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Structure of Modification I of Cortisone Acetate, 21-Acetoxy-17 α -hydroxy-4-pregnene-3,11,20-trione,* C₂₃H₃₀O₆

BY J. A. KANTERS, A. DE KOSTER AND V. J. VAN GEERESTEIN

Laboratorium voor Kristal- en Structuurchemie, Rijksuniversiteit, Padualaan 8, 3508 TB Utrecht, The Netherlands

AND L. A. VAN DIJCK

Organon International BV, Analytical R & D Laboratories, PO Box 20, 5340 BH Oss, The Netherlands

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Abstract. $M_r = 402.49$, orthorhombic, $P2_12_12_1$, $a = 8.018$ (4), $b = 12.579$ (7), $c = 20.963$ (1) Å, $Z = 4$, $V = 2114$ (2) Å³, $D_x = 1.264$ g cm⁻³, Mo $K\alpha$, $\lambda = 0.71069$ Å, $\mu = 0.53$ cm⁻¹, $F(000) = 864$, $T = 294$ K, $R = 0.049$ for 1302 observed reflections with $I > 2.5\sigma(I)$. Rings A , B and C have distorted 1α -sofa (A) and distorted chair (B and C) conformations in both modifications I and II [Declercq, Germain & Van Meerssche (1972). *Cryst. Struct. Commun.* **1**, 59–62]. The differences between the two forms relate to ring D which is a 13β -envelope in I and a $13\beta,14\alpha$ -half chair in II, and to the orientation of the side chains, where the average differences of the torsion angles about C(17)–C(20) and C(20)–C(21) are 16 (1) and 14 (1)° respectively. In I the molecules are connected by an intermolecular head-to-tail H bond [O(17)–H...O(3)], while in II there is an O(17)–H...O(22) head-to-head H bond. The packing mode of I is 221, of II 212.

Introduction. Three anhydrous polymorphs of cortisone acetate (CA), hereafter called mod I, mod II and mod III, have been reported in the literature (Callow & Kennard, 1961; Kuhnert-Brandstätter & Grimm, 1968*a,b*; Carless, Moustafa & Rapson, 1966; Shirotani & Sekiguchi, 1981). In pharmaceutical literature the high-melting polymorph is called mod I (Kuhnert-

Brandstätter & Grimm, 1968*a,b*) which is identical to modification III of Callow & Kennard (1961). In this paper the pharmaceutical nomenclature will be followed.

Both mod I and mod II were shown to be orthorhombic with space group $P2_12_12_1$, $Z = 4$ (Callow & Kennard, 1961), but with considerably different cell dimensions (for mod II: $a = 11.048$, $b = 7.102$, $c = 27.095$ Å), indicating a different packing of CA molecules. A sample manufactured by Laboratoires Français de Chimiothérapie (Roussel) was also analysed by Callow & Kennard (1961) and this form (mod III) was reported to be monoclinic ($P2_1$) with three independent molecules in the unit cell.

A structure analysis of mod II, which can easily be prepared from solvents like benzene or chloroform, was performed by Declercq, Germain & Van Meerssche (1972). The present paper reports the analysis of mod I.

Experimental. Crystallization of CA from absolute ethanol affords the mono-ethanolate from which solvent-free CA was obtained by heating at 373 K. The resulting powder, consisting of mod I, was used to seed a solution of CA in hot toluene. Slow cooling of this solution yields a mixture of single crystals of mod I, which appear as prisms, and of mod II, which are in the form of fine needles. A crystal of mod I with dimensions $0.3 \times 0.4 \times 1.0$ mm was selected for the analysis. D_m not determined.

* IUPAC name: 17 α -hydroxy-3,11,20-trioxo-4-pregnen-21-yl acetate.

