Table 2. *Selected bond lengths* (Å) *and angles* (°)

O(1)—C(13)	1·207 (4)	N(3)—C(22)	1·521 (5)
O(2)—C(13)	1·359 (5)	C(1)—C(2)	1·522 (5)
O(2)—C(17)	1·457 (4)	C(1)—C(5)	1·531 (5)
O(5)—N(2)	1·218 (6)	C(2)—C(3)	1·354 (5)
O(6)—N(2)	1·212 (5)	C(2)—C(13)	1·470 (4)
N(1)—C(3)	1·379 (4)	C(4)—C(5)	1·346 (5)
N(1)—C(4)	1·378 (8)	C(17)—C(18)	1·500 (5)
N(2)—C(8)	1·455 (6)	C(17)—C(21)	1·503 (7)
N(3)—C(18)	1·500 (4)	C(19)—C(20)	1·510 (6)
N(3)—C(19)	1·500 (6)	C(20)—C(21)	1·514 (5)

C(13)—O(2)—C(17)	116·1 (3)	N(1)—C(4)—C(5)	119·7 (3)
C(3)—N(1)—C(4)	123·2 (3)	C(1)—C(5)—C(4)	120·9 (3)
O(5)—N(2)—O(6)	122·4 (4)	O(1)—C(13)—O(2)	121·6 (4)
O(5)—N(2)—C(8)	117·9 (4)	O(1)—C(13)—C(2)	127·6 (4)
O(6)—N(2)—C(8)	119·7 (4)	O(2)—C(13)—C(2)	111·0 (3)
C(18)—N(3)—C(.9)	111·2 (3)	O(2)—C(17)—C(18)	110·1 (3)
C(18)—N(3)—C(22)	107·6 (3)	O(2)—C(17)—C(21)	106·8 (3)
C(19)—N(3)—C(22)	113·2 (3)	C(18)—C(17)—C(21)	113·2 (3)
C(2)—C(1)—C(5)	109·8 (3)	N(3)—C(18)—C(17)	112·4 (4)
C(1)—C(2)—C(3)	121·4 (3)	N(3)—C(19)—C(20)	110·1 (3)
C(1)—C(2)—C(13)	118·8 (4)	C(19)—C(20)—C(21)	111·2 (3)
C(3)—C(2)—C(13)	119·9 (4)	C(17)—C(21)—C)(20)	111·6 (3)
N(1)—C(3)—C(2)	119·1 (3)		

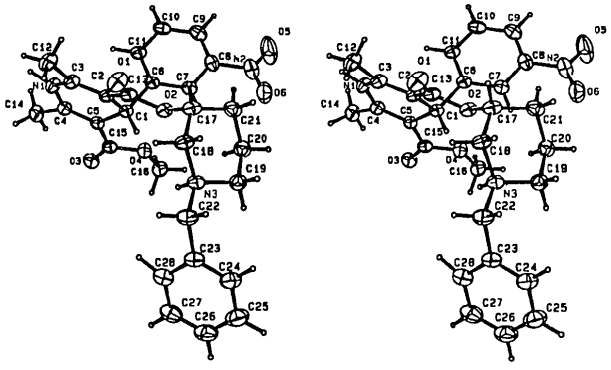

Fig. 1. An *ORTEP*II drawing (Johnson, 1976) of the molecule with the numbering system. The thermal ellipsoids are depicted at the 30% level.

10^{-6}). Scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). Final fractional coordinates and equivalent B values are listed in Table 1. Bond distances and angles are listed in Table 2.* Fig. 1 shows a stereoview of the molecule with the atomic numbering.

Related literature. The title compound is a newly developed dihydropyridine-type calcium antagonist and shows potent and long lasting antihypertensive, as well as anti-anginal, effects. The synthesis, pharmacological activities and pharmacokinetic studies are discussed in detail in a special issue of *Arzneimittel Forschung/Drug Research* (1988) on benidipine hydrochloride.

* 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 53396 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of the Flavone Hymenoxin

BY WILLIAM H. WATSON* AND RAM P. KASHYAP

Department of Chemistry, Texas Christian University, Fort Worth, Texas 76129, *USA*

FENG GAO† AND TOM J. MABRY

Department of Botany, The University of Texas, Austin, Texas 78713, *USA*

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Abstract. 2-(3,4-Dimethoxyphenyl)-5,7-dihydroxy-6,8-dimethoxy-4*H*-chromen-4-one, $C_{19}H_{18}O_8$, M_r = 374·38, monoclinic, $P2_1/n$, a = 9·026 (4), b = 15·054 (6), c = 12·829 (6) Å, β = 100·98 (4)°, V = 1711 (1) Å^3, Z = 4, D_x = 1·450 g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ = 1·07 cm^{-1}, F(000) = 784, T = 295 K, R = 0·0778 for 2280 independent reflections. The nearly planar AB ring system (0·04 Å r.m.s.d.), O(1)

* Author to whom correspondence should be addressed.

