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13-Methyl-3-oxo-2,3,6,7,8,9,10,11,-12,13,14,15,16,17-tetradecahydro-1*H*-cyclopenta[*a*]phenanthren-17-yl 4-nitrobenzoate

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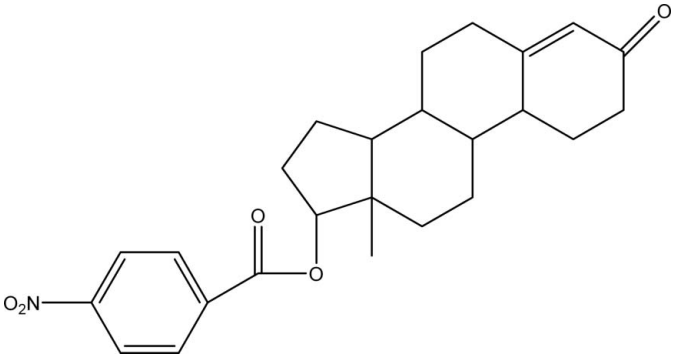
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.037; wR factor = 0.099; data-to-parameter ratio = 7.9.

The title compound, $C_{25}H_{29}NO_5$, is built up from three six-membered and one five-membered fused rings. The two six-membered rings of hexamethylene have chair conformations.

Related literature

For related literature, see: Alvarez-Ginarte *et al.* (2005); Cremer & Pople (1975).



Experimental

Crystal data

$C_{25}H_{29}NO_5$
M_r = 423.49
Orthorhombic, $P2_12_12_1$
a = 10.8866 (6) Å
b = 11.5321 (6) Å
c = 17.6671 (9) Å
V = 2218.0 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 298 (2) K
0.32 × 0.21 × 0.12 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.964, T_{max} = 0.990
25354 measured reflections
2225 independent reflections
1757 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
3 standard reflections
frequency: 60 min
intensity decay: 0.3%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.099
S = 1.03
2225 reflections
282 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Version 1.05; Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SG2168).

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supplementary materials

Acta Cryst. (2007). E**63**, o3022 [**doi:10.1107/S1600536807025020**]

13-Methyl-3-oxo-2,3,6,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1*H*-cyclopenta[*a*]phenanthren-17-yl 4-nitrobenzoate

Y.-Y. Ye

Comment

Testosterone derivatives exhibit a high level of biological activity and have been widely used as hormone treatments (Alvarez-Ginarte *et al.*, 2005). 13-Methyl-3-oxo-2,3,6,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro- 1*H*-cyclopenta[*a*]phenanthren-17-yl 4-nitrobenzoate, (I), was obtained from the reaction of 4-nitrobenzoyl chloride and 17-hydroxy-13-methyl- 1,7,8,10,11,12,13,15,16,17-decahydro-2*H*-cyclopenta[*a*]phenanthren- 3(6*H*,9*H*,14*H*)-one, as colorless crystals suitable for X-ray crystallographic analysis.

The molecular structure of (I) is built up from three six-membered and one five-membered fused rings. (Fig. 1). The C11—C14/C7/C6 and C3—C6/C14/C15 rings have chair conformations (Cremer & Pople, 1975). The dihedral angles between the C6/C7/C12/C13 and C4/C5/C14/C15 planes is 1.91 (23) ^{o}. Atoms C10, C19, C20, C21, C22, C23, C24, C25, O2, O5 and N1 are coplanar to within 0.0848 Å, and atoms O3 and O4 deviate from this plane by −0.2783 (40) and −0.1457 (48) Å, respectively.

Experimental

17-hydroxy-13-methyl-1,7,8,10,11,12,13,15,16,17-decahydro-2*H*- cyclopenta[*a*]phenanthren-3(6*H*,9*H*,14*H*)-one (2.74 g, 10 mmol) and pyridine (20 mmol) were added to CH_2Cl_2 (20 ml) with stirring. 4-nitrobenzoyl chloride (1.86 g, 10 mmol) in CH_2Cl_2 (10 ml) was added dropwise over 15 minutes. The mixture was stirred at room temperature for 3 h and filtered. The filtrate was concentrated *in vacuo*. The residue was purified by flash chromatography on silica gel, eluting with petroleum ether (303–333 *K*)-diethyl ether, to give the product as a pale-yellow solid (1.28 g, 30.2%). A solution of the compound in ethanol was concentrated gradually at room temperature to afford colourless prisms (m.p. 472–475 K).

