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3-Oxoandrost-4-en-17β-yl 1*H*-imidazole-1-carboxylate

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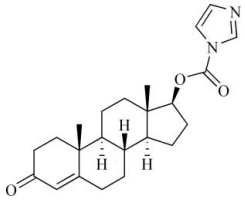
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.006 Å; R factor = 0.059; wR factor = 0.185; data-to-parameter ratio = 10.5.

The title compound, $C_{23}H_{30}N_2O_3$, prepared in a one-step reaction from the commercially available 1,1′-carbonyldiimidazole (CDI) and testosterone, crystallizes with two independent molecules in the asymmetric unit. The imidazole carbonyloxy group is in an equatorial position. The molecules are joined in layers parallel to the *ac* plane *via* C—H···O intermolecular interactions.

Related literature

For physiological properties and industrial uses of carbamates, see Adams & Baron (1965). For carbamate use in organic synthesis as a protecting group of the amine functionality, see Greene & Wuts (1991). For carbamate synthesis using CDI, see: Staab (1962); Totleben *et al.* (1997); Rannard & Davis (2000); D'Addona & Bochet (2001); Fischer (2002); Tang *et al.* (2004); Mulvihill *et al.* (2004); Herbez & Fischer (2005). For related structures, see: Roberts *et al.* (1973). For related literature, see: Cremer & Pople (1975).



Experimental

Crystal data

$C_{23}H_{30}N_2O_3$
M_r = 382.49
Monoclinic, $P2_1$
a = 7.4140 (4) Å
b = 21.259 (1) Å
c = 13.0036 (7) Å
β = 92.595 (3)°
V = 2047.4 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 293 (2) K
0.28 × 0.20 × 0.15 mm

Data collection

Bruker APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2000)
T_{min} = 0.984, T_{max} = 0.993
35029 measured reflections
5369 independent reflections
3477 reflections with $I > 2\sigma(I)$
R_{int} = 0.056

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.185
S = 1.06
5369 reflections
509 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C16—H16A···O1^{i}	0.97	2.54	3.455 (6)	157
C39—H39A···O4^{i}	0.97	2.52	3.460 (6)	162
C44—H44···N2^{ii}	0.93	2.58	3.414 (6)	149

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

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZL2077).

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supplementary materials

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3-Oxoandrost-4-en-17β-yl 1*H*-imidazole-1-carboxylate

M. Ramos Silva, A. Matos Beja, V. M. Moreira, R. C. Santos and J. A. R. Salvador

Comment

The title compound (Fig. 1) crystallizes with two independent molecules in the unit cell. The molecules exhibit very similar conformations with all ring junctions fused *trans*. The overall r.m.s fit of these molecules is 0.112 Å. When viewed down the C17—O2 bond the molecules exhibit a staggered conformation with C13 and C20 anti to each other and C16 *gauche* to C20. The C17 imidazole carbonyloxy group is in an equatorial position with an angle to the Cremer & Pople normal (Cremer & Pople, 1975) of 62.7 (3)° [and 63.1 (2)° for molecule 2]. Ring A (See the Fig.1 for a definition of the ring labels) adopts a conformation between sofa and half-chair with C&P puckering parameters Q = 0.461 (4) Å, θ = 55.1 (7)°, φ = 14.8 (8)° [for molecule 1] and Q = 0.456 (5) Å, θ = 55.1 (6)°, φ = 15.1 (7)° [for molecule 2]. Rings B and C have distorted chair conformations. The 5-membered ring adopts a envelope conformation (on C13) with C&P parameters Q2 = 0.473 (4) Å, φ2 = 186.7 (6)° [for molecule 1] Q2 = 0.479 (4) Å, φ2 = 186.6 (5)° [for molecule 2]. The C18 and C19 methyl groups bond to the ring skeleton in axial positions. The unsubstituted testosterone crystallizes with two independent molecules in the unit cell (Roberts *et al.*, 1973). The two independent molecules show similar features with the molecules of the title compound, even in the conformation of the ring D, that adopts a envelope conformation on C13 for one of the molecules and is twisted on C13—C14 for the other symmetry independent molecule. No classic hydrogen bonds can be found in this compound due to the lack of conventional donors. A search for weak C—H···O interactions shows three close contacts (Table 1) that join the molecules in layers parallel to the *ac* plane (Fig. 2).

Experimental

All reagents were obtained from Sigma–Aldrich Co. Acetonitrile was dried and purified prior to use according to standard procedures. A solution of testosterone (150 mg, 0.52 mmol) and CDI (110 mg, 0.68 mmol) was refluxed in anhydrous acetonitrile (5 ml). After 5 h the reaction was complete (TLC control). Water (20 ml) was added to the mixture and the resulting precipitate was dissolved in ethyl ether (50 ml). The aqueous phase was extracted twice with diethyl ether (2 × 50 ml). The organic phase was then washed with water (50 ml), brine (50 ml), dried with anhydrous Na_2SO_4, filtered, evaporated to dryness to afford the title compound (164 mg, 83%): mp (acetone/hexane) 166–168 °C; IR 1672, 1758 cm^{-1}; ^{1}H-NMR ($CDCl_3$, 600 MHz) δ 0.91 (s, 3H, 18-CH_3), 1.17 (s, 3H, 19-CH_3), 0.8–2.5 (several multiplets, 19H, all aliphatic C—H and CH_2), 4.80 (m, 1H, 17α-H), 5.70 (brs, 1H, 4-H), 7.03 (brs, 1H, aromatic-H), 7.38 (brs, 1H, aromatic-H), 8.09 (brs, 1H, aromatic-H); ^{13}C-NMR ($CDCl_3$, 150.8 MHz) δ 12.1, 17.2, 20.3, 23.2, 27.2, 31.2, 32.5, 33.7, 35.2, 35.5, 36.4, 38.4, 42.7, 49.8, 53.4 (all aliphatic C—H, CH_2 and CH_3), 86.5 (C17), 116.9 (aromatic-C), 123.9 (C4), 130.4 (aromatic-C), 136.8 (aromatic-C), 148.4 (OCO), 170.3 (C5), 199.1 (C3). EI—MS m/*z* (%): 382.1 (54) M^+, 271.3 (48), 253.3 (96), 147.3 (84), 133.2 (42), 105.2 (76), 91.2 (82), 69.1 (100). Anal. calcd. for $C_{23}H_{30}N_2O_3$: C 72.22, H 7.91, N 7.32, found: C 72.00, H 8.00, N 7.50.

