Fig. 2. A perspective view of the molecule.

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Structure of Aurmillone, an Isoflavone from *Millettia auriculata**

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Abstract. 5,7-Dihydroxy-8-methoxy-3-{4-[(3-methyl-2-butenyl)oxy]phenyl}-4H-1-benzopyran-4-one, $C_{21}H_{20}O_6$, $M_r = 368.4$, monoclinic, $P2_1/n$, $a = 10.494$ (1), $b = 15.347$ (2), $c = 11.456$ (2) Å, $\beta = 102.86$ (1)°, $Z = 4$, $V = 1798.7$ Å^3, $D_m = 1.33$, $D_x = 1.359$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.7107 Å, μ(Mo $K\alpha$) = 0.107 mm^{-1}, $F(000) = 776$, $T = 293$ K, $R = 0.062$ for 1345 observed reflections. The benzopyrone moiety is almost planar with the angle between the rings 2.3 (9)°. The crystal structure is stabilized by a network of hydrogen bonds. There are no unusual bond distances or angles.

Introduction. The plant *Millettia auriculata* has insecticidal and pesticidal properties (Chopra, Nayar & Chopra 1956). A number of isoflavonoids have been isolated from its roots, leaves and stems. The title compound, aurmillone, was isolated from the seeds of *Millettia auriculata* (supplied by B. N. Dwivedi Nainital, India). Needle-shaped crystals were obtained from benzene. Since the melting point (448–449 K) of this compound (1) differed widely from the earlier reported value of 430–431 K (Subbaraju & Srimannarayana, 1978) and was rather close to the melting

point reported for isoaurmillone (435–436 K) (Gupta, Bhattacharya, Mitra & Aditya-Chaudhury, 1983), it was decided to establish the structure unequivocally by X-ray diffraction.

(1)
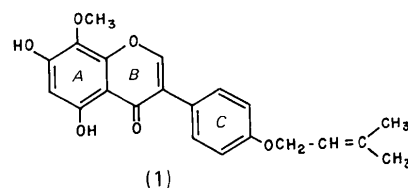
Experimental. Crystal approx. 0.45 × 0.40 × 0.35 mm. D_m by flotation in aqueous KI solution. Enraf–Nonius CAD-4F/11 M diffractometer, graphite-monochromated Mo $K\alpha$ radiation. Lattice parameters from 22 reflections (6 < θ < 15°). $\omega/2\theta$ scan mode, scan speed 1° min^{-1}, $\theta \le 24$°; h 0 to 12, k 0 to 18, l −13 to 13. 2616 reflections measured, 1345 judged significant ($|F_o| \ge 3\sigma|F_o|$). No correction for absorption. Three standard reflections every 2000 s; 4% variation in intensity. Structure solved by direct methods, program *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). Full-matrix least-squares refinement of scale factor, positional and

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anisotropic thermal parameters (isotropic thermal parameters only for H atoms, located from a difference map) converged to $R = 0.062$ and $wR = 0.060$. $\sum w(|F_o| - |F_c|)^2$ minimized; $w = (3.5 + 1.0|F_o| + 0.025 |F_o|^2)^{-1}$. Max. $\Delta/\sigma = 0.1$. Final $\Delta\rho$ excursions $< |0.3| e\text{Å}^{-3}$. No correction for secondary extinction. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). *LALS* (Gantzel, Sparks & Trueblood, 1961) for refinement.

Discussion. The atomic parameters with their e.s.d.'s and the equivalent isotropic temperature factors are given in Table 1.* Bond lengths and bond angles involving the non-hydrogen atoms are given in Table 2. Fig. 1 gives a perspective view of the molecule and the numbering of the atoms.

