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9α-Chlorocortisol, an Active Cortisol Derivative

BY CHARLES M. WEEKS AND WILLIAM L. DUAX

Medical Foundation of Buffalo, 73 High Street, Buffalo, New York 14203, *U.S.A.*

AND MANFRED E. WOLFF

Department of Pharmaceutical Chemistry, School of Pharmacy, University of California, San Francisco, California 94122, *U.S.A.*

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Abstract. $C_{21}H_{29}ClO_5$, monoclinic, $P2_1$, $a=12{\cdot}580$ (6), $b=7{\cdot}658$ (4), $c=10{\cdot}687$ (6) Å, $\beta=115{\cdot}03$ (5)°, $Z=2$, $M=396{\cdot}92$, $D_c=1{\cdot}41$, $D_m=1{\cdot}43$g cm^{-3}, m.p. 190°C (with decomposition). The molecular conformation, including the *A*-ring and C(17) side-chain orientations, is similar to that of cortisol and 9α-bromocortisol.

Introduction. The crystal structure of 9α-chlorocortisol was determined as part of a study of structure-activity relationships among the 9α-derivatives of cortisol. A crystal with dimensions $0{\cdot}05\times0{\cdot}2\times1{\cdot}0$ mm was used for the X-ray measurements of the lattice parameters and intensities. The systematic absences ($0k0$; $k=2n+1$) indicated the space group to be $P2_1$ (C_2^2, No. 4), and the cell constants were determined by a least-squares analysis of the 2θ values for 31 reflections having 2θ in the range 50–60° [at 20°C; λ(Cu $K\alpha$) = 1·54178 Å]. The intensities of the 1678 independent reflections with $2\theta<130°$ were measured on a GE XRD-5 diffractometer by the stationary-counter stationary-crystal technique using Cu $K\alpha$ radiation monochromated by balanced nickel and cobalt filters. After the Lorentz and polarization corrections [$(1+\cos^2 2\theta)/2\sin 2\theta$] had been applied, normalized structure factor amplitudes were computed, and the phases were found by the *MULTAN* program (Germain, Main & Woolfson, 1971).

The atomic parameters of 9α-chlorocortisol were refined by block-diagonal least-squares calculations. After four cycles of anisotropic refinement, a difference map showed the locations of 22 of the 29 hydrogen atoms, and a second difference map did not reveal any additional atoms. The missing hydrogens included all of the hydroxyl hydrogens. Missing hydrogens bonded to carbon atoms were placed at their geometrically expected positions by assuming tetrahedral and trigonal planar geometry at carbon atoms with sp^3 and sp^2 hydridization respectively. The parameters for all the atoms including the hydrogens were then refined for two final cycles using data for which $\sin\theta/\lambda>0{\cdot}15$. Reflections (171 during the final cycle) for which $|F_c|/|F_o|$ was less than 0·7 were also excluded from the refinement. Weights were chosen such that $\langle w\Delta^2\rangle$ would be independent of $|F_o|$ where $w^{-1}=\{1+[(|F_o|-b)/a]^2\}$ and the constants a and b were taken to be 7e and 5e respectively. The R index was defined as $\sum(||F_o|-|F_c||)/\sum|F_o|$, and its final value was 6·5% for the 1277 reflections having $I>3\sigma$ and 8·0% for all data. The final refined positional and thermal parameters are given in Table 1.*

Discussion. The crystallographically observed conformation of 9α-chlorocortisol (9α-chloro-11β,17α,21-trihydroxy-4-pregnene-3,20-dione) is shown in Fig. 1. This figure also illustrates the atomic numbering and the non-hydrogen thermal vibration ellipsoids scaled to 50% probability. The interatomic distances and bond angles involving the nonhydrogen atoms are

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30528 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. *Atomic coordinates and temperature factors*