Table 1. Atomic fractional coordinates and equivalent isotropic thermal parameters (\AA^2) with e.s.d.'s in parentheses
$$U_{\text{eq}} = (U_{11} + U_{22} + U_{33})/3.$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
O(3)	0.0930 (6)	0.2792 (2)	0.5379 (2)	0.083 (1)
O(11)	-0.3145 (4)	0.7231 (3)	0.5890 (3)	0.093 (2)
O(17)	-0.0159 (4)	1.0711 (2)	0.5424 (1)	0.060 (1)
O(20)	-0.0952 (5)	1.1842 (3)	0.6891 (2)	0.074 (1)
O(21)	-0.3933 (5)	1.2317 (3)	0.6383 (2)	0.075 (1)
O(22)	-0.4596 (5)	1.1098 (4)	0.7114 (2)	0.097 (2)
C(1)	-0.1253 (7)	0.5310 (3)	0.5462 (2)	0.063 (2)
C(2)	-0.1199 (8)	0.4087 (4)	0.5512 (3)	0.070 (2)
C(3)	0.0578 (8)	0.3698 (4)	0.5531 (2)	0.064 (2)
C(4)	0.1832 (7)	0.4404 (4)	0.5766 (2)	0.064 (2)
C(5)	0.1486 (6)	0.5395 (3)	0.6011 (2)	0.056 (1)
C(6)	0.2852 (7)	0.6004 (4)	0.6334 (3)	0.079 (2)
C(7)	0.2887 (6)	0.7183 (4)	0.6136 (3)	0.066 (2)
C(8)	0.1188 (5)	0.7675 (3)	0.6252 (2)	0.049 (1)
C(9)	-0.0148 (5)	0.7079 (3)	0.5855 (2)	0.042 (1)
C(10)	-0.0247 (6)	0.5866 (3)	0.6000 (2)	0.049 (1)
C(11)	-0.1819 (6)	0.7672 (4)	0.5858 (2)	0.056 (1)
C(12)	-0.1798 (6)	0.8868 (3)	0.5774 (2)	0.051 (1)
C(13)	-0.0516 (5)	0.9370 (3)	0.6230 (2)	0.044 (1)
C(14)	0.1162 (5)	0.8850 (3)	0.6097 (2)	0.047 (1)
C(15)	0.2439 (6)	0.9566 (3)	0.6437 (3)	0.062 (1)
C(16)	0.1706 (6)	1.0695 (4)	0.6360 (3)	0.058 (2)
C(17)	-0.0090 (6)	1.0581 (3)	0.6099 (2)	0.050 (1)
C(18)	-0.1093 (7)	0.9245 (4)	0.6924 (2)	0.060 (1)
C(19)	-0.1070 (7)	0.5672 (4)	0.6663 (2)	0.063 (2)
C(20)	-0.1288 (7)	1.1359 (3)	0.6408 (2)	0.052 (1)
C(21)	-0.2939 (7)	1.1541 (3)	0.6082 (2)	0.062 (2)
C(22)	-0.4659 (7)	1.2003 (6)	0.6920 (3)	0.075 (2)
C(23)	-0.5604 (9)	1.2880 (6)	0.7230 (3)	0.107 (2)

Enraf-Nonius CAD-4 diffractometer, Zr-filtered Mo $K\alpha$ radiation, ω - 2θ scan, $\omega = (0.65 + 0.35 \tan^2\theta)$ with a threshold $\sigma(I)/I < 1\%$ or a maximum scan time of 60 s, horizontal and vertical slit widths 3.0 and 4.0 mm respectively. Lattice dimensions from 25 reflections in the θ range 10.5 – 14.8° . Within one octant of the reflection sphere (h 0–8, k 0–13, l 0–22) with $\theta_{\text{max}} = 22^\circ$, 1546 intensities, 1302 above the $2.5\sigma(I)$ level. Four periodically measured standard reflections (123 , $\bar{1}\bar{2}\bar{3}$, $1\bar{2}\bar{3}$, $\bar{1}2\bar{3}$) showed insignificant changes. Corrections for Lorentz–polarization effects, but not for absorption. Structure solved with *MULTAN80* (Main *et al.*, 1980); best E map gave 23 out of 29 non-H atoms, remaining atoms from difference synthesis; H atoms at $C(sp^3)$ and $C(sp^2)$ at calculated positions, H of OH group located in difference synthesis and refined with a fixed B of 0.05 \AA^2 , all other H's included in refinement riding on bonded C with constant B of 0.05 \AA^2 . Anisotropic, weighted full-matrix (two blocks) refinement on F resulted in $R = 0.049$ and $wR = 0.060$ with $w = [\sigma^2(F_o) + 0.003646F_o^2]^{-1}$, $S = 1.16$. Average Δ/σ 0.04, max. 0.18 for non-H atoms. Max. excursions in final difference Fourier synthesis $\pm 0.17 e \text{ \AA}^{-3}$. No correction for secondary extinction. Scattering factors for C and O from Cromer & Mann (1968), for H from Stewart, Davidson & Simpson (1965). Calculations