Refinement

H atoms were added at calculated positions and refined using a riding model. H atoms were given isotropic displacement parameters equal to 1.2(or 1.5 for methyl H atoms) times the equivalent isotropic displacement parameters of their parent atoms, with C—H distances of 0.96 (C_{methyl}), 0.97 ($C_{methylene}$), 0.98 ($C_{methine}$) and 0.93 Å ($C_{aromatic}$). In the absence of significant anomalous dispersion effects, Friedel pairs were averaged. The absolute configuration was assigned arbitrarily.

Figures

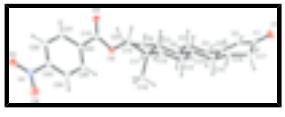

Fig. 1. The structure of (I), shown with 30% probability displacement ellipsoids.

13-Methyl-3-oxo-2,3,6,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro- 1*H*-cyclopenta[*a*]phenanthren-17-yl 4-nitrobenzoate

Crystal data

$C_{25}H_{29}NO_5$	F_{000} = 904
M_r = 423.49	D_x = 1.268 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 25 reflections
a = 10.8866 (6) Å	θ = 10.0–14.8º
b = 11.5321 (6) Å	μ = 0.09 mm^{-1}
c = 17.6671 (9) Å	T = 298 (2) K
V = 2218.0 (2) Å^3	Prism, colourless
Z = 4	0.32 × 0.21 × 0.12 mm

Data collection

Enraf-Nonius CAD-4 diffractometer	R_{int} = 0.042
Radiation source: fine-focus sealed tube	θ_{max} = 25.0º
Monochromator: graphite	θ_{min} = 2.1º
T = 298(2) K	h = −12→12
ω/2–θ scans	k = −13→11
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	l = −21→21
T_{min} = 0.964, T_{max} = 0.990	3 standard reflections
25354 measured reflections	every 60 min
2225 independent reflections	intensity decay: 0.3%
1757 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0431P)^2 + 0.439P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.037	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.099	$\Delta\rho_{max}$ = 0.21 e Å^{-3}
S = 1.03	$\Delta\rho_{min}$ = −0.13 e Å^{-3}
2225 reflections	Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
282 parameters	Extinction coefficient: 0.0045 (9)
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983)
Secondary atom site location: difference Fourier map	Flack parameter: ?
Hydrogen site location: inferred from neighbouring sites	

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	0.5768 (3)	0.6312 (3)	0.42364 (14)	0.1174 (10)
O2	0.4737 (2)	0.51962 (18)	1.01883 (11)	0.0694 (6)
O3	0.5297 (3)	0.6795 (2)	1.08166 (12)	0.0873 (7)
O4	0.6884 (3)	0.1759 (3)	1.30309 (17)	0.1259 (12)
O5	0.7028 (3)	0.3232 (3)	1.37676 (16)	0.1229 (12)
N1	0.6778 (3)	0.2788 (3)	1.31684 (18)	0.0888 (9)
C1	0.5414 (4)	0.5881 (3)	0.48331 (19)	0.0869 (10)
C2	0.4313 (3)	0.6266 (3)	0.52155 (16)	0.0694 (8)
H2	0.3846	0.6845	0.4989	0.083*
C3	0.3929 (3)	0.5836 (3)	0.58751 (15)	0.0605 (7)
C4	0.2712 (3)	0.6168 (3)	0.62039 (17)	0.0730 (9)
H4A	0.2157	0.5512	0.6172	0.088*
H4B	0.2364	0.6797	0.5910	0.088*
C5	0.2828 (3)	0.6547 (3)	0.70291 (16)	0.0671 (8)
H5A	0.3268	0.7278	0.7052	0.081*
H5B	0.2014	0.6670	0.7238	0.081*
C6	0.3497 (3)	0.5657 (2)	0.75022 (15)	0.0552 (7)
H6	0.3006	0.4945	0.7509	0.066*
C7	0.3685 (3)	0.6050 (3)	0.83138 (15)	0.0590 (7)
H7	0.4229	0.6725	0.8284	0.071*
C8	0.2595 (3)	0.6435 (4)	0.87880 (18)	0.0820 (10)
H8A	0.1906	0.5910	0.8726	0.098*
H8B	0.2337	0.7213	0.8651	0.098*
C9	0.3092 (3)	0.6400 (3)	0.96115 (18)	0.0824 (10)
H9A	0.3118	0.7172	0.9828	0.099*
H9B	0.2580	0.5911	0.9928	0.099*
C10	0.4384 (3)	0.5895 (3)	0.95376 (15)	0.0671 (8)
H10	0.4975	0.6529	0.9475	0.081*
C11	0.4355 (3)	0.5171 (2)	0.88165 (15)	0.0576 (7)
C12	0.5594 (3)	0.4892 (3)	0.84696 (16)	0.0742 (9)
H12A	0.5988	0.4284	0.8761	0.089*
H12B	0.6114	0.5575	0.8490	0.089*
C13	0.5457 (3)	0.4499 (3)	0.76461 (16)	0.0727 (9)