(*a*) Final atomic coordinates and anisotropic thermal parameters for the nonhydrogen atoms. The thermal parameters are of the form $\exp[-2\pi^2(U_{11}h^2a^{*2}+2U_{12}hka^*b^*+\ldots)]$. The standard deviations of the last two figures are given in parentheses.

	x/A	y/B	z/C	U11	U22	U33	U12	U13	U23
C(1)	0.3304 (7)	0.6541 (13)	0.7969 (8)	0.0750(47)	0.0850(55)	0.0614(42)	-0.0195(50)	0.0266(38)	-0.0003(46)
C(2)	0.3019 (7)	0.7642 (15)	0.6659 (9)	0.0735(47)	0.0920(64)	0.0794(49)	-0.0070(52)	0.0304(41)	-0.0000(55)
C(3)	0.2561 (6)	0.6597 (13)	0.5393 (7)	0.0725(45)	0.0755(51)	0.0625(41)	0.0099(45)	0.0330(36)	-0.0015(42)
C(4)	0.1799 (6)	0.5146 (11)	0.5379 (7)	0.0670(40)	0.0649(46)	0.0487(35)	0.0133(36)	0.0183(30)	-0.0029(34)
C(5)	0.1611 (6)	0.4645 (12)	0.6487 (7)	0.0488(35)	0.0713(49)	0.0605(41)	0.0108(37)	0.0147(31)	-0.0030(40)
C(6)	0.0664 (7)	0.3347 (12)	0.6336 (8)	0.0732(46)	0.0771(52)	0.0504(38)	-0.0123(44)	0.0133(35)	-0.0126(39)
C(7)	0.0917 (6)	0.2105 (12)	0.7514 (7)	0.0621(39)	0.0653(50)	0.0650(39)	-0.0044(37)	0.0168(32)	-0.0023(38)
C(8)	0.1590 (5)	0.2897 (9)	0.8960 (6)	0.0393(28)	0.0555(39)	0.0456(30)	-0.0044(27)	0.0082(23)	-0.0139(28)
C(9)	0.2642 (5)	0.4023 (10)	0.9006 (6)	0.0496(35)	0.0664(44)	0.0465(32)	0.0005(34)	0.0205(28)	-0.0100(33)
C(10)	0.2237 (5)	0.5495 (10)	0.7872 (7)	0.0508(34)	0.0581(40)	0.0613(38)	-0.0007(33)	0.0222(30)	-0.0109(35)
C(11)	0.3519 (6)	0.4610 (14)	1.0479 (8)	0.0658(44)	0.1015(68)	0.0591(42)	-0.0336(49)	0.0257(36)	-0.0258(47)
C(12)	0.3763 (6)	0.3189 (13)	1.1598 (7)	0.0583(40)	0.0914(57)	0.0567(39)	-0.0086(41)	0.0250(33)	-0.0016(40)
C(13)	0.2642 (5)	0.2327 (11)	1.1557 (6)	0.0397(29)	0.0600(40)	0.0560(34)	0.0009(33)	0.0135(26)	-0.0107(37)
C(14)	0.1964 (5)	0.1582 (10)	1.0089 (7)	0.0471(32)	0.0586(41)	0.0608(37)	-0.0025(34)	0.0190(29)	-0.0133(35)
C(15)	0.1029 (6)	0.0434 (10)	1.0256 (7)	0.0606(38)	0.0540(39)	0.0674(42)	-0.0124(34)	0.0246(33)	-0.0101(35)
C(16)	0.1662 (7)	-0.0333 (12)	1.1717 (9)	0.0807(48)	0.0589(45)	0.0782(50)	-0.0092(42)	0.0346(42)	-0.0105(42)
C(17)	0.2848 (6)	0.0629 (10)	1.2446 (7)	0.0570(36)	0.0517(39)	0.0604(38)	0.0056(32)	0.0172(30)	-0.0129(34)
C(18)	0.1912 (7)	0.3642 (11)	1.1945 (8)	0.0737(46)	0.0569(44)	0.0664(43)	0.0011(38)	0.0344(38)	-0.0105(38)
C(19)	0.1340 (8)	0.6749 (13)	0.8028 (9)	0.0880(55)	0.0730(56)	0.0933(56)	0.0062(48)	0.0505(48)	-0.0143(49)
C(20)	0.3187 (7)	0.1047 (11)	1.3951 (8)	0.0798(49)	0.0547(48)	0.0804(50)	0.0060(40)	0.0426(42)	0.0036(41)
C(21)	0.4460 (7)	0.1145 (13)	1.4910 (8)	0.0793(48)	0.0769(59)	0.0594(41)	-0.0001(47)	0.0191(38)	0.0032(41)
CL(9)	0.3569 (1)	0.2500 (—)	0.8531 (1)	0.0665(9)	0.0929(13)	0.0663(9)	0.0208(11)	0.0314(8)	0.0009(11)
O(3)	0.2756 (6)	0.6944 (10)	0.4397 (6)	0.1187(48)	0.0968(53)	0.0837(39)	0.0066(41)	0.0540(37)	0.0063(38)
O(11)	0.3093 (7)	0.6166 (9)	1.0808 (6)	0.1445(55)	0.0693(40)	0.0813(38)	-0.0383(42)	0.0571(39)	-0.0265(34)
O(17)	0.3780 (5)	-0.0313 (10)	1.2350 (5)	0.0834(36)	0.1018(46)	0.0737(34)	0.0317(37)	0.0326(29)	-0.0072(35)
O(20)	0.2473 (5)	0.1277 (9)	1.4409 (5)	0.0929(38)	0.0829(42)	0.0752(33)	0.0099(36)	0.0458(31)	0.0012(32)
O(21)	0.4652 (5)	0.1773 (9)	1.6236 (5)	0.1070(41)	0.0876(42)	0.0565(28)	-0.0114(36)	0.0320(28)	-0.0147(30)