Table 2. Bond distances (\AA) and bond angles ($^\circ$) for non-H atoms, with e.s.d.'s in parentheses

O(3)–C(3)	1.217 (6)	C(8)–C(9)	1.550 (6)
O(11)–C(11)	1.201 (6)	C(8)–C(14)	1.513 (5)
O(17)–C(17)	1.426 (5)	C(9)–C(10)	1.558 (5)
O(20)–C(20)	1.211 (6)	C(9)–C(11)	1.534 (6)
O(21)–C(21)	1.409 (6)	C(10)–C(19)	1.558 (6)
O(21)–C(22)	1.328 (7)	C(11)–C(12)	1.515 (6)
O(22)–C(22)	1.210 (9)	C(12)–C(13)	1.539 (6)
C(1)–C(2)	1.543 (6)	C(13)–C(14)	1.522 (6)
C(1)–C(10)	1.553 (6)	C(13)–C(17)	1.585 (5)
C(2)–C(3)	1.507 (9)	C(13)–C(18)	1.535 (6)
C(3)–C(4)	1.429 (8)	C(14)–C(15)	1.539 (6)
C(4)–C(5)	1.377 (6)	C(15)–C(16)	1.545 (6)
C(5)–C(6)	1.498 (7)	C(16)–C(17)	1.547 (7)
C(5)–C(10)	1.511 (7)	C(17)–C(20)	1.517 (6)
C(6)–C(7)	1.540 (7)	C(20)–C(21)	1.507 (7)
C(7)–C(8)	1.516 (6)	C(22)–C(23)	1.49 (1)
C(21)–O(21)–C(22)	114.9 (5)	C(9)–C(11)–C(12)	118.2 (4)
C(2)–C(1)–C(10)	112.7 (4)	C(11)–C(12)–C(13)	110.0 (4)
C(1)–C(2)–C(3)	110.6 (5)	C(12)–C(13)–C(14)	107.5 (3)
O(3)–C(3)–C(4)	121.1 (5)	C(12)–C(13)–C(17)	115.5 (3)
O(3)–C(3)–C(10)	120.6 (6)	C(12)–C(13)–C(18)	110.2 (4)
C(2)–C(3)–C(4)	118.2 (5)	C(14)–C(13)–C(17)	101.0 (3)
C(3)–C(4)–C(5)	123.3 (5)	C(14)–C(13)–C(18)	113.3 (4)
C(4)–C(5)–C(6)	119.0 (4)	C(17)–C(13)–C(18)	109.1 (3)
C(4)–C(5)–C(10)	122.3 (4)	C(8)–C(14)–C(13)	113.1 (3)
C(6)–C(5)–C(10)	118.6 (4)	C(8)–C(14)–C(15)	117.6 (4)
C(5)–C(6)–C(7)	112.6 (4)	C(13)–C(14)–C(15)	104.6 (3)
C(6)–C(7)–C(8)	109.5 (4)	C(14)–C(15)–C(16)	103.7 (4)
C(7)–C(8)–C(9)	109.7 (4)	C(15)–C(16)–C(17)	107.8 (4)
C(7)–C(8)–C(14)	112.1 (4)	O(17)–C(17)–C(13)	105.9 (3)
C(9)–C(8)–C(14)	110.3 (3)	O(17)–C(17)–C(16)	112.1 (4)
C(8)–C(9)–C(10)	113.8 (3)	O(17)–C(17)–C(20)	109.0 (3)
C(8)–C(9)–C(11)	111.5 (3)	C(13)–C(17)–C(16)	103.2 (3)
C(10)–C(9)–C(11)	115.5 (4)	C(13)–C(17)–C(20)	114.2 (4)
C(1)–C(10)–C(9)	108.2 (4)	C(16)–C(17)–C(20)	112.3 (4)
C(1)–C(10)–C(19)	109.0 (3)	O(20)–C(20)–C(17)	122.7 (5)
C(1)–C(10)–C(19)	110.9 (4)	O(20)–C(20)–C(21)	119.9 (4)
C(5)–C(10)–C(9)	109.9 (4)	C(17)–C(20)–C(21)	117.4 (4)
C(5)–C(10)–C(19)	108.3 (4)	O(21)–C(21)–C(20)	113.5 (4)
C(9)–C(10)–C(19)	110.4 (3)	O(21)–C(21)–O(22)	123.1 (6)
O(11)–C(11)–C(9)	123.3 (4)	O(21)–C(21)–C(23)	111.9 (6)
O(11)–C(11)–C(12)	118.4 (4)	O(22)–C(22)–C(23)	124.9 (6)

