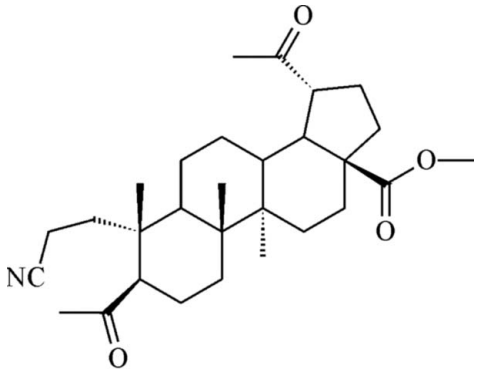
Received 29 March 2007; accepted 12 April 2007

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.036; wR factor = 0.075; data-to-parameter ratio = 12.5.

The title compound, $C_{29}H_{43}NO_4$, a seco-lupane triterpene, was synthesized from naturally occurring betulin. In the molecule, all bond lengths and angles are normal. The crystal structure is stabilized by weak intermolecular C—H···O hydrogen bonds and ordinary van der Waals interactions.

Related literature

For related literature, see: Allen (2002); Allen *et al.* (1987); Krasutsky (2006); Tolstikova *et al.* (2006); Urban *et al.* (2005); Valterova *et al.* (1983).



Experimental

Crystal data

$C_{29}H_{43}NO_4$
M_r = 469.64
Monoclinic, $P2_1$
a = 9.6438 (6) Å
b = 8.9672 (5) Å
c = 15.0549 (9) Å
β = 103.2030 (10)°
V = 1267.50 (13) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 100 (2) K
0.35 × 0.25 × 0.20 mm

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer
Absorption correction: none
22169 measured reflections
3928 independent reflections
3240 reflections with $I > 2\sigma(I)$
R_{int} = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.075
S = 1.04
3928 reflections
313 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C28—H28A···O1^{i}	0.98	2.60	3.178 (2)	118
C28—H28B···O1^{ii}	0.98	2.59	3.272 (3)	127

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

This work was supported by the Russian Foundation for Basic Research (Grant No 05–03-82832) and the Federal Program on Support of Leading Scientific Schools (Grant No SS-1153.2006.3).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RK2010).

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 doi:10.1107/S1600536807018223

supplementary materials

Acta Cryst. (2007). E**63**, o2603 [**doi:10.1107/S1600536807018223**]

(9*R*,15*R*)-Methyl 3,15-diacetyl-4-(2-cyanoethyl)-4,9,10-trimethylperhydrocyclopenta[*a*]phenanthrene-13-carboxylate

O. B. Flekhter, N. I. Medvedeva and K. Yu. Suponitsky

Comment

In the last decade, the real burst of activity in the pharmacological properties of lupane triterpenoids was observed after revealing promising antiviral and antineoplastic agents among these compounds (Tolstikova *et al.*, 2006; Krasutsky, 2006). The influence of A-ring modification in lupane series on the increasing of anti-cancer activity has been shown (Urban *et al.*, 2005). In this connection design and synthesis of new seco-lupane triterpenoids is of great interest. Herein we report the crystal structure of the title compound, (I), which was synthesized via ozonolysis of 3-cyano-3,4-seco-4(23),20 (29)-lupdiene-28-oic acid methyl ester described by Valterova *et al.* (1983).

In (I) (Fig.1), all bond lengths and angles show normal values (Allen *et al.*, 1987; CSD, Version 5.27, Allen, 2002). The cyclohexane rings adopt a chair conformation. The five-membered cycle is characterized by envelope conformation with the C17 atom being displaced from C18—C19—C21—C22 plane so that the C17—C18—C19—C21 and C19—C21—C22—C17 torsion angles are equal to 26.4 (2) and -27.3 (2)° correspondingly. The value of the C22—C21—C19—C20 torsion angle which describes the orientation of the acetyl group is equal to -120.4 (2)°. The acetyl group attached to the C5 atom adopts the equatorial position as well as the cyanoethyl substituent attached to the C10 atom.

The crystal packing is stabilized by the weak intermolecular C–H···O hydrogen bonds (Table 1) and ordinary Van der Waals interactions.