H13A	0.6266	0.4379	0.7431	0.087*
H13B	0.5027	0.3762	0.7635	0.087*
C14	0.4756 (3)	0.5373 (3)	0.71588 (15)	0.0588 (7)
H14	0.5233	0.6093	0.7165	0.071*
C15	0.4674 (3)	0.4983 (3)	0.63285 (16)	0.0677 (8)
H15	0.4254	0.4232	0.6314	0.081*
C16	0.5959 (4)	0.4828 (5)	0.5982 (2)	0.1153 (17)
H16A	0.6489	0.5429	0.6184	0.138*
H16B	0.6284	0.4089	0.6152	0.138*
C17	0.6043 (5)	0.4862 (5)	0.5178 (2)	0.1240 (18)
H17A	0.5685	0.4158	0.4973	0.149*
H17B	0.6902	0.4876	0.5034	0.149*
C18	0.3607 (3)	0.4057 (3)	0.89454 (18)	0.0748 (9)
H18A	0.3478	0.3673	0.8470	0.112*
H18B	0.2828	0.4251	0.9166	0.112*
H18C	0.4049	0.3551	0.9280	0.112*
C19	0.5181 (3)	0.5761 (3)	1.07838 (16)	0.0619 (7)
C20	0.5542 (3)	0.4962 (3)	1.14058 (15)	0.0575 (7)
C21	0.5865 (3)	0.5425 (3)	1.21044 (17)	0.0690 (8)
H21	0.5827	0.6223	1.2177	0.083*
C22	0.6237 (3)	0.4725 (3)	1.26874 (18)	0.0730 (9)
H22	0.6444	0.5037	1.3156	0.088*
C23	0.6298 (3)	0.3553 (3)	1.25628 (17)	0.0692 (8)
C24	0.5963 (3)	0.3054 (3)	1.18887 (17)	0.0721 (9)
H24	0.5996	0.2255	1.1823	0.087*
C25	0.5576 (3)	0.3771 (3)	1.13105 (17)	0.0673 (8)
H25	0.5334	0.3450	1.0851	0.081*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.159 (3)	0.108 (2)	0.0856 (17)	0.012 (2)	0.0421 (18)	0.0313 (16)
O2	0.0882 (15)	0.0667 (13)	0.0533 (11)	−0.0137 (12)	−0.0126 (11)	−0.0002 (10)
O3	0.1123 (19)	0.0689 (14)	0.0806 (14)	−0.0207 (14)	−0.0277 (14)	−0.0006 (12)
O4	0.163 (3)	0.110 (2)	0.104 (2)	0.058 (2)	0.003 (2)	0.0158 (19)
O5	0.165 (3)	0.125 (2)	0.0785 (17)	−0.045 (2)	−0.0390 (18)	0.0294 (17)
N1	0.088 (2)	0.103 (3)	0.075 (2)	0.001 (2)	−0.0039 (17)	0.0159 (19)
C1	0.107 (3)	0.083 (2)	0.070 (2)	0.003 (2)	0.012 (2)	0.0148 (19)
C2	0.082 (2)	0.0657 (19)	0.0599 (17)	−0.0003 (18)	−0.0080 (16)	0.0069 (16)
C3	0.0682 (18)	0.0609 (17)	0.0522 (16)	−0.0072 (16)	−0.0090 (14)	0.0021 (14)
C4	0.0680 (19)	0.086 (2)	0.0650 (18)	0.0065 (18)	−0.0126 (15)	0.0062 (18)
C5	0.0616 (18)	0.073 (2)	0.0664 (18)	0.0098 (16)	−0.0093 (15)	−0.0032 (16)
C6	0.0535 (15)	0.0537 (16)	0.0583 (16)	−0.0012 (13)	−0.0051 (13)	0.0011 (13)
C7	0.0609 (17)	0.0580 (17)	0.0582 (16)	−0.0039 (14)	−0.0051 (14)	−0.0029 (14)
C8	0.084 (2)	0.093 (2)	0.069 (2)	0.023 (2)	−0.0012 (18)	−0.0100 (19)
C9	0.104 (3)	0.079 (2)	0.0640 (19)	0.017 (2)	0.0001 (19)	−0.0116 (17)
C10	0.084 (2)	0.0626 (18)	0.0547 (16)	−0.0082 (17)	−0.0088 (16)	0.0023 (14)
C11	0.0620 (17)	0.0588 (16)	0.0520 (15)	−0.0055 (14)	−0.0008 (14)	−0.0005 (13)