Refinement

The methyl groups were idealized based on difference electron density synthesis, then refined as a rigid groups allowed to rotate but not tip with C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C). Other H atoms were placed in calculated positions with C—H = 0.93 (aromatic, alkene), 0.97 (methylene) and 0.98 Å (methine), and refined in riding mode with U_{iso}(H) = 1.2U_{eq}(C). The absolute structure could not be determined from the X-ray analysis, due to the lack of significant anomalous dispersion at the Mo *K*α wavelength. The absolute configuration of the parent molecule was chosen to fix the enantiomer. Friedel pairs were merged.

Figures

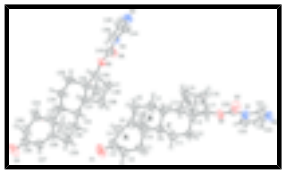

Fig. 1. *ORTEPII* (Johnson, 1976) plot of the title compound. Displacement ellipsoids are drawn at the 50% level.

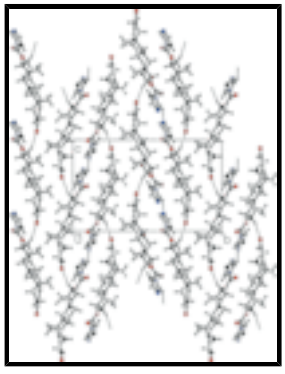

Fig. 2. Packing diagram of the title compound. C—H···O interactions are shown as dashed lines.

3-Oxoandrost-4-en-17β-yl 1*H*-imidazole-1-carboxylate

Crystal data

$C_{23}H_{30}N_2O_3$	F_{000} = 824
M_r = 382.49	D_x = 1.241 Mg m^{-3}
Monoclinic, $P2_1$	Mo *K*α radiation λ = 0.71073 Å
a = 7.4140 (4) Å	Cell parameters from 7710 reflections
b = 21.259 (1) Å	θ = 2.5–21.8º
c = 13.0036 (7) Å	μ = 0.08 mm^{-1}
β = 92.595 (3)º	*T* = 293 (2) K
V = 2047.4 (2) Å^3	Prism, colourless
Z = 4	0.28 × 0.20 × 0.15 mm

Data collection

Bruker APEX CCD area-detector	5369 independent reflections

diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

T = 293(2) K

φ and ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 2000)

T_{min} = 0.984, T_{max} = 0.993

35029 measured reflections

3477 reflections with $I > 2\sigma(I)$

R_{int} = 0.056

θ_{max} = 28.8º

θ_{min} = 1.8º

$h = -10 \rightarrow 10$

$k = -28 \rightarrow 28$

$l = -15 \rightarrow 17$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)]$ = 0.059

$wR(F^2)$ = 0.185

S = 1.06

5369 reflections

509 parameters

1 restraint

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

$w = 1/[\sigma^2(F_o^2) + (0.1115P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max}$ = 0.31 e Å^{-3}

$\Delta\rho_{min}$ = −0.24 e Å^{-3}

Extinction correction: none

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 $F^2 > \sigma(F^2)$ 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}
O4	0.3608 (6)	0.4263 (2)	1.4092 (3)	0.0954 (13)
O5	0.6049 (3)	0.52488 (14)	0.61113 (19)	0.0472 (6)
O6	0.4473 (4)	0.59321 (18)	0.5081 (2)	0.0685 (9)
N3	0.7188 (4)	0.55449 (17)	0.4623 (2)	0.0461 (7)
N4	0.8782 (6)	0.5714 (2)	0.3252 (3)	0.0723 (12)
C24	0.6065 (6)	0.4456 (2)	1.1792 (3)	0.0550 (11)
H24A	0.7298	0.4399	1.1591	0.066*
H24B	0.5831	0.4904	1.1821	0.066*

C25	0.5872 (7)	0.4174 (3)	1.2862 (3)	0.0663 (13)
H25A	0.6240	0.3736	1.2857	0.080*
H25B	0.6663	0.4396	1.3354	0.080*
C26	0.3972 (7)	0.4218 (2)	1.3191 (3)	0.0639 (12)
C27	0.2576 (6)	0.4165 (2)	1.2379 (3)	0.0570 (11)
H27	0.1381	0.4170	1.2568	0.068*
C28	0.2877 (5)	0.41091 (18)	1.1378 (3)	0.0459 (9)
C29	0.1384 (5)	0.3954 (2)	1.0602 (3)	0.0544 (10)
H29A	0.0236	0.4000	1.0923	0.065*
H29B	0.1498	0.3517	1.0397	0.065*
C30	0.1377 (5)	0.4365 (2)	0.9646 (3)	0.0507 (10)
H30A	0.1041	0.4791	0.9825	0.061*
H30B	0.0485	0.4207	0.9141	0.061*
C31	0.3237 (4)	0.43713 (18)	0.9178 (3)	0.0389 (8)
H31	0.3543	0.3943	0.8967	0.047*
C32	0.4657 (4)	0.45997 (18)	0.9999 (3)	0.0391 (8)
H32	0.4238	0.5009	1.0239	0.047*
C33	0.4773 (5)	0.4157 (2)	1.0971 (3)	0.0433 (8)
C34	0.6539 (5)	0.4712 (2)	0.9566 (3)	0.0485 (10)
H34A	0.7309	0.4920	1.0084	0.058*
H34B	0.7082	0.4310	0.9410	0.058*
C35	0.6425 (5)	0.5118 (2)	0.8585 (3)	0.0477 (9)
H35A	0.6025	0.5538	0.8757	0.057*
H35B	0.7616	0.5153	0.8310	0.057*
C36	0.5129 (4)	0.48383 (17)	0.7771 (3)	0.0372 (7)
C37	0.3256 (5)	0.48013 (18)	0.8246 (3)	0.0399 (8)
H37	0.2958	0.5225	0.8480	0.048*
C38	0.1969 (5)	0.4650 (2)	0.7315 (3)	0.0518 (10)
H38A	0.0763	0.4807	0.7421	0.062*
H38B	0.1906	0.4200	0.7193	0.062*
C39	0.2824 (5)	0.4992 (2)	0.6408 (3)	0.0476 (9)
H39A	0.3030	0.4701	0.5851	0.057*
H39B	0.2042	0.5328	0.6149	0.057*
C40	0.4609 (5)	0.5257 (2)	0.6849 (3)	0.0430 (8)
H40	0.4425	0.5689	0.7086	0.052*
C41	0.5809 (5)	0.4202 (2)	0.7391 (3)	0.0490 (9)
H41A	0.4987	0.4044	0.6861	0.073*
H41B	0.5883	0.3910	0.7954	0.073*
H41C	0.6983	0.4254	0.7120	0.073*
C42	0.5437 (6)	0.3489 (2)	1.0693 (3)	0.0541 (10)
H42A	0.6583	0.3520	1.0382	0.081*
H42B	0.4576	0.3296	1.0219	0.081*
H42C	0.5562	0.3239	1.1307	0.081*
C43	0.5756 (5)	0.5597 (2)	0.5277 (3)	0.0488 (9)
C44	0.7243 (6)	0.5805 (3)	0.3668 (3)	0.0601 (11)
H44	0.6286	0.6025	0.3350	0.072*
C45	0.9769 (7)	0.5355 (3)	0.3962 (4)	0.0729 (14)
H45	1.0931	0.5208	0.3869	0.087*
C46	0.8832 (5)	0.5247 (2)	0.4805 (3)	0.0584 (11)