Experimental

A solution of 3-cyano-3,4-seco-4(23),20 (29)-lupdiene-28-oic acid methyl ester (2 mmol, 0.94 g) (obtained as described by Valterova *et al.* (1983)) in CH_2Cl_2 (50 ml) was treated with ozone at 213 K. Then AcOH (10 ml) and Zn dust (1 g) were added at 273 K. After stirring for 1 h organic layer was filtered, washed by the saturated aqueous Na_2CO_3 (3×20 ml), H_2O (3×20 ml), dried over Na_2SO_4. The solvent was evaporated in vacuo. Crude material was subjected to column chromatography on silica gel and eluted with $CHCl_3$ to yield 0.65 g (70%) of the pure product which was crystallized from $CHCl_3$—MeOH for a few days to obtain colorless single crystals of (I) suitable for the X-ray analysis.

Refinement

The H(C) atoms were positioned geometrically with C–H distances of 0.98Å for the mehtyl groups and 0.99Å for all the other hydrogen atoms. All the H atoms were refined within the riding model with $U_{iso}(H)=1.5U_{eq}$(parent atom) for the methyl groups and $U_{iso}(H)=1.2U_{eq}$(parent atom) for the other atoms. Due to the absence of any significant anomalous scatterers in the molecule, the 3429 Friedel pairs were merged before the final refinement.

Crystallographic data for (I) have been deposited with the Cambridge Crystallographic Data Centre (CCDC No. 630832).

Figures

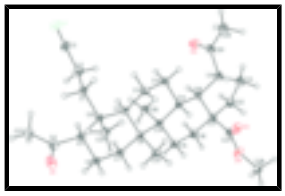


Fig. 1. View of (I), showing the atom-numbering scheme and displacement ellipsoids drawn at the 50% probability level. Hydrogen atoms are presented as spheres of arbitrary radius.

(9*R*,15R)-Methyl 3,15-diacetyl-4-(2-cyanoethyl)-4,9,10- trimethylperhydrocyclopenta[a]phenanthrene-13-carboxylate

Crystal data

$C_{29}H_{43}NO_4$	F_{000} = 512
M_r = 469.64	D_x = 1.231 Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2yb	Cell parameters from 4959 reflections
a = 9.6438 (6) Å	θ = 2.3–29.4º
b = 8.9672 (5) Å	μ = 0.08 mm^{-1}
c = 15.0549 (9) Å	T = 100 (2) K
β = 103.2030 (10)º	Prism, colorless
V = 1267.50 (13) Å^3	0.35 × 0.25 × 0.20 mm
Z = 2	

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer	3240 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.044
Monochromator: graphite	θ_{max} = 30.1º
T = 100(2) K	θ_{min} = 2.2º
φ and ω scans	h = −13→13
Absorption correction: none	k = −12→12
22169 measured reflections	l = −21→21
3928 independent reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.036	$w = 1/[\sigma^2(F_o^2) + (0.034P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.075	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.04	$\Delta\rho_{max}$ = 0.28 e Å^{-3}

3928 reflections	$\Delta\rho_{min} = -0.15$ e Å^{-3}
313 parameters	Extinction correction: none
1 restraint	Absolute structure: Flack (1983)
Primary atom site location: structure-invariant direct methods	Flack parameter: 0 (10)
Secondary atom site location: difference Fourier map	

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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	0.81804 (13)	0.42597 (15)	0.44072 (9)	0.0291 (3)
O2	−0.01272 (14)	0.91331 (16)	−0.01542 (8)	0.0284 (3)
O3	−0.08700 (14)	0.88309 (15)	0.32481 (9)	0.0290 (3)
O4	−0.04260 (14)	1.11365 (15)	0.38054 (9)	0.0254 (3)
N1	0.58447 (18)	0.2989 (2)	0.00197 (10)	0.0312 (4)
C1	0.56180 (17)	0.41733 (19)	0.21485 (10)	0.0168 (3)
H1A	0.6443	0.3509	0.2375	0.020*
H1B	0.4765	0.3531	0.1967	0.020*
C2	0.5850 (2)	0.4973 (2)	0.12878 (12)	0.0234 (4)
H2A	0.5073	0.5700	0.1072	0.028*
H2B	0.6763	0.5526	0.1435	0.028*
C3	0.58753 (19)	0.3876 (2)	0.05651 (12)	0.0228 (4)
C4	0.81035 (18)	0.5007 (2)	0.37217 (12)	0.0210 (4)
C5	0.68342 (17)	0.60158 (19)	0.33681 (11)	0.0168 (3)
H5A	0.7066	0.6631	0.2867	0.020*
C6	0.66693 (17)	0.70940 (19)	0.41249 (11)	0.0169 (3)
H6A	0.6377	0.6534	0.4619	0.020*
H6B	0.7596	0.7578	0.4385	0.020*
C7	0.55630 (17)	0.82771 (18)	0.37542 (11)	0.0162 (3)
H7A	0.5910	0.8890	0.3303	0.019*
H7B	0.5464	0.8942	0.4261	0.019*
C8	0.40807 (17)	0.76478 (18)	0.32973 (10)	0.0136 (3)
C9	0.42655 (17)	0.63972 (18)	0.25990 (11)	0.0143 (3)
H9A	0.4628	0.6940	0.2117	0.017*
C10	0.54217 (17)	0.51798 (18)	0.29564 (11)	0.0145 (3)
C11	0.28304 (17)	0.57591 (19)	0.20955 (11)	0.0179 (3)
H11A	0.2988	0.5012	0.1644	0.021*