Table 1 (*cont.*)

(*b*) Final atomic coordinates for the hydrogen atoms. The average refined value for *B*iso was 4·9 Å^2, and the average standard deviations of the *x*, *y*, and *z* coordinates and the isotropic thermal parameters were 0·009, 0·016, 0·011, and 2·3, respectively. 'T' indicates a hydrogen which was not found on a Fourier difference map and which was placed at its theoretically (see text) expected position. The hydroxyl hydrogens could not be located and are not listed in this table.

	x	*y*	*z*
H(1*A*)	0·387	0·582	0·790
H(1*B*)	0·359	0·725	0·869
T(2*A*)	0·385	0·812	0·660
T(2*B*)	0·247	0·851	0·656
T(4)	0·142	0·448	0·453
H(6*A*)	0·033	0·278	0·540
H(6*B*)	−0·018	0·419	0·607
H(7*A*)	0·142	0·141	0·739
H(7*B*)	0·017	0·160	0·748
H(8)	0·115	0·379	0·907
T(11*A*)	0·432	0·482	1·039
H(12*A*)	0·401	0·233	1·134
H(12*B*)	0·440	0·353	1·268
H(14)	0·251	0·101	0·996
H(15*A*)	0·060	−0·036	0·944
H(15*B*)	0·046	0·111	1·025
H(16*A*)	0·181	−0·155	1·185
H(16*B*)	0·125	−0·008	1·217
H(18*A*)	0·115	0·306	1·205
H(18*B*)	0·148	0·452	1·124
H(18*C*)	0·241	0·432	1·267
H(19*A*)	0·070	0·622	0·798
H(19*B*)	0·091	0·787	0·725
H(19*C*)	0·158	0·707	0·871
H(21*A*)	0·481	0·216	1·434
H(21*B*)	0·495	−0·013	1·514

given in Fig. 2, and there are no unusual values. The standard deviations of the distance and angle measurements are in the ranges 0·009–0·014 Å and 0·5–0·7°, respectively. The carbon–hydrogen distances are in the range 0·8–1·18 Å. The C(9)–Cl distance of 1·865 Å is long, but this is expected since the C(9)–halogen distances have also been found to be lengthened in 9α-fluorocortisol (Weeks, Duax & Wolff, 1973), 9α-bromocortisol (Weeks & Duax, 1973), and 9α-bromo-17β-hydroxy-17α-methylandrost-4-ene-3,11-dione (Cooper, Lu & Norton, 1968).