H46	0.9208	0.5020	0.5388	0.070*
O1	0.1144 (8)	0.2584 (3)	0.9092 (3)	0.1107 (17)
O2	−0.0777 (4)	0.16321 (15)	0.0963 (2)	0.0527 (7)
O3	0.0849 (4)	0.10079 (18)	−0.0054 (2)	0.0704 (10)
N1	−0.2036 (4)	0.12987 (17)	−0.0533 (3)	0.0499 (8)
N2	−0.3770 (6)	0.1086 (3)	−0.1914 (3)	0.0769 (12)
C1	−0.1158 (7)	0.2347 (3)	0.6623 (3)	0.0613 (12)
H1A	−0.2386	0.2377	0.6338	0.074*
H1B	−0.0838	0.1905	0.6662	0.074*
C2	−0.1071 (8)	0.2626 (3)	0.7710 (4)	0.0735 (15)
H2A	−0.1524	0.3054	0.7685	0.088*
H2B	−0.1836	0.2382	0.8147	0.088*
C3	0.0854 (9)	0.2624 (3)	0.8168 (4)	0.0749 (16)
C4	0.2241 (8)	0.2713 (2)	0.7454 (4)	0.0658 (13)
H4	0.3425	0.2732	0.7721	0.079*
C5	0.1980 (6)	0.2772 (2)	0.6434 (3)	0.0521 (10)
C6	0.3493 (6)	0.2959 (2)	0.5765 (3)	0.0571 (11)
H6A	0.4626	0.2928	0.6165	0.068*
H6B	0.3331	0.3395	0.5563	0.068*
C7	0.3611 (5)	0.2562 (2)	0.4808 (3)	0.0540 (10)
H7A	0.4004	0.2141	0.5000	0.065*
H7B	0.4500	0.2741	0.4369	0.065*
C8	0.1783 (5)	0.25260 (18)	0.4214 (3)	0.0407 (8)
H8	0.1423	0.2951	0.3994	0.049*
C9	0.0352 (5)	0.2262 (2)	0.4930 (3)	0.0431 (8)
H9	0.0829	0.1860	0.5190	0.052*
C10	0.0127 (5)	0.2687 (2)	0.5893 (3)	0.0452 (9)
C11	−0.1461 (5)	0.2111 (2)	0.4358 (3)	0.0556 (11)
H11A	−0.2218	0.1882	0.4818	0.067*
H11B	−0.2069	0.2503	0.4178	0.067*
C12	−0.1260 (5)	0.1725 (2)	0.3380 (3)	0.0499 (9)
H12A	−0.0819	0.1308	0.3563	0.060*
H12B	−0.2430	0.1679	0.3024	0.060*
C13	0.0042 (5)	0.20374 (18)	0.2669 (3)	0.0392 (8)
C14	0.1874 (4)	0.21055 (18)	0.3266 (3)	0.0391 (8)
H14	0.2226	0.1685	0.3510	0.047*
C15	0.3172 (5)	0.2288 (2)	0.2433 (3)	0.0532 (10)
H15A	0.4392	0.2151	0.2618	0.064*
H15B	0.3175	0.2739	0.2326	0.064*
C16	0.2416 (6)	0.1938 (2)	0.1453 (3)	0.0520 (10)
H16A	0.2195	0.2231	0.0889	0.062*
H16B	0.3257	0.1619	0.1240	0.062*
C17	0.0648 (5)	0.1638 (2)	0.1769 (3)	0.0453 (9)
H17	0.0883	0.1207	0.2006	0.054*
C18	−0.0707 (6)	0.2660 (2)	0.2266 (3)	0.0537 (10)
H18A	−0.1896	0.2596	0.1960	0.081*
H18B	0.0068	0.2824	0.1758	0.081*
H18C	−0.0767	0.2953	0.2825	0.081*
C19	−0.0630 (7)	0.3341 (2)	0.5599 (4)	0.0604 (12)