H11B	0.2376	0.5246	0.2538	0.021*
C12	0.18349 (18)	0.69727 (19)	0.16060 (11)	0.0178 (3)
H12A	0.0894	0.6531	0.1331	0.021*
H12B	0.2228	0.7396	0.1108	0.021*
C13	0.16510 (17)	0.82152 (18)	0.22637 (11)	0.0148 (3)
H13A	0.1242	0.7750	0.2751	0.018*
C14	0.31060 (16)	0.89212 (18)	0.27426 (10)	0.0139 (3)
C15	0.28187 (18)	1.02064 (18)	0.33786 (11)	0.0169 (3)
H15A	0.2451	0.9768	0.3883	0.020*
H15B	0.3732	1.0706	0.3651	0.020*
C16	0.17522 (18)	1.13830 (19)	0.28897 (12)	0.0194 (4)
H16A	0.1607	1.2151	0.3332	0.023*
H16B	0.2136	1.1880	0.2410	0.023*
C17	0.03325 (17)	1.06386 (19)	0.24615 (12)	0.0176 (3)
C18	0.06059 (17)	0.94109 (19)	0.18050 (11)	0.0168 (3)
H18A	0.1074	0.9921	0.1360	0.020*
C19	−0.08772 (17)	0.8938 (2)	0.12546 (11)	0.0201 (3)
H19A	−0.1244	0.8084	0.1562	0.024*
C20	−0.09095 (18)	0.85498 (19)	0.02707 (12)	0.0210 (4)
C21	−0.1825 (2)	1.0356 (2)	0.12690 (14)	0.0275 (4)
H21A	−0.2235	1.0706	0.0640	0.033*
H21B	−0.2613	1.0130	0.1569	0.033*
C22	−0.08328 (19)	1.1552 (2)	0.18104 (13)	0.0239 (4)
H22A	−0.1353	1.2198	0.2157	0.029*
H22B	−0.0415	1.2181	0.1398	0.029*
C23	0.92889 (19)	0.5040 (2)	0.32213 (13)	0.0259 (4)
H23A	0.9981	0.4255	0.3460	0.039*
H23B	0.9762	0.6014	0.3309	0.039*
H23C	0.8895	0.4873	0.2569	0.039*
C24	0.50213 (17)	0.4071 (2)	0.36394 (11)	0.0181 (3)
H24A	0.5646	0.3197	0.3698	0.027*
H24B	0.4029	0.3757	0.3419	0.027*
H24C	0.5135	0.4554	0.4236	0.027*
C25	0.34075 (17)	0.7021 (2)	0.40602 (11)	0.0168 (3)
H25A	0.4138	0.6500	0.4514	0.025*
H25B	0.2644	0.6322	0.3795	0.025*
H25C	0.3015	0.7843	0.4355	0.025*
C26	0.38475 (18)	0.96652 (19)	0.20385 (11)	0.0178 (3)
H26A	0.3136	1.0193	0.1577	0.027*
H26B	0.4307	0.8896	0.1742	0.027*
H26C	0.4567	1.0375	0.2353	0.027*
C27	−0.03637 (17)	1.0043 (2)	0.32057 (12)	0.0200 (4)
C28	−0.1174 (2)	1.0780 (2)	0.45091 (13)	0.0275 (4)
H28A	−0.1034	1.1585	0.4961	0.041*
H28B	−0.0802	0.9845	0.4808	0.041*
H28C	−0.2193	1.0669	0.4235	0.041*
C29	−0.2014 (2)	0.7430 (2)	−0.01687 (13)	0.0277 (4)
H29A	−0.2181	0.7514	−0.0834	0.042*
H29B	−0.2904	0.7623	0.0021	0.042*