A list of all the torsional angles involving nonhydrogen atoms is given in Table 2. The intra-ring torsional angles show that the *A*-ring conformation is intermediate between a 2β-sofa and a 1α,2β-half-chair,* and the *B* and *C*-rings have normal chair conformations. The *D*-ring conformation is intermediate between a 13β,14α-half-chair and a 13β-envelope, but more nearly a half-chair as shown by the parameters $\Delta = 10{\cdot}9$ and $\varphi_m = 47{\cdot}9$ (Altona, Geise & Romers, 1968). Inspection of the torsional angles involving the C(17) side chain shows that it has the usual corticoid orientation (Weeks, Duax & Wolff, 1973). Atoms O(20) and O(21) are *cis* coplanar, and O(20) is oriented over the *D*-ring in agreement with solution spectral measurements (Wellman & Djerassi, 1965).

Close intermolecular contacts between nonhydrogen atoms are shown in Table 3. All hydroxyl oxygen atoms are involved in close contacts whose magnitudes are outside the range commonly observed for steroid hydrogen bonding (2·70–2·90 Å). This absence of strong hydrogen bonding is uncommon in the corticoid series.

The glucocorticoid activity of cortisol as measured by the glycogen deposition and anti-inflammatory

* An ideal sofa conformation is defined as a six-membered ring conformation in which five atoms are coplanar. An ideal half-chair conformation, in the case of six-membered rings, is that conformation in which four contiguous atoms are coplanar and the two remaining atoms are equidistant from the plane but on opposite sides of the plane (Bucourt & Hainaut, 1965). In the case of steroid rings, the identity and directions of deviation of the out-of-plane atom(s) should also be indicated.

Table 2. *All torsional angles involving nonhydrogen atoms*

φ is the torsional angle *i–j–k–l*. The sign convention of Klyne & Prelog (1960) is used.

