

bonds corresponds with the H···O distances as well as with the N···O distances [N(4)···O(5^v) 2.745 (1) > N(1)···O(3ⁱ) 2.815 (1) > N(2)···O(3ⁱⁱⁱ) 2.926 (1) > N(2)···O(5ⁱⁱ) 2.957 (1) Å].

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Structure of Prudomestin

BY VIRINDER S. PARMAR, ANAND VARDHAN AND RAKESH K. SHARMA

Department of Chemistry, University of Delhi, Delhi-110 007, India

AND NATHANIEL W. ALCOCK AND WILLIAM ERRINGTON

Department of Chemistry, University of Warwick, Coventry CV4 7AL, England

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Abstract. 3,5,7-Trihydroxy-8-methoxy-2-(4-methoxyphenyl)-4*H*-1-benzopyran-4-one, $C_{17}H_{14}O_7$, $M_r = 330.3$, triclinic, $P\bar{1}$, $a = 6.579$ (5), $b = 8.441$ (7), $c = 13.893$ (14) Å, $\alpha = 101.92$ (9), $\beta = 103.38$ (6), $\gamma = 97.44$ (6)°, $V = 721$ Å³, $Z = 2$, $D_x = 1.52$ g cm⁻³, Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å, $\mu = 1.2$ cm⁻¹, $F(000) = 344$, $T = 200$ K, $R = 0.076$ for 1534 unique observed [$I/\sigma(I) \geq 2.0$] reflections. The definitive structure of prudomestin is established by this investigation. The molecule is essentially planar with the methyl groups projecting above and below the plane. The major intermolecular force within a layer of molecules in the solid state results from hydrogen bonding.

Introduction. *Prunus domestica Linn.* is the tree of the well-known fruit plum and grows ideally in the western temperate Himalayas and is cultivated for its fruit in India. Its fruits are used as a mild laxative and demulcent and, in combination with other drugs, they are used in leucorrhoea, irregular menstruation and debility following miscarriage (Chopra, Nayar & Chopra, 1956). The prudomestin used in this study

was extracted from the wood of *Prunus domestica Linn.* The objective of this investigation was to provide an unambiguous identification of the structure of prudomestin.

Experimental. The prudomestin was isolated from the hot-water soluble portion of the benzene solubles of an alcoholic extract from the heartwood of *Prunus domestica Linn.* collected from Baramulla, Kashmir, India. The compound was recrystallized from chloroform–petrol and separated as yellow needles.

Data were collected with a Siemens P3R3 four-circle diffractometer in ω – 2θ mode. The crystal was held at 200 K with an Oxford Cryosystems Cryostream Cooler (version 2.4); this temperature was chosen because the crystal diffracted rather poorly under ambient conditions. Maximum 2θ was 50° with scan range ± 0.7 ° (2θ) around the $K\alpha_1$ – $K\alpha_2$ angles, scan speed 3–29° min⁻¹, depending on the intensity of the 2 s prescan; backgrounds were measured at each end of the scan for 0.25 of the scan time. hkl ranges were 0/7, -9/10, -15/15 (unique reflections only). Three standard reflections were

Table 1. *Atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^3$)*

	x	y	z	U_{eq}
O(1)	1557 (6)	5065 (4)	7652 (3)	25 (1)
O(2)	1036 (7)	662 (4)	7152 (3)	38 (2)
O(3)	-1638 (6)	1203 (5)	8315 (3)	35 (2)
O(4)	-3622 (6)	3228 (5)	9288 (3)	35 (2)
O(5)	-1950 (6)	8782 (4)	9320 (3)	30 (2)
O(6)	880 (6)	8180 (4)	8176 (3)	26 (1)
O(7)	8372 (6)	3910 (5)	5315 (3)	35 (2)
C(1)	116 (8)	5324 (6)	8202 (4)	19 (2)
C(2)	1927 (9)	3502 (6)	7311 (4)	24 (2)
C(3)	785 (9)	2230 (7)	7524 (5)	27 (2)
C(4)	-689 (9)	2425 (7)	8114 (5)	25 (2)
C(5)	-1002 (9)	4062 (6)	8450 (4)	21 (2)
C(6)	-2470 (8)	4435 (6)	9040 (4)	22 (2)
C(7)	-2740 (9)	6027 (7)	9328 (4)	25 (2)
C(8)	-1594 (8)	7246 (6)	9035 (4)	22 (2)
C(9)	-152 (8)	6931 (6)	8488 (4)	23 (2)
C(10)	3559 (8)	3545 (6)	6774 (4)	21 (2)
C(11)	4597 (9)	5042 (7)	6684 (5)	26 (2)
C(12)	6163 (9)	5111 (7)	6214 (5)	28 (2)
C(13)	6799 (9)	3686 (7)	5773 (4)	26 (2)
C(14)	5801 (9)	2215 (7)	5856 (5)	29 (2)
C(15)	4214 (10)	2128 (7)	6345 (5)	32 (2)
C(16)	3109 (9)	8691 (7)	8696 (5)	36 (3)
C(17)	8790 (11)	2485 (8)	4696 (5)	44 (3)

monitored every 200 reflections and showed slight changes during data collection. Unit-cell dimensions and standard deviations were obtained by least-squares fit to 15 reflections ($16 < 2\theta < 39^\circ$). Reflections were processed using profile analysis to give 2538 unique reflections of which 1534 were considered observed [$I/\sigma(I) \geq 2.0$] and used in refinement. These were corrected for Lorentz and polarization but not for absorption effects. Crystal dimensions were $0.06 \times 0.18 \times 0.78$ mm.