The benzopyrone ring system *A,B* is nearly planar, the angle between the rings being 2·3 (9)°. The torsion angles C(1)–C(2)–C(7)–O(1) and C(3)–C(2)–C(7)–C(6) are 3·1 (9) and 2·0 (9)° respectively. These values compare with those reported for the structure of 3-benzoyl-5,8-dihydroxyflavone (Schmalle, Jarchow, Hausen & Schulz, 1982), where the angle between rings *A* and *B* is 1·5°, and the corresponding torsion angles are 0·0 (1·1)° and −2·8 (1·0)°. The angle between the benzopyrone ring and the phenyl ring (*C*) is 58·9 (9)°. As is generally found in the structures of flavones substituted by a hydroxy group at C(3) (Fig. 1), the H of the −OH group at C(3) is hydrogen bonded to the carbonyl group O(2) (Schmalle *et al.*, 1982; Cantrell & Gerdom, 1979; Ting, Watson & Dominguez, 1972). In aurmillone, this hydrogen bond has parameters O(2)···O(3) 2·564 (7), O(2)···H 1·87 Å and O(3)–H···O(2) 131°. In addition, there is a stabilizing intermolecular hydrogen bond between O(2) and the symmetry-related O(4) at $(x-\frac{1}{2}, \frac{1}{2}-y, \frac{1}{2}+z)$ with parameters O(2)···O(4) 2·763 (8) Å and O(4)–H···O(2) 159°.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42596 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
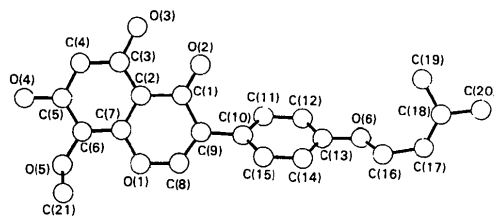

Fig. 1. A perspective view of the molecule.

Table 1. *Atomic coordinates* $(\times 10^4)$, *with e.s.d.'s in parentheses, and equivalent isotropic thermal parameters for non-hydrogen atoms*

$$B_{eq} = \tfrac{8}{3}\pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j.$$

	x	y	z	$B_{eq}(\text{Å}^2)$
O(1)	7038 (4)	1323 (3)	4341 (4)	3·70
O(2)	6403 (4)	2434 (3)	7387 (4)	4·11
O(3)	8394 (4)	3420 (3)	7475 (4)	4·35
O(4)	10875 (4)	2965 (3)	4584 (4)	4·10
O(5)	9235 (4)	1693 (3)	3492 (4)	4·00
O(6)	1359 (4)	363 (3)	7528 (4)	3·86
C(1)	6591 (6)	2091 (4)	6453 (5)	2·91
C(2)	7665 (5)	2353 (4)	5950 (4)	2·69
C(3)	8555 (6)	2996 (4)	6474 (5)	3·02
C(4)	9622 (6)	3202 (4)	6012 (5)	3·55
C(5)	9806 (6)	2769 (4)	5000 (5)	3·05
C(6)	8941 (6)	2141 (4)	4441 (5)	2·98
C(7)	7881 (6)	1947 (4)	4920 (5)	2·90
C(8)	5998 (6)	1109 (4)	4799 (5)	3·51
C(9)	5723 (5)	1429 (4)	5798 (4)	3·15
C(10)	4568 (5)	1127 (3)	6210 (4)	2·96
C(11)	4694 (5)	780 (4)	7360 (4)	3·41
C(12)	3598 (5)	531 (4)	7748 (4)	3·54
C(13)	2372 (5)	618 (3)	7041 (5)	3·21
C(14)	2219 (5)	950 (4)	5880 (4)	3·53
C(15)	3319 (5)	1191 (4)	5476 (5)	3·62
C(16)	50 (5)	551 (4)	6885 (5)	4·19
C(17)	−868 (5)	255 (4)	7655 (5)	3·80
C(18)	−1021 (5)	667 (4)	8640 (5)	3·91
C(19)	−337 (7)	1484 (5)	9101 (6)	6·23
C(20)	−1901 (6)	315 (5)	9374 (5)	4·77
C(21)	8400 (6)	1821 (5)	2358 (5)	4·81