H19A	−0.0642	0.3601	0.6202	0.091*
H19B	−0.1837	0.3297	0.5309	0.091*
H19C	0.0119	0.3532	0.5103	0.091*
C20	−0.0498 (6)	0.1293 (2)	0.0131 (3)	0.0489 (9)
C21	−0.2160 (7)	0.1032 (3)	−0.1489 (3)	0.0613 (11)
H21	−0.1204	0.0836	−0.1799	0.074*
C22	−0.4744 (7)	0.1406 (3)	−0.1202 (4)	0.0767 (15)
H22	−0.5958	0.1510	−0.1297	0.092*
C23	−0.3730 (6)	0.1546 (3)	−0.0363 (4)	0.0679 (13)
H23	−0.4081	0.1764	0.0215	0.082*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O4	0.119 (3)	0.119 (4)	0.0497 (19)	0.014 (3)	0.020 (2)	−0.005 (2)
O5	0.0411 (13)	0.0571 (17)	0.0436 (14)	0.0055 (12)	0.0039 (11)	0.0076 (12)
O6	0.064 (2)	0.069 (2)	0.074 (2)	0.0250 (17)	0.0128 (16)	0.0204 (17)
N3	0.0397 (16)	0.055 (2)	0.0429 (17)	0.0004 (15)	0.0004 (12)	0.0064 (15)
N4	0.060 (2)	0.102 (4)	0.056 (2)	−0.013 (2)	0.0086 (18)	0.008 (2)
C24	0.049 (2)	0.070 (3)	0.047 (2)	−0.002 (2)	−0.0018 (17)	0.001 (2)
C25	0.074 (3)	0.077 (3)	0.048 (2)	0.003 (3)	−0.003 (2)	0.003 (2)
C26	0.088 (3)	0.055 (3)	0.050 (2)	0.005 (2)	0.012 (2)	−0.002 (2)
C27	0.062 (3)	0.055 (2)	0.057 (2)	0.004 (2)	0.019 (2)	0.002 (2)
C28	0.044 (2)	0.039 (2)	0.055 (2)	0.0040 (16)	0.0098 (16)	0.0046 (17)
C29	0.039 (2)	0.065 (3)	0.060 (2)	−0.0052 (18)	0.0147 (17)	0.007 (2)
C30	0.0296 (18)	0.066 (3)	0.057 (2)	−0.0014 (17)	0.0044 (16)	0.004 (2)
C31	0.0299 (16)	0.039 (2)	0.048 (2)	−0.0010 (14)	0.0018 (14)	0.0015 (15)
C32	0.0315 (17)	0.040 (2)	0.0452 (19)	−0.0021 (14)	0.0011 (14)	−0.0017 (15)
C33	0.0400 (18)	0.046 (2)	0.0445 (19)	0.0060 (16)	0.0054 (15)	0.0056 (17)
C34	0.0324 (18)	0.062 (3)	0.051 (2)	−0.0120 (17)	−0.0032 (15)	0.0060 (19)
C35	0.0335 (18)	0.055 (2)	0.055 (2)	−0.0145 (16)	0.0010 (15)	0.0067 (19)
C36	0.0322 (17)	0.0354 (18)	0.0438 (19)	0.0016 (14)	0.0008 (13)	0.0000 (15)
C37	0.0293 (16)	0.041 (2)	0.050 (2)	−0.0007 (14)	0.0014 (14)	−0.0028 (16)
C38	0.0347 (18)	0.067 (3)	0.053 (2)	−0.0025 (18)	−0.0029 (16)	0.000 (2)
C39	0.042 (2)	0.053 (2)	0.048 (2)	0.0041 (17)	−0.0025 (15)	0.0004 (17)
C40	0.0391 (18)	0.042 (2)	0.049 (2)	0.0034 (16)	0.0040 (15)	0.0012 (16)
C41	0.047 (2)	0.041 (2)	0.059 (2)	0.0074 (17)	0.0046 (17)	−0.0005 (18)
C42	0.051 (2)	0.054 (3)	0.059 (2)	0.0133 (19)	0.0109 (18)	0.0094 (19)
C43	0.047 (2)	0.054 (2)	0.046 (2)	−0.0035 (18)	0.0027 (16)	0.0040 (19)
C44	0.053 (2)	0.080 (3)	0.047 (2)	−0.002 (2)	0.0009 (18)	0.012 (2)
C45	0.052 (3)	0.096 (4)	0.071 (3)	0.005 (3)	0.012 (2)	0.001 (3)
C46	0.043 (2)	0.075 (3)	0.057 (2)	0.007 (2)	−0.0027 (18)	0.007 (2)
O1	0.158 (5)	0.121 (4)	0.051 (2)	0.007 (3)	−0.008 (2)	0.006 (2)
O2	0.0515 (16)	0.0621 (19)	0.0442 (14)	0.0077 (14)	−0.0022 (11)	−0.0110 (14)
O3	0.068 (2)	0.072 (2)	0.070 (2)	0.0285 (18)	−0.0107 (16)	−0.0182 (17)
N1	0.0468 (18)	0.059 (2)	0.0432 (17)	0.0013 (15)	−0.0005 (14)	−0.0033 (15)
N2	0.076 (3)	0.092 (3)	0.062 (2)	−0.012 (3)	−0.007 (2)	−0.008 (2)
C1	0.058 (3)	0.076 (3)	0.051 (2)	0.003 (2)	0.0150 (19)	−0.001 (2)