The structure was solved by direct methods using *SHELXTL-Plus* (Sheldrick, 1988). Anisotropic temperature factors were used for all non-H atoms. H atoms were given fixed isotropic temperature factors, $U = 0.08 \text{ \AA}^2$. Those defined by the molecular geometry were inserted at calculated positions and not refined.

Final refinement was on F by least-squares methods refining 217 parameters. Largest positive and negative peaks on a final difference Fourier synthesis were of height $\pm 0.4 \text{ e \AA}^{-3}$. A weighting scheme of the form $w = 1/[\sigma^2(F) + gF^2]$ with $g = 0.000882$ was used and shown to be satisfactory by a weight analysis.

Final $R = 0.076$, $wR = 0.079$ and $S = 2.1$; the relatively high R factors are considered to be a consequence of poor crystal quality. Maximum shift/e.s.d. in final cycle was 0.09. Computing was performed with *SHELXTL-Plus* on a DEC MicroVAX II. Scattering factors in the analytical form and anomalous-dispersion factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV).

Table 2. *Bond lengths (Å) and bond angles (°) with e.s.d.'s in parentheses*

O(1)—C(1)	1.361 (8)	O(2)—C(3)	1.364 (7)
O(4)—C(6)	1.340 (7)	O(1)—C(2)	1.379 (7)
O(3)—C(4)	1.251 (8)	O(5)—C(8)	1.343 (7)
O(6)—C(9)	1.379 (7)	O(6)—C(16)	1.440 (6)
O(7)—C(13)	1.347 (8)	O(7)—C(17)	1.425 (8)
C(1)—C(5)	1.364 (8)	C(1)—C(9)	1.379 (8)
C(2)—C(3)	1.353 (9)	C(2)—C(10)	1.443 (9)
C(3)—C(4)	1.412 (10)	C(4)—C(5)	1.421 (8)
C(5)—C(6)	1.429 (9)	C(6)—C(7)	1.368 (8)
C(7)—C(8)	1.380 (9)	C(8)—C(9)	1.366 (9)
C(10)—C(11)	1.399 (8)	C(10)—C(15)	1.389 (8)
C(11)—C(12)	1.343 (10)	C(12)—C(13)	1.395 (9)
C(13)—C(14)	1.367 (9)	C(14)—C(15)	1.373 (10)
C(1)—O(1)—C(2)	121.1 (5)	C(9)—O(6)—C(16)	114.2 (5)
C(13)—O(7)—C(17)	117.3 (5)	O(1)—C(1)—C(5)	121.7 (5)
O(1)—C(1)—C(9)	116.1 (5)	C(5)—C(1)—C(9)	122.2 (6)
O(1)—C(2)—C(3)	118.0 (6)	O(1)—C(2)—C(10)	110.9 (5)
C(3)—C(2)—C(10)	131.2 (5)	O(2)—C(3)—C(2)	119.3 (6)
O(2)—C(3)—C(4)	117.1 (5)	C(2)—C(3)—C(4)	123.6 (5)
O(3)—C(4)—C(3)	120.4 (5)	O(3)—C(4)—C(5)	123.5 (6)
C(3)—C(4)—C(5)	116.1 (5)	C(1)—C(5)—C(4)	119.5 (6)
C(1)—C(5)—C(6)	118.5 (5)	C(4)—C(5)—C(6)	122.0 (5)
O(4)—C(6)—C(5)	120.3 (5)	O(4)—C(6)—C(7)	120.0 (6)
C(5)—C(6)—C(7)	119.6 (5)	C(6)—C(7)—C(8)	119.2 (6)
O(5)—C(8)—C(7)	116.8 (5)	O(5)—C(8)—C(9)	120.7 (5)
C(7)—C(8)—C(9)	122.5 (5)	O(6)—C(9)—C(1)	121.7 (5)
O(6)—C(9)—C(8)	120.1 (5)	C(1)—C(9)—C(8)	118.1 (5)
C(2)—C(10)—C(11)	120.9 (5)	C(2)—C(10)—C(15)	122.3 (5)
C(11)—C(10)—C(15)	116.8 (6)	C(10)—C(11)—C(12)	121.8 (6)
C(11)—C(12)—C(13)	121.3 (6)	O(7)—C(13)—C(12)	115.9 (5)
O(7)—C(13)—C(14)	126.6 (6)	C(12)—C(13)—C(14)	117.5 (6)
C(13)—C(14)—C(15)	121.8 (6)	C(10)—C(15)—C(14)	120.8 (6)