C12	0.0649 (19)	0.093 (2)	0.0649 (18)	0.0100 (19)	−0.0058 (16)	0.0130 (17)
C13	0.0654 (18)	0.092 (2)	0.0603 (18)	0.0240 (17)	0.0034 (16)	0.0112 (16)
C14	0.0567 (16)	0.0630 (17)	0.0566 (15)	−0.0011 (14)	−0.0036 (14)	0.0069 (14)
C15	0.080 (2)	0.0688 (19)	0.0547 (16)	0.0097 (17)	0.0063 (16)	0.0094 (15)
C16	0.111 (3)	0.164 (4)	0.072 (2)	0.061 (3)	0.027 (2)	0.032 (3)
C17	0.135 (4)	0.141 (4)	0.096 (3)	0.049 (3)	0.042 (3)	0.045 (3)
C18	0.094 (2)	0.0658 (19)	0.0640 (18)	−0.0165 (18)	0.0028 (17)	−0.0028 (16)
C19	0.0582 (17)	0.071 (2)	0.0569 (16)	−0.0101 (16)	−0.0024 (14)	−0.0061 (15)
C20	0.0537 (16)	0.0661 (18)	0.0527 (15)	−0.0036 (14)	−0.0029 (13)	−0.0052 (14)
C21	0.0726 (19)	0.0699 (19)	0.0645 (18)	−0.0074 (16)	−0.0075 (16)	−0.0082 (16)
C22	0.074 (2)	0.085 (2)	0.0596 (18)	−0.0061 (18)	−0.0081 (16)	−0.0034 (17)
C23	0.0625 (18)	0.080 (2)	0.0647 (19)	0.0009 (17)	−0.0020 (15)	0.0075 (17)
C24	0.075 (2)	0.0687 (19)	0.072 (2)	0.0060 (18)	−0.0014 (17)	−0.0054 (17)
C25	0.0710 (19)	0.0702 (19)	0.0605 (17)	−0.0008 (17)	−0.0026 (16)	−0.0087 (16)

Geometric parameters (Å, °)

O1—C1	1.228 (4)	C10—H10	0.9800
O2—C19	1.328 (3)	C11—C12	1.516 (4)
O2—C10	1.455 (3)	C11—C18	1.538 (4)
O3—C19	1.201 (4)	C12—C13	1.531 (4)
O4—N1	1.217 (4)	C12—H12A	0.9700
O5—N1	1.207 (4)	C12—H12B	0.9700
N1—C23	1.482 (4)	C13—C14	1.530 (4)
C1—C2	1.446 (5)	C13—H13A	0.9700
C1—C17	1.490 (6)	C13—H13B	0.9700
C2—C3	1.333 (4)	C14—C15	1.537 (4)
C2—H2	0.9300	C14—H14	0.9800
C3—C4	1.496 (4)	C15—C16	1.538 (5)
C3—C15	1.505 (4)	C15—H15	0.9800
C4—C5	1.527 (4)	C16—C17	1.424 (5)
C4—H4A	0.9700	C16—H16A	0.9700
C4—H4B	0.9700	C16—H16B	0.9700
C5—C6	1.511 (4)	C17—H17A	0.9700
C5—H5A	0.9700	C17—H17B	0.9700
C5—H5B	0.9700	C18—H18A	0.9600
C6—C7	1.518 (4)	C18—H18B	0.9600
C6—C14	1.535 (4)	C18—H18C	0.9600
C6—H6	0.9800	C19—C20	1.487 (4)
C7—C8	1.520 (4)	C20—C25	1.384 (4)
C7—C11	1.532 (4)	C20—C21	1.390 (4)
C7—H7	0.9800	C21—C22	1.370 (4)
C8—C9	1.553 (5)	C21—H21	0.9300
C8—H8A	0.9700	C22—C23	1.371 (5)
C8—H8B	0.9700	C22—H22	0.9300
C9—C10	1.529 (5)	C23—C24	1.372 (4)
C9—H9A	0.9700	C24—C25	1.380 (4)
C9—H9B	0.9700	C24—H24	0.9300
C10—C11	1.524 (4)	C25—H25	0.9300