H29C	−0.1676	0.6422	0.0020	0.042*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0249 (7)	0.0290 (7)	0.0327 (7)	0.0033 (6)	0.0051 (6)	0.0120 (6)
O2	0.0330 (7)	0.0298 (7)	0.0219 (6)	−0.0016 (7)	0.0050 (6)	0.0015 (6)
O3	0.0333 (7)	0.0242 (7)	0.0345 (7)	−0.0056 (6)	0.0180 (6)	−0.0054 (6)
O4	0.0321 (7)	0.0211 (7)	0.0276 (7)	−0.0016 (6)	0.0163 (6)	−0.0035 (5)
N1	0.0383 (10)	0.0336 (10)	0.0241 (8)	0.0031 (8)	0.0121 (8)	−0.0035 (7)
C1	0.0196 (8)	0.0145 (8)	0.0172 (8)	0.0024 (7)	0.0062 (6)	0.0001 (7)
C2	0.0327 (10)	0.0205 (9)	0.0186 (8)	0.0027 (8)	0.0090 (7)	0.0002 (7)
C3	0.0252 (9)	0.0276 (10)	0.0175 (8)	0.0071 (8)	0.0090 (7)	0.0035 (8)
C4	0.0190 (8)	0.0182 (8)	0.0257 (9)	−0.0024 (7)	0.0051 (7)	−0.0020 (7)
C5	0.0160 (8)	0.0187 (8)	0.0164 (8)	0.0007 (7)	0.0050 (6)	0.0020 (7)
C6	0.0144 (8)	0.0188 (8)	0.0167 (8)	−0.0024 (7)	0.0017 (6)	−0.0007 (7)
C7	0.0176 (8)	0.0163 (8)	0.0148 (7)	−0.0026 (7)	0.0038 (6)	−0.0023 (7)
C8	0.0148 (8)	0.0144 (8)	0.0126 (7)	−0.0009 (6)	0.0046 (6)	−0.0011 (6)
C9	0.0168 (8)	0.0132 (8)	0.0132 (7)	0.0005 (6)	0.0042 (6)	−0.0004 (6)
C10	0.0158 (7)	0.0137 (8)	0.0146 (7)	0.0016 (6)	0.0045 (6)	0.0009 (6)
C11	0.0177 (8)	0.0159 (8)	0.0183 (8)	0.0028 (7)	0.0005 (7)	−0.0034 (7)
C12	0.0171 (8)	0.0175 (8)	0.0172 (8)	0.0019 (7)	0.0009 (7)	−0.0038 (7)
C13	0.0153 (8)	0.0138 (8)	0.0162 (7)	0.0007 (6)	0.0053 (6)	−0.0018 (6)
C14	0.0153 (7)	0.0132 (7)	0.0135 (7)	−0.0010 (6)	0.0039 (6)	−0.0024 (6)
C15	0.0197 (8)	0.0151 (8)	0.0168 (8)	−0.0006 (7)	0.0062 (7)	−0.0031 (7)
C16	0.0208 (9)	0.0155 (8)	0.0233 (9)	0.0001 (7)	0.0081 (7)	−0.0025 (7)
C17	0.0179 (8)	0.0144 (8)	0.0209 (8)	0.0005 (7)	0.0053 (7)	−0.0028 (7)
C18	0.0157 (8)	0.0171 (8)	0.0181 (8)	0.0020 (7)	0.0048 (6)	−0.0018 (7)
C19	0.0168 (8)	0.0203 (9)	0.0222 (8)	0.0045 (7)	0.0026 (7)	−0.0011 (7)
C20	0.0190 (8)	0.0194 (9)	0.0217 (8)	0.0072 (7)	−0.0015 (7)	0.0010 (7)
C21	0.0211 (9)	0.0275 (10)	0.0318 (10)	0.0083 (8)	0.0016 (8)	−0.0062 (8)
C22	0.0227 (9)	0.0201 (9)	0.0291 (10)	0.0063 (7)	0.0062 (8)	0.0010 (8)
C23	0.0207 (9)	0.0257 (9)	0.0323 (10)	0.0032 (8)	0.0079 (8)	−0.0006 (8)
C24	0.0180 (8)	0.0188 (8)	0.0177 (8)	−0.0007 (7)	0.0046 (6)	0.0032 (7)
C25	0.0191 (8)	0.0176 (8)	0.0156 (8)	−0.0012 (7)	0.0077 (7)	0.0002 (7)
C26	0.0186 (8)	0.0180 (8)	0.0186 (8)	0.0003 (7)	0.0076 (7)	0.0017 (7)
C27	0.0172 (8)	0.0205 (9)	0.0228 (9)	0.0032 (7)	0.0057 (7)	−0.0020 (7)
C28	0.0304 (10)	0.0292 (10)	0.0271 (10)	−0.0017 (9)	0.0157 (8)	−0.0041 (8)
C29	0.0277 (10)	0.0271 (10)	0.0260 (9)	0.0001 (8)	0.0015 (8)	−0.0047 (8)