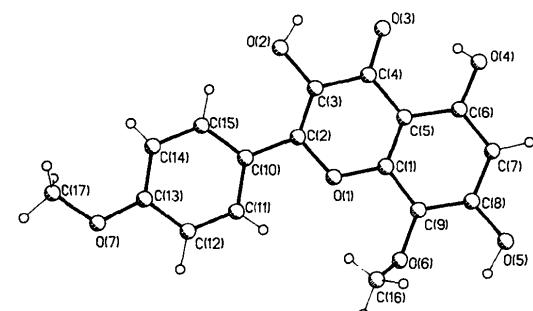


Fig. 1. View of the molecule showing the atomic numbering scheme.

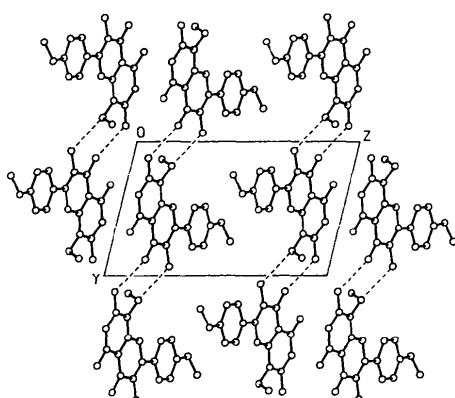
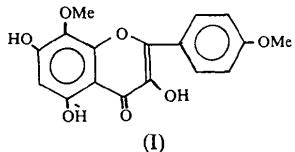


Fig. 2. Packing diagram viewed down the x axis.

Final atomic coordinates are given in Table 1, and selected bond lengths and angles in Table 2.* A view of prudomestin with the atomic numbering scheme is shown in Fig. 1. The molecular packing is shown in Fig. 2.

Discussion. The X-ray results identify prudomestin as 3,5,7-trihydroxy-8-methoxy-2-(4-methoxyphenyl)-4*H*-1-benzopyran-4-one (I).



The sample of prudomestin used in this study, and also that of Wollenweber, Dietz, Schilling, Favre-

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54876 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: LI0106]

Bonvin & Smith (1985), exhibited a bathochromic shift in its UV spectrum in the presence of NaOAc/H₃BO₃. This observation suggests but does not confirm an *ortho*-dihydroxy system (Harborne, Mabry & Mabry, 1975); definitive evidence for the absence of such a grouping is provided by this investigation.

The C–O skeleton of the molecule is essentially planar with a mean deviation from the plane of 0.05 Å; the methyl C atoms project above and below this plane. Bond lengths and angles are unexceptional. The molecules stack in parallel rows with an alternating pattern of up–down methoxy groups. The main attractive forces between the molecules in a layer involve hydrogen bonding between OH groups and methoxy or C=O groups, Fig. 2.

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Structure of (20*R*)-5*α*-Pregnano[3,4-*c*][1,2,5]oxadiazol-20-ol (HS1011)

BY DOMINIQUE MAES, LODE WYNNS AND JOHN LISGARTEN

Department of Ultrastructure, Instituut voor Molekulare Biologie, Vrije Universiteit Brussel, Paardenstraat 65, B-1640 Sint-Genesius Rode, Belgium

AND DAVID LISGARTEN AND REX PALMER*

Department of Crystallography, Birkbeck College, University of London, Malet Street, London WC1E 7HX, England

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Abstract. C₂₁H₃₂N₂O₂, $M_r = 344$, orthorhombic, P2₁2₁2₁, $a = 7.353$ (1), $b = 10.153$ (1), $c = 25.324$ (3) Å, $V = 1891$ Å³, $Z = 4$, $D_x = 1.21$ g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 5.30$ cm⁻¹, $F(000) = 752$, room temperature, $R = 0.054$ for 2968 observed reflections. All rings of the steroid skeleton are *trans* connected. Ring *A* is strained and rings *C* and *D* are in chair conformations. Ring *D* has an intermediate envelope-half-chair conformation. The oxadiazole ring is planar. The methyl and hydroxyl side groups

linked through C(18) to the steroid skeleton at C(17) are equatorial and axial respectively.

Introduction. Medicinal chemists have modified the structure of testosterone in various ways (Drill & Riegel, 1958) with the object of increasing the anabolic (nitrogen retention) propensity and decreasing its effect as a male hormone. This assumes that the target receptors associated with these two effects are sufficiently different to be sensitive to small changes in the structure of the drug molecule and to react accordingly. One successful approach has been to

* To whom correspondence should be addressed.