C2	0.093 (4)	0.076 (4)	0.053 (3)	0.012 (3)	0.022 (3)	−0.002 (2)
C3	0.127 (5)	0.050 (3)	0.048 (3)	0.010 (3)	0.007 (3)	−0.001 (2)
C4	0.087 (3)	0.050 (3)	0.059 (3)	0.006 (2)	−0.015 (2)	−0.001 (2)
C5	0.061 (2)	0.041 (2)	0.054 (2)	0.0102 (19)	−0.0036 (19)	−0.0081 (18)
C6	0.049 (2)	0.058 (3)	0.064 (3)	−0.0038 (19)	−0.0077 (19)	−0.011 (2)
C7	0.0345 (19)	0.063 (3)	0.064 (3)	−0.0026 (18)	−0.0030 (17)	−0.009 (2)
C8	0.0330 (17)	0.042 (2)	0.047 (2)	−0.0007 (14)	0.0010 (14)	−0.0015 (16)
C9	0.0370 (18)	0.047 (2)	0.045 (2)	0.0025 (15)	0.0056 (15)	0.0010 (16)
C10	0.044 (2)	0.044 (2)	0.048 (2)	0.0032 (16)	0.0026 (16)	−0.0010 (17)
C11	0.0352 (19)	0.079 (3)	0.053 (2)	−0.0157 (19)	0.0100 (17)	−0.006 (2)
C12	0.041 (2)	0.052 (2)	0.056 (2)	−0.0165 (17)	−0.0002 (16)	−0.0011 (19)
C13	0.0355 (17)	0.041 (2)	0.0410 (18)	0.0022 (15)	0.0029 (14)	−0.0013 (15)
C14	0.0320 (17)	0.0361 (19)	0.049 (2)	0.0028 (14)	0.0033 (14)	0.0015 (15)
C15	0.041 (2)	0.064 (3)	0.056 (2)	−0.0024 (18)	0.0108 (17)	−0.001 (2)
C16	0.052 (2)	0.056 (3)	0.049 (2)	0.0015 (19)	0.0109 (17)	−0.0061 (18)
C17	0.049 (2)	0.042 (2)	0.045 (2)	0.0039 (17)	−0.0026 (16)	−0.0019 (16)
C18	0.053 (2)	0.048 (2)	0.060 (2)	0.0117 (18)	0.0021 (19)	0.001 (2)
C19	0.068 (3)	0.052 (3)	0.061 (3)	0.018 (2)	0.005 (2)	−0.003 (2)
C20	0.050 (2)	0.051 (2)	0.045 (2)	0.0051 (18)	−0.0010 (17)	−0.0004 (18)
C21	0.064 (3)	0.068 (3)	0.052 (2)	0.003 (2)	−0.003 (2)	−0.008 (2)
C22	0.051 (3)	0.101 (4)	0.077 (3)	−0.003 (3)	−0.010 (2)	0.004 (3)
C23	0.057 (3)	0.089 (4)	0.058 (3)	0.006 (3)	0.006 (2)	−0.003 (3)

Geometric parameters (Å, °)

O4—C26	1.218 (5)	O1—C3	1.215 (6)
O5—C43	1.323 (5)	O2—C20	1.324 (5)
O5—C40	1.467 (4)	O2—C17	1.454 (5)
O6—C43	1.207 (5)	O3—C20	1.202 (5)
N3—C44	1.361 (5)	N1—C21	1.365 (5)
N3—C46	1.385 (5)	N1—C23	1.388 (6)
N3—C43	1.395 (5)	N1—C20	1.399 (5)
N4—C44	1.299 (6)	N2—C21	1.297 (6)
N4—C45	1.382 (7)	N2—C22	1.379 (7)
C24—C25	1.528 (6)	C1—C2	1.531 (7)
C24—C33	1.539 (6)	C1—C10	1.553 (6)
C24—H24A	0.9700	C1—H1A	0.9700
C24—H24B	0.9700	C1—H1B	0.9700
C25—C26	1.494 (7)	C2—C3	1.521 (9)
C25—H25A	0.9700	C2—H2A	0.9700
C25—H25B	0.9700	C2—H2B	0.9700
C26—C27	1.449 (7)	C3—C4	1.430 (8)
C27—C28	1.336 (6)	C4—C5	1.338 (6)
C27—H27	0.9300	C4—H4	0.9300
C28—C29	1.501 (6)	C5—C6	1.503 (6)
C28—C33	1.527 (5)	C5—C10	1.526 (6)
C29—C30	1.518 (6)	C6—C7	1.509 (6)
C29—H29A	0.9700	C6—H6A	0.9700
C29—H29B	0.9700	C6—H6B	0.9700

C30—C31	1.533 (5)	C7—C8	1.531 (5)
C30—H30A	0.9700	C7—H7A	0.9700
C30—H30B	0.9700	C7—H7B	0.9700
C31—C37	1.518 (5)	C8—C14	1.528 (5)
C31—C32	1.544 (5)	C8—C9	1.548 (5)
C31—H31	0.9800	C8—H8	0.9800
C32—C34	1.547 (5)	C9—C11	1.541 (5)
C32—C33	1.574 (5)	C9—C10	1.559 (5)
C32—H32	0.9800	C9—H9	0.9800
C33—C42	1.551 (6)	C10—C19	1.541 (6)
C34—C35	1.539 (5)	C11—C12	1.526 (6)
C34—H34A	0.9700	C11—H11A	0.9700
C34—H34B	0.9700	C11—H11B	0.9700
C35—C36	1.518 (5)	C12—C13	1.520 (5)
C35—H35A	0.9700	C12—H12A	0.9700
C35—H35B	0.9700	C12—H12B	0.9700
C36—C40	1.529 (5)	C13—C18	1.519 (6)
C36—C41	1.533 (5)	C13—C17	1.530 (5)
C36—C37	1.547 (5)	C13—C14	1.541 (5)
C37—C38	1.541 (5)	C14—C15	1.531 (5)
C37—H37	0.9800	C14—H14	0.9800
C38—C39	1.546 (6)	C15—C16	1.558 (6)
C38—H38A	0.9700	C15—H15A	0.9700
C38—H38B	0.9700	C15—H15B	0.9700
C39—C40	1.527 (6)	C16—C17	1.531 (6)
C39—H39A	0.9700	C16—H16A	0.9700
C39—H39B	0.9700	C16—H16B	0.9700
C40—H40	0.9800	C17—H17	0.9800
C41—H41A	0.9600	C18—H18A	0.9600
C41—H41B	0.9600	C18—H18B	0.9600
C41—H41C	0.9600	C18—H18C	0.9600
C42—H42A	0.9600	C19—H19A	0.9600
C42—H42B	0.9600	C19—H19B	0.9600
C42—H42C	0.9600	C19—H19C	0.9600
C44—H44	0.9300	C21—H21	0.9300
C45—C46	1.344 (7)	C22—C23	1.331 (7)
C45—H45	0.9300	C22—H22	0.9300
C46—H46	0.9300	C23—H23	0.9300
C43—O5—C40	115.6 (3)	C20—O2—C17	117.2 (3)
C44—N3—C46	106.2 (3)	C21—N1—C23	106.2 (4)
C44—N3—C43	125.4 (4)	C21—N1—C20	125.3 (4)
C46—N3—C43	128.3 (3)	C23—N1—C20	128.5 (4)
C44—N4—C45	104.6 (4)	C21—N2—C22	104.9 (4)
C25—C24—C33	112.9 (4)	C2—C1—C10	112.5 (4)
C25—C24—H24A	109.0	C2—C1—H1A	109.1
C33—C24—H24A	109.0	C10—C1—H1A	109.1
C25—C24—H24B	109.0	C2—C1—H1B	109.1
C33—C24—H24B	109.0	C10—C1—H1B	109.1
H24A—C24—H24B	107.8	H1A—C1—H1B	107.8