performed with *SHELX76* (Sheldrick, 1976) (refinement) and the *EUCLID* package (Spek, 1982) (geometry and illustrations) on the CDC CYBER-175 computer of the University of Utrecht.

Discussion. The atomic coordinates and equivalent isotropic temperature factors are presented in Table 1,* bond distances and bond angles are given in Table 2 and a view of the molecule with atom numbering is shown in Fig. 1. Taking into account the standard deviations of the bond distances and angles in mod II (Declercq, Germain & Van Meerssche, 1972), which are in the ranges 0.11 – 0.21 \AA and 0.6 – 1.2° respectively, the corresponding distances and angles of mod I and mod II agree within 3σ .

* Lists of structure factors, anisotropic thermal parameters, H-atom positions, bond distances, bond angles and torsion angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39999 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

In the steroid backbone ring *A* has a 1α -sofa conformation, rings *B* and *C* are slightly distorted chairs and ring *D* is a $13\beta,14\alpha$ -half chair. The ring junctions *B/C* and *C/D* are both *trans*. Comparison of mod I and mod II reveals moderate differences for rings *B* and *C* which in mod I are more distorted from the ideal chair as follows from the endocyclic torsion angles and puckering parameters (Cremer & Pople, 1975) (Table 3). The most notable differences relate to ring *D*, which is a $13\beta,14\alpha$ -half chair in mod I, but a 13β -envelope in mod II.

The side chain consists of two planar four-atom fragments, C(17),C(20),O(20),C(21) and the acetate group O(21),C(22),O(22),C(23), which are connected by C(21)—O(21); the angle between the least-squares planes is $77.2(3)^\circ$ and carbonyl O(22) is synperiplanar with respect to C(21). In mod II the side chain also consists of these two planar parts, the angle between the least-squares planes here being $87.0(6)^\circ$.

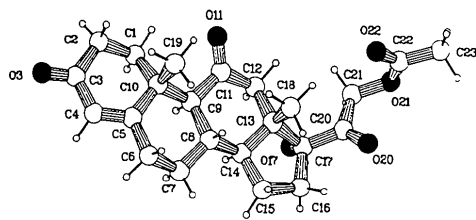


Fig. 1. Perspective view of modification I of cortisone acetate showing the atom-numbering system.

The main differences relate to the torsion angles about C(17)—C(20) and C(20)—C(21) which show average differences of $16(1)$ and $14(1)^\circ$ respectively (Table 3). An intermolecular H bond with O—H, O...O and O—H...O, $0.88(4)$, $2.761(4)$ Å and $168(4)^\circ$, respectively, involves O(17)—H as donor and carbonyl O(3) as acceptor. The donor and acceptor molecules are separated by one translation along **b** and so an infinite chain of head-to-tail H bonds is formed in this direction. According to the classification of Duax & Norton (1975) this packing arrangement can be described as $Oc_{17}a_{16}b_7-221$, indicating that the steroid thickness, width and length are at 17° to *c*, 16° to *a* and 7° to *b*, respectively, and that the molecules in the cell are packed two molecules thick, two wide and one long.

