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References

FLIPPEN-ANDERSON, J. L., GILARDI, R., GEORGE, C., MARCHAND, A. P. & DAVE, P. R. (1989). *Acta Cryst.* C**45**, 1171–1174.

International Tables for X-ray Crystallography (1974). Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

MARCHAND, A. P., EARLYWINE, A. D. & HEEG, M. J. (1986). *J. Org. Chem.* **51**, 4096–4100.

MARCHAND, A. P., GOODIN, D. B., HOSSAIN, M. R. & VAN DER HELM, D. (1984). *J. Org. Chem.* **49**, 2897–2900.

MARCHAND, A. P. & HAYES, B. R. (1977). *Tetrahedron Lett.* pp. 1027–1028.

Nicolet Instrument Corporation (1986). *SHELXTL* for Desktop 30 (Microeclipse), PN-269-104340, April, 1986. Nicolet Instrument Corp., Madison, Wisconsin, USA.

WATSON, W. H., NAGL, A., MARCHAND, A. P. & CHENERA, B. (1988). *Acta Cryst.* C**44**, 806–808.

Acta Cryst. (1990). C**46**, 1129–1131

Structures of (4*R*)-*cis*-4,5-Dihydro-2-methyl-4,5-diphenyl-6*H*-cyclopenta[*b*]furan-6-one and *N*,*N*-Dimethyl-3,4-diphenyl-2,2′-bifuran-5-amine

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Abstract. (I) $C_{20}H_{16}O_2$, $M_r = 288{\cdot}35$, orthorhombic, $P2_12_12_1$, $a = 8{\cdot}665$ (1), $b = 8{\cdot}808$ (1). $c = 19{\cdot}766$ (1) Å, $V = 1508{\cdot}6$ (1) Å^3, $Z = 4$, $D_x = 1{\cdot}27$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 5{\cdot}6$ cm^{-1}, $F(000) = 608$, $T = 123$ K, $R = 0{\cdot}056$ for 1181 unique observed reflections. (II) $C_{22}H_{19}NO_2$, $M_r = 329{\cdot}40$, monoclinic, $P2_1/c$, $a = 13{\cdot}076$ (4), $b = 8{\cdot}203$ (3), $c = 19{\cdot}605$ (2) Å, $\beta = 124{\cdot}95$ (3)°, $V = 1723{\cdot}6$ (1) Å^3, $Z = 4$, $D_x = 1{\cdot}27$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 5{\cdot}7$ cm^{-1}, $F(000) = 696$, $T = 123$ K, $R = 0{\cdot}055$ for 2038 unique observed reflections. The C(4)—C(5) bond distance in structure (I) is unusually long at 1·59 Å. The two furan rings in structure (II) are coplanar; the dihedral angle between the planes is 1·9°.

Experimental. A clear, thin plate of dimensions 0·03 × 0·13 × 0·26 mm (I), and a clear, chunky plate of dimensions 0·10 × 0·08 × 0·05 mm (II) were used for intensity measurements on Syntex $P2_1$ (I) and $P1$ (II) diffractometers controlled by a Harris computer. Intensity measurements were made using graphite-monochromatized Cu $K\alpha$ radiation. The $\theta/2\theta$ step-scan technique was used with a scan speed of 2° min^{-1} and a scan width over 3·4° to $2\theta_{max} = 138°$. Ten reflections monitored periodically showed no loss of intensity during the data collection. Of the 1626 (I), 2892 (II) unique reflections measured, 1181 (I), 2038 (II) had intensities greater than $3\sigma(I)$. Standard deviations in the intensities were approximated by the equation: $\sigma^2(I) = \sigma^2(I)_{\text{counting statistics}} + (DI)^2$ where the coefficient [$D = 0{\cdot}0163$ (I), 0·0128 (II)] of I was calculated from the variations in intensities of the monitored reflections. Range of *hkl*: h 0→10, k 0→10, l 0→22 (I), h −15→12, k 0→9, l 0→18 (II). Unit-cell parameters were determined accurately by least-squares fit of Cu $K\alpha_1$ 2θ values [λ(Cu $K\alpha_1$) = 1·5406 Å] for 25 high 2θ reflections ($120 < 2\theta < 138°$) (Duchamp, 1977). Lorentz and polarization corrections appropriate for a monochromator with 50% perfect character were applied but no correction for absorption was made. The structures were solved by direct methods, using *RANTAN*81 (Yao Jia-Xing, 1981) (I) and *DIREC* (Duchamp, 1984*a*) (II). H atoms found in difference maps were very close to positions generated using planar or tetrahedral geometry, so generated positions were used. The structure was refined by full-matrix least squares with all the coordinates and anisotropic thermal parameters for non-H atoms included in the refinement. Hydrogen thermal parameters were included in the calculations but not refined. The H atoms, with assigned temperature factors 0·5 Å^2 higher than the equivalent average isotropic values of the atoms of attachment, were included but only their positions were varied in the refinement. The function minimized in the refinement was $\sum w(F_o^2 - F_c^2)^2$, where weights w were $1/\sigma^2(F_o^2)$. Atomic form factors were from Doyle & Turner (1968), except for hydrogen which was from Stewart, Davidson & Simpson (1965). In the final cycle, Δ/σ was <0·5. The final R, wR were 0·056, 0·081 (I) and