Geometric parameters (Å, °)

O1—C4	1.219 (2)	C14—C26	1.558 (2)
O2—C20	1.213 (2)	C14—C15	1.563 (2)
O3—C27	1.199 (2)	C15—C16	1.538 (2)
O4—C27	1.344 (2)	C15—H15A	0.9900
O4—C28	1.447 (2)	C15—H15B	0.9900
N1—C3	1.139 (2)	C16—C17	1.527 (2)
C1—C2	1.542 (2)	C16—H16A	0.9900

C1—C10	1.560 (2)	C16—H16B	0.9900
C1—H1A	0.9900	C17—C27	1.528 (2)
C1—H1B	0.9900	C17—C18	1.542 (2)
C2—C3	1.471 (3)	C17—C22	1.546 (2)
C2—H2A	0.9900	C18—C19	1.540 (2)
C2—H2B	0.9900	C18—H18A	1.0000
C4—C23	1.506 (2)	C19—C20	1.515 (2)
C4—C5	1.517 (2)	C19—C21	1.569 (2)
C5—C6	1.530 (2)	C19—H19A	1.0000
C5—C10	1.554 (2)	C20—C29	1.503 (2)
C5—H5A	1.0000	C21—C22	1.540 (3)
C6—C7	1.518 (2)	C21—H21A	0.9900
C6—H6A	0.9900	C21—H21B	0.9900
C6—H6B	0.9900	C22—H22A	0.9900
C7—C8	1.545 (2)	C22—H22B	0.9900
C7—H7A	0.9900	C23—H23A	0.9800
C7—H7B	0.9900	C23—H23B	0.9800
C8—C25	1.548 (2)	C23—H23C	0.9800
C8—C9	1.575 (2)	C24—H24A	0.9800
C8—C14	1.589 (2)	C24—H24B	0.9800
C9—C11	1.529 (2)	C24—H24C	0.9800
C9—C10	1.565 (2)	C25—H25A	0.9800
C9—H9A	1.0000	C25—H25B	0.9800
C10—C24	1.542 (2)	C25—H25C	0.9800
C11—C12	1.525 (2)	C26—H26A	0.9800
C11—H11A	0.9900	C26—H26B	0.9800
C11—H11B	0.9900	C26—H26C	0.9800
C12—C13	1.528 (2)	C28—H28A	0.9800
C12—H12A	0.9900	C28—H28B	0.9800
C12—H12B	0.9900	C28—H28C	0.9800
C13—C18	1.525 (2)	C29—H29A	0.9800
C13—C14	1.558 (2)	C29—H29B	0.9800
C13—H13A	1.0000	C29—H29C	0.9800
C27—O4—C28	115.91 (14)	C14—C15—H15B	108.8
C2—C1—C10	116.92 (14)	H15A—C15—H15B	107.7
C2—C1—H1A	108.1	C17—C16—C15	109.82 (14)
C10—C1—H1A	108.1	C17—C16—H16A	109.7
C2—C1—H1B	108.1	C15—C16—H16A	109.7
C10—C1—H1B	108.1	C17—C16—H16B	109.7
H1A—C1—H1B	107.3	C15—C16—H16B	109.7
C3—C2—C1	109.90 (15)	H16A—C16—H16B	108.2
C3—C2—H2A	109.7	C16—C17—C27	110.18 (14)
C1—C2—H2A	109.7	C16—C17—C18	108.34 (13)
C3—C2—H2B	109.7	C27—C17—C18	112.85 (14)
C1—C2—H2B	109.7	C16—C17—C22	119.19 (14)
H2A—C2—H2B	108.2	C27—C17—C22	105.82 (13)
N1—C3—C2	176.7 (2)	C18—C17—C22	100.24 (13)
O1—C4—C23	121.87 (16)	C13—C18—C19	119.12 (14)
O1—C4—C5	120.88 (15)	C13—C18—C17	113.83 (13)