† Currently: Department of Chemistry and Cancer Research Institute, Arizona State University, Tempe, Arizona, USA.

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to C(10), and the planar *C* ring (0·001 Å r.m.s.d.) are almost coplanar with an interplanar angle of only 4·4 (4)°. The methyl groups at C(6) and C(8) are rotated out of the molecular plane on opposite sides with torsion angles C(5)C(6)O(6)C(11) = −94·5 (4) and C(7)C(8)O(8)C(12) = 109·0 (4)°. The methyl groups of ring *C* are coplanar with the ring, C(3′)-C(4′)O(4′)C(13) = 0·2 (5) and C(6′)C(5′)O(5′)C(14) = 1·3 (5)°. The carbonyl group forms an intramolecular hydrogen bond with O(5), O(5)···O(4) = 2·612 (5), H(5O)···O(4) = 1·85 (4) Å, O(5)—H(5O)···O(4) = 151·9 (8)°, and an intermolecular hydrogen bond with O(7) of an adjacent molecule, O(7)···O(4) $(-0{\cdot}5+x, \quad -0{\cdot}5-y, \quad 0{\cdot}5+z)$ = 2·689 (5), H(7O)···O(4) = 1·92 (4) Å, and O(7)—H(7O)···O(4) = 163·2 (8)°.

Experimental. Hymenoxin was previously reported from *Hymenoxys scaposa* (Thomas & Mabry, 1967); however, the sample analyzed in this work was obtained from *Hymenoxys furneri* (Gao, Wang & Mabry, 1990). A colorless, poor-quality crystal of dimensions 0·40 × 0·15 × 0·10 mm was mounted on a Nicolet $R3M/\mu$ update of a $P2_1$ diffractometer; data collected in the ω mode ($3 \le 2\theta \le 55°$), variable scan rate of 4 to 29·3 ° min^{-1} using graphite-monochromated Mo $K\alpha$ radiation; lattice parameters from the least-squares refinement of 25 reflections ($22{\cdot}97 \le 2\theta \le 28{\cdot}3°$), systematic extinctions ($h0l$, $h+l=2n+1$; $0k0$, $k=2n+1$) consistent with space group $P2_1/n$; monitored reflections (222 and $\bar{3}0\bar{1}$) showed random variations of 2%, data corrected by linear interpolation, 4361 reflections measured, 3944 independent ($-11 \le h \le 11$, $0 \le k \le 19$, $0 \le l \le 16$), equivalent reflections averaged (R_{merge} = 0·0078), 2280 had $I \ge 3\sigma(I)$; Lorentz–polarization corrections and ψ-scan absorption correction (transmission factors 1·000 to 0·698) applied; structure solved by direct methods, block-cascade least-squares refinement, on *F*, H atoms located in difference map, methyl H atoms allowed to ride on attached atom, all other H atoms refined with iso-

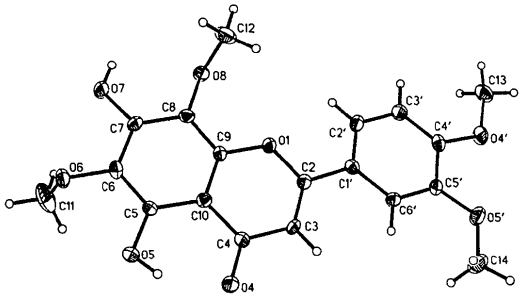

Fig. 1. Drawing of the title compound. Thermal ellipsoids are shown at the 30% probability level while H atoms are represented by spheres of arbitrary size.