Comparison with mod II shows that in mod II an intermolecular H bond links O(17)—H to carbonyl O(22) of the side chain of a molecule translated along **b**, thus forming an infinite chain of head-to-head H bonds. The packing mode of mod II is entirely different, as follows from its classification $Oa_{38}b_{39}c_{11}-212$. The H bonds and packing modes of mod I and mod II are shown in Figs. 2 and 3. As is frequently observed in oxo-steroids (Duax & Norton, 1975) the carbonyl groups in mod I have short intermolecular contacts, O(3) with C(17) and C(20) (both at $x, -1+y, z$) and C(21)($\frac{1}{2}+x, \frac{3}{2}-y, 1-z$) at $3.268(5)$, $3.326(6)$ and $3.303(6)$ Å respectively, O(11) with C(7)($-1+x, y, z$) at $3.224(6)$ Å and O(22) with C(15) and C(16) (both at $-1+x, y, z$) at $3.373(7)$ and $3.398(7)$ Å respectively.

Table 3. Comparison of endocyclic and side-chain torsion angles ($^\circ$) and puckering parameters θ and φ (Cremer & Pople, 1975) of modification I (present work) and modification II (Declercq, Germain & Van Meerssche, 1972) of cortisone acetate

Ring A	Mod I	Mod II	Ring C	Mod I	Mod II
C(10)—C(1)—C(2)—C(3)	-54.7 (6)	-54 (1)	C(14)—C(8)—C(9)—C(11)	-45.3 (5)	-49.6 (9)
C(1)—C(2)—C(3)—C(4)	25.8 (6)	31 (1)	C(8)—C(9)—C(11)—C(12)	43.1 (5)	54.1 (9)
C(2)—C(3)—C(4)—C(5)	4.1 (7)	-1 (2)	C(9)—C(11)—C(12)—C(13)	-49.0 (5)	-57.9 (9)
C(3)—C(4)—C(5)—C(10)	-5.5 (7)	-7 (2)	C(11)—C(12)—C(13)—C(14)	56.2 (4)	58.2 (8)
C(4)—C(5)—C(10)—C(1)	-22.6 (5)	-16 (1)	C(12)—C(13)—C(14)—C(8)	-64.6 (4)	-62.7 (8)
C(5)—C(10)—C(1)—C(2)	52.1 (5)	46 (1)	C(13)—C(14)—C(8)—C(9)	59.2 (5)	57.1 (9)
θ	57.5 (6)	58 (2)	θ	14.2 (5)	6.9 (9)
φ	2.0 (8)	12 (2)	φ	267.6 (2)	239 (7)
Ring B			Ring D		
C(10)—C(5)—C(6)—C(7)	-47.4 (6)	-49 (1)	C(17)—C(13)—C(14)—C(15)	44.8 (4)	48.4 (8)
C(5)—C(6)—C(7)—C(8)	54.7 (6)	52 (1)	C(13)—C(14)—C(15)—C(16)	-34.9 (5)	-32.6 (9)
C(6)—C(7)—C(8)—C(9)	-59.8 (5)	-56 (1)	C(14)—C(15)—C(16)—C(17)	10.5 (6)	3 (1)
C(7)—C(8)—C(9)—C(10)	57.8 (5)	59.2 (9)	C(15)—C(16)—C(17)—C(13)	16.4 (5)	26.9 (9)
C(8)—C(9)—C(10)—C(5)	-46.5 (5)	-54.4 (9)	C(16)—C(17)—C(13)—C(14)	-37.1 (4)	-46.5 (8)
C(9)—C(10)—C(5)—C(6)	42.1 (5)	49 (1)	θ	193.6 (7)	184 (1)
θ	13.6 (5)	8 (1)			
φ	158.4 (2)	199 (1)			
Side chain					
C(13)—C(17)—C(20)—C(21)	-79.1 (5)	-94.4 (9)	C(17)—C(20)—C(21)—O(21)	-176.8 (4)	168.5 (7)
C(13)—C(17)—C(20)—O(20)	102.3 (5)	85 (1)	O(20)—C(20)—C(21)—O(21)	1.9 (6)	-11 (1)
C(16)—C(17)—C(20)—C(21)	163.9 (4)	149.0 (8)	C(20)—C(21)—O(21)—C(22)	-75.2 (5)	-82 (1)
C(16)—C(17)—C(20)—O(20)	-14.7 (6)	-32 (1)	C(21)—O(21)—C(22)—C(23)	176.7 (5)	178 (1)
O(17)—C(17)—C(20)—C(21)	39.1 (5)	24 (1)	C(21)—O(21)—C(22)—O(22)	-5.9 (8)	-3 (2)
O(17)—C(17)—C(20)—O(20)	-139.5 (4)	-156.8 (9)			