C23—C4—C5	117.17 (16)	C19—C18—C17	105.54 (13)
C4—C5—C6	109.15 (13)	C13—C18—H18A	105.8
C4—C5—C10	114.57 (14)	C19—C18—H18A	105.8
C6—C5—C10	111.78 (13)	C17—C18—H18A	105.8
C4—C5—H5A	107.0	C20—C19—C18	113.49 (13)
C6—C5—H5A	107.0	C20—C19—C21	108.51 (14)
C10—C5—H5A	107.0	C18—C19—C21	103.96 (14)
C7—C6—C5	110.60 (13)	C20—C19—H19A	110.2
C7—C6—H6A	109.5	C18—C19—H19A	110.2
C5—C6—H6A	109.5	C21—C19—H19A	110.2
C7—C6—H6B	109.5	O2—C20—C29	121.30 (16)
C5—C6—H6B	109.5	O2—C20—C19	122.69 (16)
H6A—C6—H6B	108.1	C29—C20—C19	115.98 (15)
C6—C7—C8	114.19 (13)	C22—C21—C19	106.17 (14)
C6—C7—H7A	108.7	C22—C21—H21A	110.5
C8—C7—H7A	108.7	C19—C21—H21A	110.5
C6—C7—H7B	108.7	C22—C21—H21B	110.5
C8—C7—H7B	108.7	C19—C21—H21B	110.5
H7A—C7—H7B	107.6	H21A—C21—H21B	108.7
C7—C8—C25	107.74 (13)	C21—C22—C17	103.90 (14)
C7—C8—C9	108.85 (12)	C21—C22—H22A	111.0
C25—C8—C9	111.53 (13)	C17—C22—H22A	111.0
C7—C8—C14	110.34 (12)	C21—C22—H22B	111.0
C25—C8—C14	110.66 (12)	C17—C22—H22B	111.0
C9—C8—C14	107.72 (12)	H22A—C22—H22B	109.0
C11—C9—C10	113.73 (13)	C4—C23—H23A	109.5
C11—C9—C8	111.70 (12)	C4—C23—H23B	109.5
C10—C9—C8	116.58 (13)	H23A—C23—H23B	109.5
C11—C9—H9A	104.4	C4—C23—H23C	109.5
C10—C9—H9A	104.4	H23A—C23—H23C	109.5
C8—C9—H9A	104.4	H23B—C23—H23C	109.5
C24—C10—C5	112.04 (13)	C10—C24—H24A	109.5
C24—C10—C1	103.90 (13)	C10—C24—H24B	109.5
C5—C10—C1	109.78 (12)	H24A—C24—H24B	109.5
C24—C10—C9	114.32 (12)	C10—C24—H24C	109.5
C5—C10—C9	106.93 (13)	H24A—C24—H24C	109.5
C1—C10—C9	109.83 (13)	H24B—C24—H24C	109.5
C12—C11—C9	111.78 (14)	C8—C25—H25A	109.5
C12—C11—H11A	109.3	C8—C25—H25B	109.5
C9—C11—H11A	109.3	H25A—C25—H25B	109.5
C12—C11—H11B	109.3	C8—C25—H25C	109.5
C9—C11—H11B	109.3	H25A—C25—H25C	109.5
H11A—C11—H11B	107.9	H25B—C25—H25C	109.5
C11—C12—C13	111.15 (13)	C14—C26—H26A	109.5
C11—C12—H12A	109.4	C14—C26—H26B	109.5
C13—C12—H12A	109.4	H26A—C26—H26B	109.5
C11—C12—H12B	109.4	C14—C26—H26C	109.5
C13—C12—H12B	109.4	H26A—C26—H26C	109.5
H12A—C12—H12B	108.0	H26B—C26—H26C	109.5