Table 1. *Atomic coordinates* (× 10⁴) *and isotropic thermal parameters* (Å² × 10³)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	*y*	*z*	U_{eq}
O(1)	5718 (3)	−940 (2)	1959 (2)	31 (1)
C(2)	6446 (4)	−640 (2)	1198 (3)	28 (1)
C(3)	7076 (4)	−1204 (2)	596 (3)	30 (1)
C(4)	6954 (4)	−2145 (2)	691 (3)	28 (1)
O(4)	7537 (3)	−2673 (2)	125 (2)	41 (1)
C(5)	5821 (4)	−3370 (2)	1594 (3)	28 (1)
O(5)	6355 (3)	−3991 (2)	1008 (2)	39 (1)
C(6)	4934 (4)	−3616 (2)	2312 (3)	31 (1)
O(6)	4572 (3)	−4500 (2)	2402 (2)	40 (1)
C(7)	4404 (4)	−2983 (2)	2944 (3)	31 (1)
O(7)	3557 (3)	−3269 (2)	3626 (2)	44 (1)
C(8)	4708 (4)	−2086 (2)	2834 (3)	28 (1)
O(8)	4056 (3)	−1483 (2)	3421 (2)	34 (1)
C(9)	5525 (4)	−1835 (2)	2087 (3)	26 (1)
C(10)	6128 (4)	−2456 (2)	1468 (3)	26 (1)
C(1′)	6450 (4)	336 (2)	1143 (3)	29 (1)
C(2′)	5700 (4)	846 (3)	1767 (3)	37 (1)
C(3′)	5679 (4)	1761 (3)	1681 (3)	38 (1)
C(4′)	6401 (4)	2181 (2)	970 (3)	33 (1)
O(4′)	6457 (3)	3073 (2)	818 (2)	42 (1)
C(5′)	7170 (4)	1665 (2)	325 (3)	32 (1)
O(5′)	7864 (3)	2137 (2)	−349 (2)	50 (1)
C(6′)	7190 (4)	759 (2)	415 (3)	29 (1)
C(11)	3153 (5)	−4712 (3)	1757 (5)	72 (2)
C(12)	5078 (5)	−1069 (3)	4266 (3)	50 (2)
C(13)	5701 (5)	3630 (3)	1453 (3)	47 (2)
C(14)	8643 (6)	1654 (3)	−1025 (4)	56 (2)

Table 2. *Bond lengths* (Å) *and angles* (°)

O(1)—C(2)	1·355 (5)	O(1)—C(9)	1·372 (4)
C(2)—C(3)	1·345 (5)	C(2)—C(1′)	1·470 (5)
C(3)—C(4)	1·428 (5)	C(4)—O(4)	1·257 (5)
C(4)—C(10)	1·432 (5)	C(5)—O(5)	1·345 (5)
C(5)—C(6)	1·381 (5)	C(5)—C(10)	1·419 (5)
C(6)—O(6)	1·380 (4)	C(6)—C(7)	1·393 (5)
O(6)—C(11)	1·423 (5)	C(7)—O(7)	1·338 (5)
C(7)—C(8)	1·391 (5)	C(8)—O(8)	1·381 (4)
C(8)—C(9)	1·370 (5)	O(8)—C(12)	1·426 (5)
C(9)—C(10)	1·402 (5)	C(1′)—C(2′)	1·378 (6)
C(1′)—C(6′)	1·400 (5)	C(2′)—C(3′)	1·381 (5)
C(3′)—C(4′)	1·372 (6)	C(4′)—O(4′)	1·358 (4)
C(4′)—C(5′)	1·410 (5)	O(4′)—C(13)	1·430 (5)
C(5′)—O(5′)	1·360 (5)	C(5′)—C(6′)	1·370 (5)
O(5′)—C(14)	1·416 (6)		
C(2)—O(1)—C(9)	120·2 (3)	O(1)—C(2)—C(3)	121·3 (3)
O(1)—C(2)—C(1′)	111·9 (3)	C(3)—C(2)—C(1′)	126·7 (3)
C(2)—C(3)—C(4)	122·0 (3)	C(3)—C(4)—O(4)	122·0 (3)
C(3)—C(4)—C(10)	116·3 (3)	O(4)—C(4)—C(10)	121·7 (3)
O(5)—C(5)—C(6)	120·0 (3)	O(5)—C(5)—C(10)	120·8 (3)
C(6)—C(5)—C(10)	119·2 (3)	C(5)—C(6)—O(6)	119·4 (3)
C(5)—C(6)—C(7)	120·7 (3)	O(6)—C(6)—C(7)	119·8 (3)
C(6)—O(6)—C(11)	111·6 (3)	C(6)—C(7)—O(7)	117·6 (3)
C(6)—C(7)—C(8)	120·6 (3)	O(7)—C(7)—C(8)	121·7 (3)
C(7)—C(8)—O(8)	118·1 (3)	C(7)—C(8)—C(9)	118·8 (3)
O(8)—C(8)—C(9)	122·8 (3)	C(8)—O(8)—C(12)	114·9 (3)
O(1)—C(9)—C(8)	116·9 (3)	O(1)—C(9)—C(10)	121·0 (3)
C(8)—C(9)—C(10)	122·1 (3)	C(4)—C(10)—C(5)	122·4 (3)
C(4)—C(10)—C(9)	119·1 (3)	C(5)—C(10)—C(9)	118·5 (3)
C(2)—C(1′)—C(2′)	121·6 (3)	C(2′)—C(1′)—C(6′)	119·5 (3)
C(2′)—C(1′)—C(6′)	118·9 (3)	C(1′)—C(2′)—C(3′)	120·7 (4)
C(2′)—C(3′)—C(4′)	120·8 (4)	C(3′)—C(4′)—O(4′)	125·9 (4)
C(3′)—C(4′)—C(5′)	119·0 (3)	O(4′)—C(4′)—C(5′)	115·1 (3)
C(4′)—O(4′)—C(13)	117·6 (3)	C(4′)—C(5′)—O(5′)	115·1 (3)
C(4′)—C(5′)—C(6′)	120·0 (4)	O(5′)—C(5′)—C(6′)	125·0 (4)
C(5′)—O(5′)—C(14)	117·7 (3)	C(1′)—C(6′)—C(5′)	120·6 (4)