C26—C25—C24	111.4 (4)	C3—C2—C1	111.1 (4)
C26—C25—H25A	109.4	C3—C2—H2A	109.4
C24—C25—H25A	109.4	C1—C2—H2A	109.4
C26—C25—H25B	109.4	C3—C2—H2B	109.4
C24—C25—H25B	109.4	C1—C2—H2B	109.4
H25A—C25—H25B	108.0	H2A—C2—H2B	108.0
O4—C26—C27	121.7 (5)	O1—C3—C4	123.5 (6)
O4—C26—C25	122.2 (5)	O1—C3—C2	120.6 (6)
C27—C26—C25	116.0 (4)	C4—C3—C2	115.8 (4)
C28—C27—C26	124.9 (4)	C5—C4—C3	125.5 (5)
C28—C27—H27	117.6	C5—C4—H4	117.3
C26—C27—H27	117.6	C3—C4—H4	117.3
C27—C28—C29	121.6 (4)	C4—C5—C6	121.3 (4)
C27—C28—C33	121.9 (4)	C4—C5—C10	122.2 (4)
C29—C28—C33	116.4 (3)	C6—C5—C10	116.4 (3)
C28—C29—C30	113.5 (4)	C5—C6—C7	113.6 (4)
C28—C29—H29A	108.9	C5—C6—H6A	108.8
C30—C29—H29A	108.9	C7—C6—H6A	108.8
C28—C29—H29B	108.9	C5—C6—H6B	108.8
C30—C29—H29B	108.9	C7—C6—H6B	108.8
H29A—C29—H29B	107.7	H6A—C6—H6B	107.7
C29—C30—C31	111.1 (3)	C6—C7—C8	111.2 (3)
C29—C30—H30A	109.4	C6—C7—H7A	109.4
C31—C30—H30A	109.4	C8—C7—H7A	109.4
C29—C30—H30B	109.4	C6—C7—H7B	109.4
C31—C30—H30B	109.4	C8—C7—H7B	109.4
H30A—C30—H30B	108.0	H7A—C7—H7B	108.0
C37—C31—C30	111.3 (3)	C14—C8—C7	111.5 (3)
C37—C31—C32	109.4 (3)	C14—C8—C9	109.3 (3)
C30—C31—C32	109.0 (3)	C7—C8—C9	109.3 (3)
C37—C31—H31	109.0	C14—C8—H8	108.9
C30—C31—H31	109.0	C7—C8—H8	108.9
C32—C31—H31	109.0	C9—C8—H8	108.9
C31—C32—C34	113.2 (3)	C11—C9—C8	113.1 (3)
C31—C32—C33	112.2 (3)	C11—C9—C10	112.7 (3)
C34—C32—C33	111.6 (3)	C8—C9—C10	112.0 (3)
C31—C32—H32	106.4	C11—C9—H9	106.1
C34—C32—H32	106.4	C8—C9—H9	106.1
C33—C32—H32	106.4	C10—C9—H9	106.1
C28—C33—C24	110.0 (3)	C5—C10—C19	108.5 (4)
C28—C33—C42	109.0 (3)	C5—C10—C1	109.8 (4)
C24—C33—C42	110.2 (3)	C19—C10—C1	110.2 (4)
C28—C33—C32	107.5 (3)	C5—C10—C9	108.1 (3)
C24—C33—C32	108.7 (3)	C19—C10—C9	112.0 (3)
C42—C33—C32	111.5 (3)	C1—C10—C9	108.3 (3)
C35—C34—C32	111.9 (3)	C12—C11—C9	113.5 (3)
C35—C34—H34A	109.2	C12—C11—H11A	108.9
C32—C34—H34A	109.2	C9—C11—H11A	108.9
C35—C34—H34B	109.2	C12—C11—H11B	108.9

C32—C34—H34B	109.2	C9—C11—H11B	108.9
H34A—C34—H34B	107.9	H11A—C11—H11B	107.7
C36—C35—C34	111.5 (3)	C13—C12—C11	111.1 (3)
C36—C35—H35A	109.3	C13—C12—H12A	109.4
C34—C35—H35A	109.3	C11—C12—H12A	109.4
C36—C35—H35B	109.3	C13—C12—H12B	109.4
C34—C35—H35B	109.3	C11—C12—H12B	109.4
H35A—C35—H35B	108.0	H12A—C12—H12B	108.0
C35—C36—C40	116.5 (3)	C18—C13—C12	111.0 (3)
C35—C36—C41	111.3 (3)	C18—C13—C17	109.6 (3)
C40—C36—C41	109.7 (3)	C12—C13—C17	116.1 (3)
C35—C36—C37	107.0 (3)	C18—C13—C14	113.1 (3)
C40—C36—C37	98.2 (3)	C12—C13—C14	107.7 (3)
C41—C36—C37	113.4 (3)	C17—C13—C14	98.9 (3)
C31—C37—C38	118.2 (3)	C8—C14—C15	118.4 (3)
C31—C37—C36	113.0 (3)	C8—C14—C13	113.1 (3)
C38—C37—C36	103.4 (3)	C15—C14—C13	103.6 (3)
C31—C37—H37	107.2	C8—C14—H14	107.0
C38—C37—H37	107.2	C15—C14—H14	107.0
C36—C37—H37	107.2	C13—C14—H14	107.0
C37—C38—C39	103.9 (3)	C14—C15—C16	104.1 (3)
C37—C38—H38A	111.0	C14—C15—H15A	110.9
C39—C38—H38A	111.0	C16—C15—H15A	110.9
C37—C38—H38B	111.0	C14—C15—H15B	110.9
C39—C38—H38B	111.0	C16—C15—H15B	110.9
H38A—C38—H38B	109.0	H15A—C15—H15B	109.0
C40—C39—C38	105.4 (3)	C17—C16—C15	105.1 (3)
C40—C39—H39A	110.7	C17—C16—H16A	110.7
C38—C39—H39A	110.7	C15—C16—H16A	110.7
C40—C39—H39B	110.7	C17—C16—H16B	110.7
C38—C39—H39B	110.7	C15—C16—H16B	110.7
H39A—C39—H39B	108.8	H16A—C16—H16B	108.8
O5—C40—C39	113.2 (3)	O2—C17—C13	109.1 (3)
O5—C40—C36	109.9 (3)	O2—C17—C16	114.4 (3)
C39—C40—C36	105.3 (3)	C13—C17—C16	105.0 (3)
O5—C40—H40	109.5	O2—C17—H17	109.4
C39—C40—H40	109.5	C13—C17—H17	109.4
C36—C40—H40	109.5	C16—C17—H17	109.4
C36—C41—H41A	109.5	C13—C18—H18A	109.5
C36—C41—H41B	109.5	C13—C18—H18B	109.5
H41A—C41—H41B	109.5	H18A—C18—H18B	109.5
C36—C41—H41C	109.5	C13—C18—H18C	109.5
H41A—C41—H41C	109.5	H18A—C18—H18C	109.5
H41B—C41—H41C	109.5	H18B—C18—H18C	109.5
C33—C42—H42A	109.5	C10—C19—H19A	109.5
C33—C42—H42B	109.5	C10—C19—H19B	109.5
H42A—C42—H42B	109.5	H19A—C19—H19B	109.5
C33—C42—H42C	109.5	C10—C19—H19C	109.5
H42A—C42—H42C	109.5	H19A—C19—H19C	109.5