C18—C13—C12	112.46 (13)	O3—C27—O4	123.00 (15)
C18—C13—C14	110.86 (13)	O3—C27—C17	127.48 (16)
C12—C13—C14	111.76 (12)	O4—C27—C17	109.38 (15)
C18—C13—H13A	107.1	O4—C28—H28A	109.5
C12—C13—H13A	107.1	O4—C28—H28B	109.5
C14—C13—H13A	107.1	H28A—C28—H28B	109.5
C26—C14—C13	111.35 (12)	O4—C28—H28C	109.5
C26—C14—C15	105.92 (13)	H28A—C28—H28C	109.5
C13—C14—C15	108.43 (12)	H28B—C28—H28C	109.5
C26—C14—C8	110.92 (12)	C20—C29—H29A	109.5
C13—C14—C8	108.25 (12)	C20—C29—H29B	109.5
C15—C14—C8	111.97 (12)	H29A—C29—H29B	109.5
C16—C15—C14	113.91 (13)	C20—C29—H29C	109.5
C16—C15—H15A	108.8	H29A—C29—H29C	109.5
C14—C15—H15A	108.8	H29B—C29—H29C	109.5
C16—C15—H15B	108.8		
C10—C1—C2—C3	−174.50 (15)	C7—C8—C14—C13	177.66 (12)
O1—C4—C5—C6	55.9 (2)	C25—C8—C14—C13	−63.19 (15)
C23—C4—C5—C6	−120.78 (17)	C9—C8—C14—C13	58.97 (14)
O1—C4—C5—C10	−70.3 (2)	C7—C8—C14—C15	−62.86 (15)
C23—C4—C5—C10	113.01 (17)	C25—C8—C14—C15	56.28 (17)
C4—C5—C6—C7	170.99 (13)	C9—C8—C14—C15	178.45 (12)
C10—C5—C6—C7	−61.22 (17)	C26—C14—C15—C16	65.12 (17)
C5—C6—C7—C8	57.12 (17)	C13—C14—C15—C16	−54.48 (17)
C6—C7—C8—C25	71.70 (16)	C8—C14—C15—C16	−173.85 (13)
C6—C7—C8—C9	−49.39 (17)	C14—C15—C16—C17	58.37 (18)
C6—C7—C8—C14	−167.39 (12)	C15—C16—C17—C27	66.66 (17)
C7—C8—C9—C11	−178.04 (13)	C15—C16—C17—C18	−57.24 (18)
C25—C8—C9—C11	63.23 (17)	C15—C16—C17—C22	−170.80 (13)
C14—C8—C9—C11	−58.39 (16)	C12—C13—C18—C19	51.31 (19)
C7—C8—C9—C10	48.85 (17)	C14—C13—C18—C19	177.25 (13)
C25—C8—C9—C10	−69.88 (17)	C12—C13—C18—C17	176.86 (14)
C14—C8—C9—C10	168.49 (12)	C14—C13—C18—C17	−57.21 (17)
C4—C5—C10—C24	56.14 (18)	C16—C17—C18—C13	59.01 (18)
C6—C5—C10—C24	−68.69 (17)	C27—C17—C18—C13	−63.27 (18)
C4—C5—C10—C1	−58.76 (17)	C22—C17—C18—C13	−175.40 (13)
C6—C5—C10—C1	176.41 (13)	C16—C17—C18—C19	−168.54 (14)
C4—C5—C10—C9	−177.86 (13)	C27—C17—C18—C19	69.18 (17)
C6—C5—C10—C9	57.31 (16)	C22—C17—C18—C19	−42.95 (16)
C2—C1—C10—C24	172.80 (14)	C13—C18—C19—C20	−86.52 (18)
C2—C1—C10—C5	−67.21 (18)	C17—C18—C19—C20	144.07 (15)
C2—C1—C10—C9	50.09 (18)	C13—C18—C19—C21	155.79 (15)
C11—C9—C10—C24	−60.36 (18)	C17—C18—C19—C21	26.38 (17)
C8—C9—C10—C24	71.84 (17)	C18—C19—C20—O2	−30.7 (2)
C11—C9—C10—C5	175.03 (13)	C21—C19—C20—O2	84.3 (2)
C8—C9—C10—C5	−52.78 (16)	C18—C19—C20—C29	151.45 (15)
C11—C9—C10—C1	55.96 (17)	C21—C19—C20—C29	−93.53 (18)
C8—C9—C10—C1	−171.85 (13)	C20—C19—C21—C22	−120.36 (16)
C10—C9—C11—C12	−169.10 (13)	C18—C19—C21—C22	0.73 (19)