C19—O2—C10	116.8 (2)	C11—C12—H12A	109.4
O5—N1—O4	124.6 (3)	C13—C12—H12A	109.4
O5—N1—C23	117.4 (3)	C11—C12—H12B	109.4
O4—N1—C23	118.0 (3)	C13—C12—H12B	109.4
O1—C1—C2	122.5 (4)	H12A—C12—H12B	108.0
O1—C1—C17	121.8 (4)	C14—C13—C12	112.8 (3)
C2—C1—C17	115.6 (3)	C14—C13—H13A	109.0
C3—C2—C1	123.6 (3)	C12—C13—H13A	109.0
C3—C2—H2	118.2	C14—C13—H13B	109.0
C1—C2—H2	118.2	C12—C13—H13B	109.0
C2—C3—C4	121.4 (3)	H13A—C13—H13B	107.8
C2—C3—C15	122.6 (3)	C13—C14—C6	111.3 (2)
C4—C3—C15	116.0 (3)	C13—C14—C15	111.9 (2)
C3—C4—C5	111.8 (3)	C6—C14—C15	112.8 (2)
C3—C4—H4A	109.3	C13—C14—H14	106.8
C5—C4—H4A	109.3	C6—C14—H14	106.8
C3—C4—H4B	109.3	C15—C14—H14	106.8
C5—C4—H4B	109.3	C3—C15—C14	110.4 (2)
H4A—C4—H4B	107.9	C3—C15—C16	110.8 (3)
C6—C5—C4	111.9 (3)	C14—C15—C16	111.2 (3)
C6—C5—H5A	109.2	C3—C15—H15	108.1
C4—C5—H5A	109.2	C14—C15—H15	108.1
C6—C5—H5B	109.2	C16—C15—H15	108.1
C4—C5—H5B	109.2	C17—C16—C15	116.9 (4)
H5A—C5—H5B	107.9	C17—C16—H16A	108.1
C5—C6—C7	112.6 (2)	C15—C16—H16A	108.1
C5—C6—C14	110.9 (2)	C17—C16—H16B	108.1
C7—C6—C14	108.4 (2)	C15—C16—H16B	108.1
C5—C6—H6	108.2	H16A—C16—H16B	107.3
C7—C6—H6	108.2	C16—C17—C1	113.6 (4)
C14—C6—H6	108.2	C16—C17—H17A	108.9
C6—C7—C8	120.2 (2)	C1—C17—H17A	108.9
C6—C7—C11	114.5 (2)	C16—C17—H17B	108.9
C8—C7—C11	104.2 (2)	C1—C17—H17B	108.9
C6—C7—H7	105.6	H17A—C17—H17B	107.7
C8—C7—H7	105.6	C11—C18—H18A	109.5
C11—C7—H7	105.6	C11—C18—H18B	109.5
C7—C8—C9	103.7 (3)	H18A—C18—H18B	109.5
C7—C8—H8A	111.0	C11—C18—H18C	109.5
C9—C8—H8A	111.0	H18A—C18—H18C	109.5
C7—C8—H8B	111.0	H18B—C18—H18C	109.5
C9—C8—H8B	111.0	O3—C19—O2	124.3 (3)
H8A—C8—H8B	109.0	O3—C19—C20	123.5 (3)
C10—C9—C8	104.5 (3)	O2—C19—C20	112.2 (3)
C10—C9—H9A	110.8	C25—C20—C21	118.8 (3)
C8—C9—H9A	110.8	C25—C20—C19	122.1 (3)
C10—C9—H9B	110.8	C21—C20—C19	119.0 (3)
C8—C9—H9B	110.8	C22—C21—C20	121.1 (3)
H9A—C9—H9B	108.9	C22—C21—H21	119.5