i	j	k	l	φ	i	j	k	l	φ	i	j	k	l	φ	i	j	k	l	φ
C10	C1	C2	C3	- 55.8°	C6	C7	C8	C14	-172.2°	C8	C9	C11	C12	39.3°	C14	C13	C17	C16	- 40.7°
C2	C1	C10	C5	44.2	C7	C8	C9	C10	56.1	C8	C9	C11	O11	- 85.8	C14	C13	C17	C20	-163.5
C2	C1	C10	C9	164.3	C7	C8	C9	C11	-169.4	C10	C9	C11	C12	172.2	C14	C13	C17	O17	77.5
C2	C1	C10	C19	- 72.7	C7	C8	C9	C19	- 58.7	C10	C9	C11	O11	47.2	C18	C13	C17	C16	78.3
C1	C2	C3	C4	36.3	C14	C8	C9	C10	-176.3	C19	C9	C11	C12	- 73.7	C18	C13	C17	C20	- 44.5
C1	C2	C3	O3	-146.6	C14	C8	C9	C11	- 41.8	C19	C9	C11	O11	161.2	C18	C13	C17	O17	-163.5
C2	C3	C4	C5	- 6.9	C14	C8	C9	C19	68.9	C9	C11	C12	C13	- 48.1	C8	C14	C15	C16	-163.9
O3	C3	C4	C5	176.0	C7	C8	C14	C13	-180.0	O11	C11	C12	C13	75.0	C13	C14	C15	C16	- 35.7
C3	C4	C5	C6	169.6	C7	C8	C14	C15	- 57.9	C11	C12	C13	C14	56.4	C14	C15	C16	C17	10.0
C3	C4	C5	C10	- 3.6	C9	C8	C14	C13	54.2	C11	C12	C13	C17	167.5	C15	C16	C17	C13	19.1
C4	C5	C6	C7	144.8	C9	C8	C14	C15	176.3	C11	C12	C13	C18	- 66.5	C15	C16	C17	C20	140.0
C10	C5	C6	C7	- 41.7	C8	C9	C10	C1	-180.0	C12	C13	C14	C8	- 61.0	C15	C16	C17	O17	- 96.3
C4	C5	C10	C1	- 16.1	C8	C9	C10	C5	- 57.5	C12	C13	C14	C15	168.3	C13	C17	C20	C21	- 93.7
C4	C5	C10	C9	-136.9	C8	C9	C10	C19	58.9	C17	C13	C14	C8	178.3	C13	C17	C20	O20	87.5
C4	C5	C10	C19	102.7	C11	C9	C10	C1	46.1	C17	C13	C14	C15	47.6	C16	C17	C20	C21	150.7
C6	C5	C10	C1	170.4	C11	C9	C10	C5	168.4	C18	C13	C14	C8	61.0	C16	C17	C20	O20	- 28.1
C6	C5	C10	C9	49.6	C11	C9	C10	C19	- 75.2	C18	C13	C14	C15	- 69.7	O17	C17	C20	C21	25.1
C6	C5	C10	C19	- 70.7	CL9	C9	C10	C1	- 65.4	C12	C13	C17	C16	-155.3	O17	C17	C20	O20	-153.7
C5	C6	C7	C8	38.4	CL9	C9	C10	C5	56.9	C12	C13	C17	C20	81.9	C17	C20	C21	O21	172.1
C6	C7	C8	C9	- 45.5	CL9	C9	C10	C19	173.4	C12	C13	C17	O17	- 37.1	O20	C20	C21	O21	- 9.1

Table 3. *Intermolecular non-hydrogen distances less than* 3·5 Å

The equivalent positions are $1=(x,y,z)$ and $2=(\bar{x},\frac{1}{2}+y,\bar{z})$. The notation 2/010 means that the second atom is at equivalent position 2, translated one unit cell in the **b** direction.

Atom 1	Atom 2	Distance	Position
C(4)	C(20)	3·36 Å	1/00$\bar{1}$
C(16)	O(3)	3·33	1/0$\bar{1}$1
C(18)	O(3)	3·47	1/001
C(20)	O(3)	3·26	1/0$\bar{1}$1
C(21)	O(3)	3·32	2/1$\bar{1}$2
Cl(9)	O(21)	3·32	1/00$\bar{1}$
O(3)	O(20)	3·34	1/01$\bar{1}$
O(11)	O(17)	3·09	1/010
O(11)	O(21)	3·27	2/103
O(17)	O(21)	2·94	2/1$\bar{1}$3

assays is considerably enhanced by 9α-fluorination. 9α-Chlorination or dehydrogenation of atoms C(1) and C(2) results in a smaller increase, and 9α-bromination decreases activity (Fried, 1961). These biological effects have been correlated with conformational features of cortisol, 9α-fluorocortisol, 9α-bromocortisol, and 6α-methylprednisolone determined through crystallographic studies.* Specifically, the pronounced bending of the *A*-rings of 9α-fluorocortisol and 6α-methylprednisolone below the *BCD*-plane appears to be related to enhancement of corticoid activity (Weeks, Duax & Wolff, 1973). The *A*-ring orientations of 9α-chlorocortisol, 9α-bromocortisol, and cortisol itself do not exhibit this pronounced bending. The strain associated with the 9α-chlorine appears to cause a flattening of the *B* and *C*-rings rather than a change in *A*-ring orientation. On the basis of numerous earlier steroid structure determinations, the torsional angles in the *B* and *C*-rings would be expected to be in the range 50–55°. The exceptionally small angles [*e.g.* C(5)–C(6)–C(7)–C(8) = 38·4° and C(8)–C(9)–C(11)–C(12) = 39·3°] observed in the 9α-chlorocortisol structure are an expression of this flattening effect. The *A*-ring orientation observed in the 9α-chlorocortisol