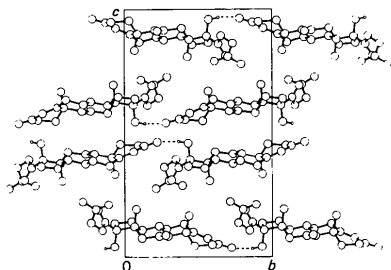


Fig. 2. Packing of modification I as seen along **a**. Hydrogen bonds are denoted by dashed lines.

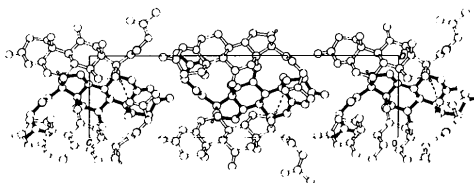


Fig. 3. Packing of modification II as seen along **a**. Hydrogen bonds are denoted by dashed lines.

Five of these six contacts involve molecules which are separated by translations along **a** or **b** and, together with the H bond that connects molecules also translated along **b**, these short contacts form a cluster of molecules parallel to the *ab* plane. The remaining contact between molecules related by the screw axis parallel to **a** connects these layers of molecules in a coil

from which the angular methyl groups project sideways.

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11 β -Hydroxy-16 α ,17 α ,21-trimethyl-5 α -pregna-1,4-diene-3,20-dione (I), C₂₄H₃₄O₃, and 11 β ,17 α -Dihydroxy-21-methyl-5 α -pregn-4-en-3,20-dione 17-Butyrate (II), C₂₆H₃₈O₅

BY JOHN C. BARNES* AND JOHN D. PATON

Chemistry Department, The University, Dundee DD1 4HN, Scotland

AND ROBERT T. LOGAN AND JAMES REDPATH

Organon Laboratories Ltd, Newhouse, Lanarkshire ML1 5SH, Scotland

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Abstract. (I) $M_r = 370.5$, monoclinic, $P2_1$, $a = 11.76$ (1), $b = 11.75$ (1), $c = 7.596$ (4) Å, $\beta = 98.85$ (8)°, $U = 1037.1$ Å³, $Z = 2$, $D_x = 1.187$ Mg m⁻³, $\text{Cu } K\alpha$, $\lambda = 1.5418$ Å, $\mu = 0.51$ mm⁻¹, $F(000) = 404$, $T = 293$ K, $R = 0.070$ for 1235 reflec-

tions. (II) $M_r = 430.6$, orthorhombic, $P2_12_12_1$, $a = 15.1065$ (11), $b = 10.6656$ (9), $c = 14.7405$ (17) Å, $U = 2374.99$ Å³, $Z = 4$, $D_x = 1.098$ Mg m⁻³, $\text{Mo } K\alpha$, $\lambda = 0.71069$ Å, $\mu = 0.05$ mm⁻¹, $F(000) = 936$, $T = 293$ K, $R = 0.044$ for 1746 reflections. Structurally (I) and (II) are typical 17-substituted steroids. In each case the molecules are linked into chains by hydrogen bonds.

* Author for correspondence.