H42B—C42—H42C	109.5	H19B—C19—H19C	109.5
O6—C43—O5	127.1 (4)	O3—C20—O2	126.9 (4)
O6—C43—N3	122.1 (4)	O3—C20—N1	122.8 (4)
O5—C43—N3	110.8 (3)	O2—C20—N1	110.3 (3)
N4—C44—N3	112.5 (4)	N2—C21—N1	112.0 (4)
N4—C44—H44	123.8	N2—C21—H21	124.0
N3—C44—H44	123.8	N1—C21—H21	124.0
C46—C45—N4	111.2 (4)	C23—C22—N2	111.5 (5)
C46—C45—H45	124.4	C23—C22—H22	124.2
N4—C45—H45	124.4	N2—C22—H22	124.2
C45—C46—N3	105.4 (4)	C22—C23—N1	105.4 (4)
C45—C46—H46	127.3	C22—C23—H23	127.3
N3—C46—H46	127.3	N1—C23—H23	127.3
C33—C24—C25—C26	−55.7 (6)	C10—C1—C2—C3	−55.5 (6)
C24—C25—C26—O4	−150.4 (5)	C1—C2—C3—O1	−150.8 (5)
C24—C25—C26—C27	33.3 (7)	C1—C2—C3—C4	33.1 (7)
O4—C26—C27—C28	−179.5 (5)	O1—C3—C4—C5	−178.7 (5)
C25—C26—C27—C28	−3.1 (7)	C2—C3—C4—C5	−2.7 (8)
C26—C27—C28—C29	170.1 (4)	C3—C4—C5—C6	170.8 (5)
C26—C27—C28—C33	−5.8 (7)	C3—C4—C5—C10	−6.0 (7)
C27—C28—C29—C30	134.0 (4)	C4—C5—C6—C7	134.2 (4)
C33—C28—C29—C30	−49.8 (5)	C10—C5—C6—C7	−48.8 (5)
C28—C29—C30—C31	51.9 (5)	C5—C6—C7—C8	51.4 (5)
C29—C30—C31—C37	−177.8 (3)	C6—C7—C8—C14	−177.9 (3)
C29—C30—C31—C32	−57.0 (4)	C6—C7—C8—C9	−57.1 (4)
C37—C31—C32—C34	−50.1 (4)	C14—C8—C9—C11	−48.8 (4)
C30—C31—C32—C34	−172.0 (3)	C7—C8—C9—C11	−171.0 (4)
C37—C31—C32—C33	−177.5 (3)	C14—C8—C9—C10	−177.4 (3)
C30—C31—C32—C33	60.6 (4)	C7—C8—C9—C10	60.4 (4)
C27—C28—C33—C24	−16.1 (6)	C4—C5—C10—C19	104.2 (5)
C29—C28—C33—C24	167.7 (4)	C6—C5—C10—C19	−72.8 (4)
C27—C28—C33—C42	104.8 (5)	C4—C5—C10—C1	−16.3 (6)
C29—C28—C33—C42	−71.4 (4)	C6—C5—C10—C1	166.8 (4)
C27—C28—C33—C32	−134.2 (4)	C4—C5—C10—C9	−134.2 (4)
C29—C28—C33—C32	49.6 (4)	C6—C5—C10—C9	48.8 (5)
C25—C24—C33—C28	46.1 (5)	C2—C1—C10—C5	46.4 (5)
C25—C24—C33—C42	−74.1 (5)	C2—C1—C10—C19	−73.1 (5)
C25—C24—C33—C32	163.5 (4)	C2—C1—C10—C9	164.2 (4)
C31—C32—C33—C28	−55.2 (4)	C11—C9—C10—C5	176.6 (4)
C34—C32—C33—C28	176.6 (3)	C8—C9—C10—C5	−54.6 (4)
C31—C32—C33—C24	−174.1 (3)	C11—C9—C10—C19	−63.9 (5)
C34—C32—C33—C24	57.6 (4)	C8—C9—C10—C19	64.9 (4)
C31—C32—C33—C42	64.3 (4)	C11—C9—C10—C1	57.7 (5)
C34—C32—C33—C42	−64.0 (4)	C8—C9—C10—C1	−173.5 (3)
C31—C32—C34—C35	49.5 (4)	C8—C9—C11—C12	48.7 (5)
C33—C32—C34—C35	177.3 (3)	C10—C9—C11—C12	177.0 (4)
C32—C34—C35—C36	−54.9 (5)	C9—C11—C12—C13	−53.9 (5)
C34—C35—C36—C40	168.3 (3)	C11—C12—C13—C18	−65.6 (4)
C34—C35—C36—C41	−64.9 (4)	C11—C12—C13—C17	168.3 (3)