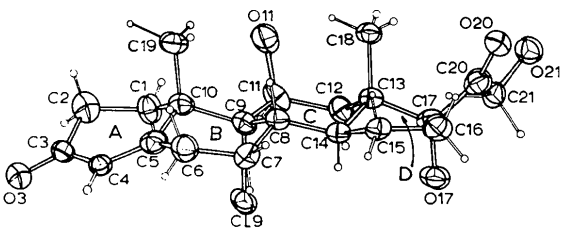

Fig. 1. Observed conformation of 9α-chlorocortisol. The atomic numbering and thermal vibration ellipsoids, scaled to 50% probability, of the nonhydrogen atoms are illustrated.

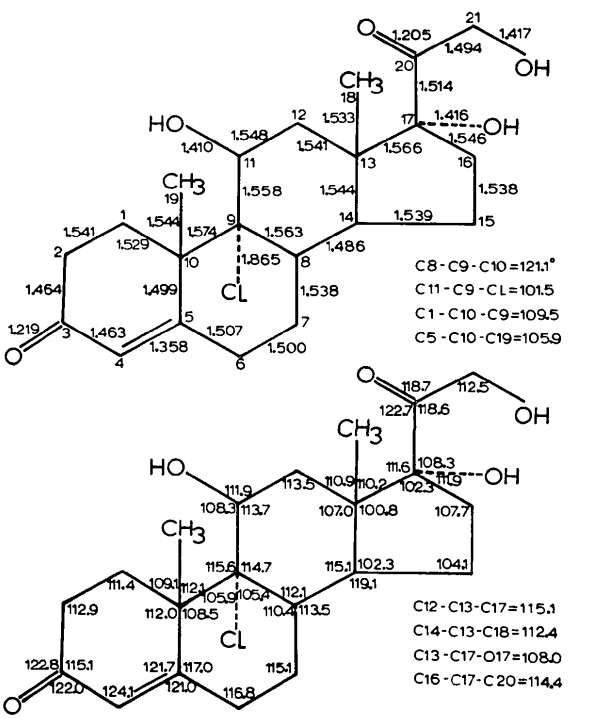

Fig. 2. Intramolecular geometry for 9α-chlorocortisol. (*a*) Interatomic distances. (*b*) Bond angles.

* Cortisol: Roberts, Coppola, Isaacs & Kennard (1973); 9α-fluorocortisol: Dupont, Dideberg & Campsteyn (1972) and Weeks, Duax & Wolff (1973); 9α-bromocortisol: Weeks & Duax (1973); 6α-methylprednisolone: Declerq, Germain & Van Meerssche (1972).

structure is not the orientation predicted on the basis of the biological data. This contradiction may arise because the bioassays measure several parameters including absorption, transport, and solubility properties in addition to affinities for intracellular receptors. Consequently, a complete understanding of the molecular features required for maximal glucocorticoid activity must await the availability of more complete data concerning the binding of these hormones to individual proteins.

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9-Carbomethoxy-4,6,6-trimethyl-*trans*-decal-3-one*

BY CAROL SAUNDERSON HUBER

Division of Biological Sciences, National Research Council of Canada, Ottawa, Canada K1A 0R6

AND ERIC J. GABE

Division of Chemistry, National Research Council of Canada, Ottawa, Canada K1A 0R9

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Abstract. $C_{15}H_{24}O_3$, triclinic, space group $P\bar{1}$; $a=8{\cdot}082$ (1), $b=14{\cdot}398$ (2), $c=6{\cdot}976$ (1) Å, $\alpha=111{\cdot}64$ (1), $\beta=103{\cdot}01$ (1), $\gamma=71{\cdot}23$ (1)°, $V=709{\cdot}27$ Å^3, based on λ(Mo $K\alpha_1$) = 0·70926 Å. $D_m=1{\cdot}180$, $D_c=1{\cdot}181$ g cm^{-3}, $Z=2$, μ(Mo $K\alpha$) = 0·86 cm^{-1}, $M=252{\cdot}34$, $F(000)=276$. The ring junction is *trans*, with both rings in chair conformation. The methyl group attached to the cyclohexanone ring is equatorial, and the methyl ester at the ring junction is axial.