tropic thermal parameters; $R = 0.0778$, $wR = 0.0745$ for 280 parameters and 2280 reflections, $S = 1.443$, $(\Delta/\sigma)_{max} = 0.022$, largest peaks in the final difference map of 0.38 and -0.38 e Å^{-3}; $\sum w(|F_o| - |F_c|)^2$ minimized with $w = [\sigma^2(F_o) + 0.00071F_o^2]^{-1}$. All computer programs supplied by Nicolet (Nicolet Instrument Corporation, 1986) for Desktop 30 Microeclipse and Nova 4/*C* configuration; atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974, Vol. IV). Fig. 1 is a drawing of the compound, Table 1 lists the atomic positional parameters, and Table 2 gives interatomic distances and angles.*

Related literature. Hymenoxin and related compounds have been reported previously (Thomas & Mabry, 1967; Gutierrez & Herz, 1988). 3,5-dihydroxy-6,7,8-trimethoxyflavone (Hansel, Khaliefi & Peller, 1981) and 5-hydroxy-6,7,2′,4′,5′-pentamethoxyflavone (Al-Yaha, Hifnawy, Mossa, El-Feraly, McPhail & McPhail, 1989) exhibit highly oxygenated ring systems, and their X-ray structures can be used for comparison.

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* Lists of H-atom coordinates, anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53380 (27 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of Ethylenediammonium Terephthalate and Tetramethylenediammonium Terephthalate

By Yoshimitsu Moritani* and Setsuo Kashino

Department of Chemistry, Faculty of Science, Okayama University, Tsushima, Okayama 700, *Japan*

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Abstract. Cu $K\alpha$, $\lambda = 1.54178$ Å, $T = 295$ K. Ethylenediammonium terephthalate (2T), $C_2H_{10}N_2^{2+}.C_8H_4O_4^{2-}$, $M_r = 226.22$, monoclinic, $P2_1/n$, $a = 8.381$ (8), $b = 9.236$ (4), $c = 7.471$ (4) Å, $\beta = 115.32$ (5)°, $V = 522.8$ (7) Å^3, $Z = 2$, $D_m = 1.438$ (2), $D_x = 1.438$ Mg m^{-3}, $\mu = 0.96$ mm^{-1}, $F(000) = 240$, $R = 0.042$ for 839 unique reflections. Tetramethylenediammonium terephthalate (4T), $C_4H_{14}N_2^{2+}.C_8H_4O_4^{2-}$, $M_r = 254.27$, triclinic, $P\bar{1}$, $a = 8.3490$ (8), $b = 11.760$ (2), $c = 8.2238$ (8) Å, $\alpha = 99.37$ (1), $\beta = 91.48$ (1), $\gamma = 125.027$ (7)°, $V = 645.3$ (1) Å^3, $Z = 2$, $D_m = 1.315$ (2), $D_x = 1.309$ Mg m^{-3}, $\mu = 0.83$ mm^{-1}, $F(000) = 272$, $R = 0.048$ for 2065 unique reflections. Both the cations and anions in 2T and two crystallographically independent cations in 4T have a center of symmetry. In these crystals, the cations and anions are held together by N—H···O hydrogen bonds to form three-dimensional networks.

Experimental. Experimental details are listed in Table 1. Both crystals obtained from aqueous solutions by slow evaporation at room temperature. D_m by flotation in benzene–CCl_4. Rigaku AFC-5 four-circle diffractometer equipped with rotating anode, ω–2θ scan method [scan speed 4° min^{-1} for 2T and 6° min^{-1} for 4T in ω, scan range in ω: $(1.2 + 0.15\tan\theta)$°], Ni-filtered Cu $K\alpha$ at 40 kV, 200 mA, background measured for 4 s on either side of the peak; three standard reflections recorded every 97 reflections, no variation in intensity. Lorentz and polarization corrections; no absorption correction. All the unique reflections used in structure analysis. The structures solved by *MULTAN*84 and refined

* On leave from Mitsui Petrochemical Industries Ltd, Waki-Cho, Kuga-Gun, Yamaguchi 740, Japan.

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