C34—C35—C36—C37	59.6 (4)	C11—C12—C13—C14	58.7 (4)
C30—C31—C37—C38	−61.0 (5)	C7—C8—C14—C15	−60.7 (5)
C32—C31—C37—C38	178.5 (3)	C9—C8—C14—C15	178.4 (3)
C30—C31—C37—C36	178.1 (3)	C7—C8—C14—C13	177.9 (3)
C32—C31—C37—C36	57.6 (4)	C9—C8—C14—C13	57.0 (4)
C35—C36—C37—C31	−62.6 (4)	C18—C13—C14—C8	60.7 (4)
C40—C36—C37—C31	176.3 (3)	C12—C13—C14—C8	−62.3 (4)
C41—C36—C37—C31	60.6 (4)	C17—C13—C14—C8	176.6 (3)
C35—C36—C37—C38	168.5 (3)	C18—C13—C14—C15	−68.7 (4)
C40—C36—C37—C38	47.3 (4)	C12—C13—C14—C15	168.3 (3)
C41—C36—C37—C38	−68.4 (4)	C17—C13—C14—C15	47.2 (4)
C31—C37—C38—C39	−158.9 (3)	C8—C14—C15—C16	−159.0 (3)
C36—C37—C38—C39	−33.2 (4)	C13—C14—C15—C16	−32.9 (4)
C37—C38—C39—C40	5.4 (4)	C14—C15—C16—C17	5.4 (4)
C43—O5—C40—C39	62.8 (4)	C20—O2—C17—C13	−178.3 (3)
C43—O5—C40—C36	−179.8 (3)	C20—O2—C17—C16	64.5 (5)
C38—C39—C40—O5	144.7 (3)	C18—C13—C17—O2	−48.2 (4)
C38—C39—C40—C36	24.7 (4)	C12—C13—C17—O2	78.5 (4)
C35—C36—C40—O5	79.9 (4)	C14—C13—C17—O2	−166.7 (3)
C41—C36—C40—O5	−47.7 (4)	C18—C13—C17—C16	74.8 (4)
C37—C36—C40—O5	−166.3 (3)	C12—C13—C17—C16	−158.5 (3)
C35—C36—C40—C39	−157.9 (3)	C14—C13—C17—C16	−43.7 (4)
C41—C36—C40—C39	74.5 (4)	C15—C16—C17—O2	143.7 (4)
C37—C36—C40—C39	−44.1 (3)	C15—C16—C17—C13	24.2 (4)
C40—O5—C43—O6	2.6 (6)	C17—O2—C20—O3	−3.5 (6)
C40—O5—C43—N3	−178.9 (3)	C17—O2—C20—N1	175.7 (3)
C44—N3—C43—O6	−6.6 (7)	C21—N1—C20—O3	−5.5 (7)
C46—N3—C43—O6	170.9 (5)	C23—N1—C20—O3	172.0 (5)
C44—N3—C43—O5	174.8 (4)	C21—N1—C20—O2	175.3 (4)
C46—N3—C43—O5	−7.7 (6)	C23—N1—C20—O2	−7.2 (6)
C45—N4—C44—N3	2.0 (6)	C22—N2—C21—N1	0.3 (6)
C46—N3—C44—N4	−1.8 (6)	C23—N1—C21—N2	−0.8 (6)
C43—N3—C44—N4	176.2 (4)	C20—N1—C21—N2	177.1 (4)
C44—N4—C45—C46	−1.4 (7)	C21—N2—C22—C23	0.3 (7)
N4—C45—C46—N3	0.3 (6)	N2—C22—C23—N1	−0.8 (7)
C44—N3—C46—C45	0.9 (5)	C21—N1—C23—C22	1.0 (6)
C43—N3—C46—C45	−177.1 (5)	C20—N1—C23—C22	−176.9 (5)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C16—H16A···O1^{i}	0.97	2.54	3.455 (6)	157
C39—H39A···O4^{i}	0.97	2.52	3.460 (6)	162
C44—H44···N2^{ii}	0.93	2.58	3.414 (6)	149

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

Fig. 1

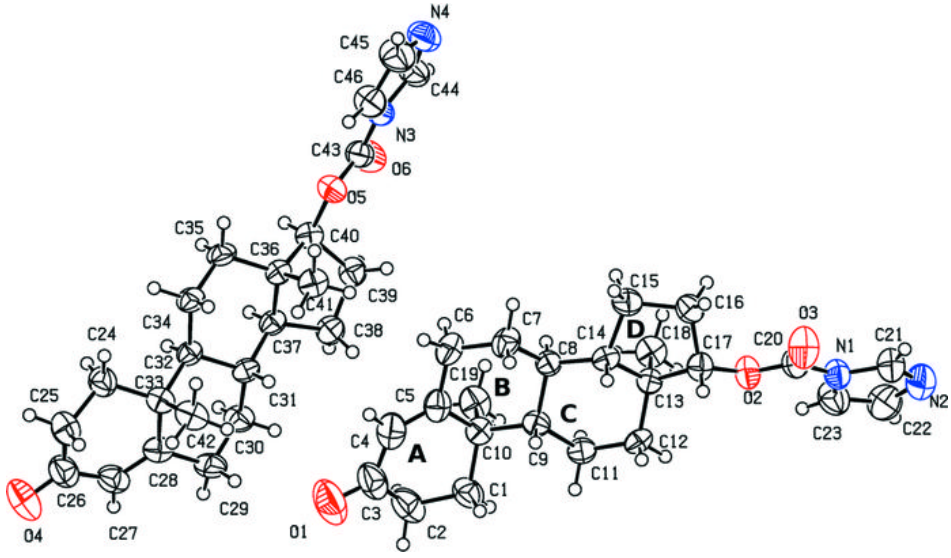
N4
C45
C46
C44
N3
C43
O6
O5
C35
C36
C40
C39
C41
C38
C37
C34
C24
C32
C33
C25
C31
C42
C30
C28
C29
C26
C27
O4
O1
C3
C2
C4
A
C5
C19
B
C10
C1
C6
C7
C8
C9
C11
C14
D
C15
C16
C18
C17
C13
C12
C
O2
O3
C20
N1
C21
N2
C22
C23

Fig. 2