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Experimental. The colourless prismatic crystals of this compound were supplied by Dr J. W. ApSimon of Carleton University. Initial precession photographs indicated that the symmetry was triclinic, and subse-

Table 1. *Fractional coordinates, vibration tensor components* (Å^2) *for the expression* $T=\exp[-2\pi^2(U_{11}a^{*2}h^2 + \ldots + 2U_{23}b^*c^*kl + \ldots]$, *and their e.s.d.'s, all* $\times 10^4$

	x	y	z	U_{11}	U_{22}	U_{33}	$2U_{23}$	$2U_{13}$	$2U_{12}$
C(1)	7083 (2)	7890 (1)	−326 (3)	351 (7)	522 (8)	608 (9)	562 (15)	110 (13)	−235 (12)
C(2)	7096 (2)	8588 (1)	1937 (3)	443 (8)	592 (9)	602 (10)	491 (16)	−179 (14)	−465 (14)
C(3)	5342 (2)	9374 (1)	2303 (2)	573 (9)	468 (8)	361 (7)	333 (12)	−132 (12)	−393 (14)
C(4)	3770 (2)	8927 (1)	1664 (2)	423 (7)	370 (7)	349 (7)	186 (11)	34 (11)	−173 (11)
C(5)	2199 (2)	7781 (1)	−1520 (2)	327 (6)	430 (7)	343 (7)	178 (11)	111 (10)	−150 (11)
C(6)	2141 (2)	7174 (1)	−3854 (2)	396 (7)	455 (8)	347 (7)	185 (11)	34 (11)	−214 (11)
C(7)	3901 (2)	6348 (1)	−4259 (2)	517 (8)	412 (8)	359 (7)	88 (12)	148 (12)	−85 (12)
C(8)	5491 (2)	6785 (1)	−3399 (2)	414 (7)	498 (8)	376 (7)	282 (12)	252 (11)	2 (12)
C(9)	5559 (2)	7372 (1)	−1043 (2)	331 (6)	382 (7)	359 (7)	287 (11)	131 (10)	−124 (10)
C(10)	3803 (2)	8222 (1)	−637 (2)	338 (6)	339 (6)	328 (6)	237 (10)	70 (10)	−102 (10)
O(11)	5214 (2)	10296 (1)	3019 (2)	861 (9)	467 (6)	601 (7)	336 (11)	−115 (13)	−582 (12)
C(12)	2066 (2)	9776 (1)	2063 (3)	512 (9)	499 (9)	519 (10)	−51 (15)	86 (15)	−2 (15)
C(13)	649 (3)	6637 (2)	−4508 (3)	601 (11)	730 (12)	506 (9)	157 (17)	−51 (15)	−670 (18)
C(14)	1786 (2)	7902 (1)	−5125 (2)	497 (8)	606 (9)	399 (8)	398 (14)	11 (13)	−114 (14)
C(15)	5878 (2)	6599 (1)	111 (2)	374 (6)	365 (7)	383 (7)	277 (11)	47 (10)	−166 (10)
O(16)	4941 (2)	6598 (1)	1210 (2)	643 (7)	602 (7)	735 (8)	770 (13)	579 (13)	7 (11)
O(17)	7397 (1)	5868 (1)	−258 (2)	441 (6)	531 (7)	824 (8)	793 (12)	326 (11)	93 (10)
C(18)	7810 (2)	5088 (1)	739 (3)	525 (9)	517 (9)	869 (13)	832 (19)	36 (17)	−119 (15)