O2—C10—C11	111.3 (2)	C20—C21—H21	119.5
O2—C10—C9	112.7 (3)	C21—C22—C23	118.3 (3)
C11—C10—C9	105.1 (3)	C21—C22—H22	120.8
O2—C10—H10	109.2	C23—C22—H22	120.8
C11—C10—H10	109.2	C22—C23—C24	122.7 (3)
C9—C10—H10	109.2	C22—C23—N1	119.2 (3)
C12—C11—C10	115.8 (3)	C24—C23—N1	118.1 (3)
C12—C11—C7	109.2 (2)	C23—C24—C25	118.2 (3)
C10—C11—C7	97.6 (2)	C23—C24—H24	120.9
C12—C11—C18	110.7 (3)	C25—C24—H24	120.9
C10—C11—C18	110.2 (2)	C24—C25—C20	120.8 (3)
C7—C11—C18	112.7 (2)	C24—C25—H25	119.6
C11—C12—C13	111.1 (3)	C20—C25—H25	119.6
O1—C1—C2—C3	−177.9 (4)	C5—C6—C14—C13	178.3 (2)
C17—C1—C2—C3	6.7 (5)	C7—C6—C14—C13	54.2 (3)
C1—C2—C3—C4	−173.0 (3)	C5—C6—C14—C15	−54.8 (3)
C1—C2—C3—C15	6.2 (5)	C7—C6—C14—C15	−179.0 (2)
C2—C3—C4—C5	−129.9 (3)	C2—C3—C15—C14	131.2 (3)
C15—C3—C4—C5	50.8 (4)	C4—C3—C15—C14	−49.6 (4)
C3—C4—C5—C6	−52.5 (4)	C2—C3—C15—C16	7.6 (4)
C4—C5—C6—C7	176.7 (2)	C4—C3—C15—C16	−173.2 (3)
C4—C5—C6—C14	54.9 (3)	C13—C14—C15—C3	177.3 (3)
C5—C6—C7—C8	54.5 (4)	C6—C14—C15—C3	50.9 (3)
C14—C6—C7—C8	177.6 (3)	C13—C14—C15—C16	−59.3 (4)
C5—C6—C7—C11	179.7 (2)	C6—C14—C15—C16	174.2 (3)
C14—C6—C7—C11	−57.2 (3)	C3—C15—C16—C17	−36.0 (6)
C6—C7—C8—C9	163.5 (3)	C14—C15—C16—C17	−159.1 (4)
C11—C7—C8—C9	33.6 (3)	C15—C16—C17—C1	49.8 (7)
C7—C8—C9—C10	−4.8 (4)	O1—C1—C17—C16	150.3 (5)
C19—O2—C10—C11	158.7 (3)	C2—C1—C17—C16	−34.2 (6)
C19—O2—C10—C9	−83.5 (3)	C10—O2—C19—O3	0.5 (5)
C8—C9—C10—O2	−147.1 (3)	C10—O2—C19—C20	−178.4 (2)
C8—C9—C10—C11	−25.7 (4)	O3—C19—C20—C25	−169.9 (3)
O2—C10—C11—C12	−76.9 (3)	O2—C19—C20—C25	9.1 (4)
C9—C10—C11—C12	160.8 (3)	O3—C19—C20—C21	9.7 (5)
O2—C10—C11—C7	167.4 (2)	O2—C19—C20—C21	−171.3 (3)
C9—C10—C11—C7	45.1 (3)	C25—C20—C21—C22	1.5 (5)
O2—C10—C11—C18	49.8 (3)	C19—C20—C21—C22	−178.1 (3)
C9—C10—C11—C18	−72.5 (3)	C20—C21—C22—C23	0.7 (5)
C6—C7—C11—C12	57.5 (3)	C21—C22—C23—C24	−2.2 (5)
C8—C7—C11—C12	−169.2 (3)	C21—C22—C23—N1	175.9 (3)
C6—C7—C11—C10	178.3 (2)	O5—N1—C23—C22	4.4 (5)
C8—C7—C11—C10	−48.4 (3)	O4—N1—C23—C22	−176.2 (4)
C6—C7—C11—C18	−66.0 (3)	O5—N1—C23—C24	−177.4 (3)
C8—C7—C11—C18	67.3 (3)	O4—N1—C23—C24	2.0 (5)
C10—C11—C12—C13	−162.8 (3)	C22—C23—C24—C25	1.4 (5)
C7—C11—C12—C13	−53.8 (3)	N1—C23—C24—C25	−176.7 (3)
C18—C11—C12—C13	70.9 (3)	C23—C24—C25—C20	0.8 (5)
C11—C12—C13—C14	54.8 (4)	C21—C20—C25—C24	−2.2 (5)

C12—C13—C14—C6	−55.0 (3)	C19—C20—C25—C24	177.3 (3)
C12—C13—C14—C15	177.7 (3)		

Fig. 1