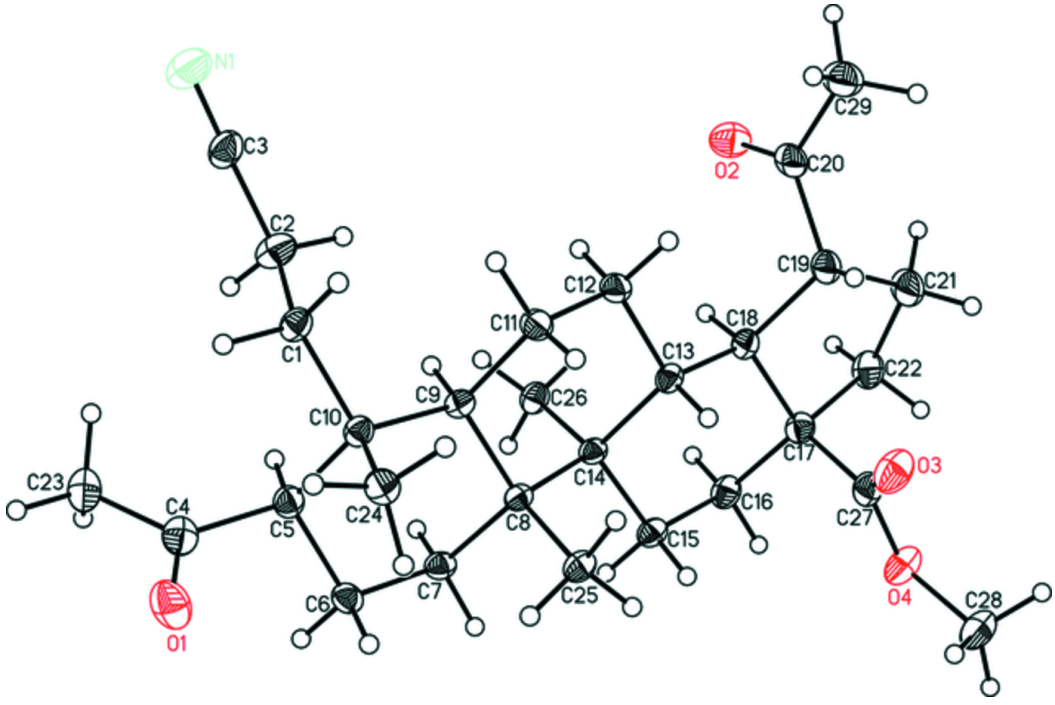
C8—C9—C11—C12	56.39 (17)	C19—C21—C22—C17	−27.28 (19)
C9—C11—C12—C13	−54.56 (18)	C16—C17—C22—C21	160.51 (15)
C11—C12—C13—C18	−177.28 (13)	C27—C17—C22—C21	−74.81 (17)
C11—C12—C13—C14	57.27 (18)	C18—C17—C22—C21	42.66 (17)
C18—C13—C14—C26	−64.09 (16)	C28—O4—C27—O3	−1.2 (3)
C12—C13—C14—C26	62.23 (17)	C28—O4—C27—C17	174.75 (14)
C18—C13—C14—C15	52.06 (16)	C16—C17—C27—O3	−132.21 (19)
C12—C13—C14—C15	178.38 (13)	C18—C17—C27—O3	−11.0 (3)
C18—C13—C14—C8	173.74 (12)	C22—C17—C27—O3	97.7 (2)
C12—C13—C14—C8	−59.94 (15)	C16—C17—C27—O4	52.10 (18)
C7—C8—C14—C26	55.22 (16)	C18—C17—C27—O4	173.35 (14)
C25—C8—C14—C26	174.37 (13)	C22—C17—C27—O4	−78.00 (17)
C9—C8—C14—C26	−63.47 (15)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C28—H28A···O1^{i}	0.98	2.60	3.178 (2)	118
C28—H28B···O1^{ii}	0.98	2.59	3.272 (3)	127

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

Fig. 1


N1
C3
C2
C1
C10
C9
C11
C12
C26
C13
C18
C19
C29
O2
C20
C21
C22
C17
O3
C27
O4
C28
C14
C16
C15
C25
C8
C24
C7
C6
C5
C4
C23
O1