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

O(1)–C(7)	1·371 (8)	C(5)–C(6)	1·381 (9)
O(1)–C(8)	1·352 (8)	C(6)–C(7)	1·377 (9)
O(2)–C(1)	1·247 (7)	C(8)–C(9)	1·335 (8)
O(3)–C(3)	1·361 (7)	C(9)–C(10)	1·470 (8)
O(4)–C(5)	1·347 (8)	C(10)–C(11)	1·399 (7)
O(5)–C(6)	1·377 (7)	C(10)–C(15)	1·394 (7)
O(5)–C(21)	1·411 (7)	C(11)–C(12)	1·376 (8)
O(6)–C(13)	1·362 (7)	C(12)–C(13)	1·366 (8)
O(6)–C(16)	1·436 (7)	C(13)–C(14)	1·400 (7)
C(1)–C(2)	1·433 (8)	C(14)–C(15)	1·387 (8)
C(1)–C(9)	1·457 (8)	C(16)–C(17)	1·513 (8)
C(2)–C(3)	1·399 (8)	C(17)–C(18)	1·334 (8)
C(2)–C(7)	1·396 (8)	C(18)–C(19)	1·482 (9)
C(3)–C(4)	1·378 (9)	C(18)–C(20)	1·482 (9)
C(4)–C(5)	1·386 (8)		
C(7)–O(1)–C(8)	118·2 (5)	C(2)–C(7)–C(6)	122·6 (6)
C(6)–O(5)–C(21)	117·1 (5)	O(1)–C(8)–C(9)	126·2 (5)
C(13)–O(6)–C(16)	118·5 (4)	C(1)–C(9)–C(8)	118·0 (5)
O(2)–C(1)–C(2)	121·1 (5)	C(1)–C(9)–C(10)	121·3 (5)
O(2)–C(1)–C(9)	122·4 (5)	C(8)–C(9)–C(10)	120·7 (5)
C(2)–C(1)–C(9)	116·5 (5)	C(9)–C(10)–C(11)	120·7 (5)
C(1)–C(2)–C(3)	122·3 (5)	C(9)–C(10)–C(15)	121·0 (5)
C(1)–C(2)–C(7)	120·4 (5)	C(11)–C(10)–C(15)	118·3 (5)
C(3)–C(2)–C(7)	117·3 (5)	C(10)–C(11)–C(12)	120·0 (5)
O(3)–C(3)–C(2)	120·2 (5)	C(11)–C(12)–C(13)	121·7 (5)
O(3)–C(3)–C(4)	118·5 (5)	O(6)–C(13)–C(12)	116·6 (5)
C(2)–C(3)–C(4)	121·2 (5)	O(6)–C(13)–C(14)	123·9 (5)
C(3)–C(4)–C(5)	119·2 (6)	C(12)–C(13)–C(14)	119·5 (5)
O(4)–C(5)–C(4)	118·1 (5)	C(13)–C(14)–C(15)	119·1 (5)
O(4)–C(5)–C(6)	120·3 (5)	C(10)–C(15)–C(14)	121·3 (5)
C(4)–C(5)–C(6)	121·6 (6)	O(6)–C(16)–C(17)	107·6 (5)
O(5)–C(6)–C(5)	118·4 (5)	C(16)–C(17)–C(18)	124·2 (5)
O(5)–C(6)–C(7)	123·3 (5)	C(17)–C(18)–C(19)	124·2 (6)
C(5)–C(6)–C(7)	118·1 (6)	C(17)–C(18)–C(20)	121·0 (5)
O(1)–C(7)–C(2)	120·7 (5)	C(19)–C(18)–C(20)	114·8 (5)
O(1)–C(7)–C(6)	116·7 (5)		

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Structure of L-Leucine: a Redetermination

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Abstract. $C_6H_{13}NO_2$, $M_r = 131.2$, monoclinic, $P2_1$, $a = 14.666$ (2), $b = 5.324$ (7), $c = 9.606$ (3) Å, $\beta = 94.06$ (3)°, $V = 748$ (1) Å^3, $Z = 4$, $D_x = 1.16$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.087$ mm^{-1}, $F(000) = 288$, room temperature, $R = 0.058$ for 686 observed reflections. The crystals studied and the conformation obtained are similar to those reported by Harding & Howieson [*Acta Cryst.* (1976), B32, 633–634], but the present structure is more accurately determined. The crystal studied and the conformation obtained are similar to those reported by Harding & Howieson but the present structure is more accurately determined, and the hydrogen-bonding scheme is clearly defined. The crystal structure contains hydrophobic layers intercalated with hydrophilic ones.

Introduction. The zwitterionic structure of L-Leu has been described by Harding & Howieson (1976). These authors refined the structure to $R = 0.13$. They reported that the atom positions had not been accurately determined because of the poor quality of their crystals. Here we present a redetermination of the structure.

Experimental. Thin colorless plates obtained by vapor diffusion of acetone into acetone–aqueous solution of amino acid. Crystal $0.05 \times 0.7 \times 1.5$ mm mounted in capillary. Philips PW 1100 diffractometer, Mo $K\alpha$ radiation, graphite monochromator. h, k, l range $= \pm 14$, 6, 11. Cell parameters from 25 reflections $(4 \le \theta \le 9°)$, ω-scan technique. Three reflections measured every 2 h as intensity control, no significant differences. 1170 independent reflections $(\theta \le 25°)$, 686 with $I > 2.5\sigma(I)$; Lp correction, absorption ignored. Structure determined by direct methods (*MULTAN*80; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Anisotropic full-matrix least-squares refinement on F with *SHELX*76 (Sheldrick, 1976). f, f'