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Table 1. *Fractional atomic coordinates and B_{eq} values ($Å^2$) with e.s.d.'s in parentheses*

$$B_{eq} = (4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

	x	y	z	B_{eq}
(I)				
O(1)	0·2567 (3)	0·2016 (3)	0·0687 (1)	2·0 (1)
C(2)	0·1454 (4)	0·0904 (4)	0·0581 (2)	1·9 (1)
C(2*M*)	0·0786 (5)	0·0849 (5)	−0·0106 (2)	2·7 (2)
C(3)	0·1193 (4)	0·0091 (4)	0·1154 (2)	2·0 (2)
C(3*A*)	0·2185 (4)	0·0699 (4)	0·1652 (2)	1·6 (1)
C(4)	0·2651 (4)	0·0481 (4)	0·2382 (2)	1·7 (1)
C(5)	0·3953 (4)	0·1744 (4)	0·2466 (2)	1·6 (1)
C(6)	0·4032 (4)	0·2621 (4)	0·1787 (2)	1·8 (1)
O(6)	0·4864 (3)	0·3709 (3)	0·1676 (1)	2·4 (1)
C(6*A*)	0·2967 (4)	0·1844 (4)	0·1357 (1)	1·7 (1)
C(1′)	0·1306 (4)	0·0543 (4)	0·2875 (1)	1·6 (1)
C(2′)	0·1356 (4)	−0·0354 (4)	0·3451 (2)	2·1 (2)
C(3′)	0·0145 (5)	−0·0347 (5)	0·3910 (2)	2·6 (2)
C(4′)	−0·1137 (5)	0·0560 (5)	0·3792 (2)	2·6 (2)
C(5′)	−0·1185 (4)	0·1465 (4)	0·3226 (2)	2·3 (2)
C(6′)	0·0037 (4)	0·1468 (4)	0·2767 (2)	1·8 (1)
C(1″)	0·3825 (4)	0·2783 (4)	0·3075 (2)	1·6 (1)
C(2″)	0·2926 (4)	0·4085 (4)	0·3062 (2)	2·0 (1)
C(3″)	0·2835 (5)	0·5027 (4)	0·3624 (2)	2·5 (2)
C(4″)	0·3637 (5)	0·4672 (4)	0·4209 (2)	2·8 (2)
C(5″)	0·4519 (5)	0·3364 (5)	0·4232 (2)	2·9 (2)
C(6″)	0·4614 (4)	0·2429 (4)	0·3671 (2)	2·1 (2)
(II)				
O(1)	−0·3438 (1)	−0·0622 (1)	0·1884 (1)	1·98 (4)
C(5)	−0·3589 (2)	−0·1525 (2)	0·1239 (1)	2·04 (6)
N	−0·4272 (1)	−0·2956 (2)	0·1043 (1)	2·17 (6)
C(*M*1)	−0·4854 (2)	−0·3246 (3)	0·1480 (1)	3·18 (8)
C(*M*2)	−0·3640 (2)	−0·4399 (3)	0·1010 (1)	3·13 (8)
C(4)	−0·2976 (2)	−0·0814 (2)	0·0941 (1)	1·91 (6)
C(1*A*)	−0·2897 (2)	−0·1387 (2)	0·0259 (1)	1·98 (6)
C(2*A*)	−0·3967 (2)	−0·1746 (2)	−0·0527 (1)	2·35 (7)
C(3*A*)	−0·3874 (2)	−0·2287 (3)	−0·1158 (1)	2·81 (7)
C(4*A*)	−0·2721 (2)	−0·2474 (2)	−0·1023 (1)	2·72 (7)
C(5*A*)	−0·1646 (2)	−0·2111 (2)	−0·0245 (1)	2·55 (7)
C(6*A*)	−0·1737 (2)	−0·1575 (2)	0·0390 (1)	2·28 (7)
C(3)	−0·2381 (2)	0·0623 (2)	0·1443 (1)	1·86 (6)
C(1*B*)	−0·1609 (2)	0·1778 (2)	0·1346 (1)	1·88 (6)
C(2*B*)	−0·0330 (2)	0·1914 (2)	0·1952 (1)	2·51 (7)
C(3*B*)	0·0386 (2)	0·2978 (3)	0·1838 (1)	2·94 (8)
C(4*B*)	−0·0160 (2)	0·3918 (3)	0·1128 (1)	3·06 (8)
C(5*B*)	−0·1423 (2)	0·3782 (3)	0·0524 (1)	3·13 (8)
C(6*B*)	−0·2144 (2)	0·2710 (2)	0·0626 (1)	2·52 (7)
C(2)	−0·2696 (2)	0·0705 (2)	0·1990 (1)	1·97 (6)
C(2′)	−0·2466 (2)	0·1815 (2)	0·2636 (1)	1·99 (6)
O(2′)	−0·1742 (1)	0·3156 (2)	0·2768 (1)	2·46 (5)
C(1′)	−0·2814 (2)	0·1833 (2)	0·3160 (1)	2·58 (7)
C(4′)	−0·2300 (2)	0·3271 (3)	0·3651 (1)	2·73 (7)
C(3′)	−0·1671 (2)	0·4025 (2)	0·3396 (1)	2·66 (7)

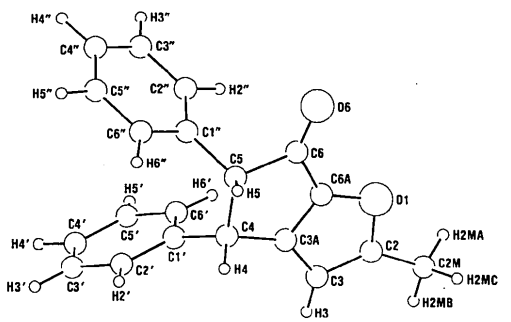

Fig. 1. Structure (I) with atom numbering.

0·055, 0·074 (II), number of parameters was 247 (I) and 283 (II), and S was 1·32 (I) and 1·76 (II). Final $\Delta\rho < 0·38$ e $Å^{-3}$ for either structure. The *CRYM* system of computer programs was used (Duchamp, 1984*b*). Final atomic coordinates can be found in Table 1.* All bond distances and angles are within the expected ranges (Table 2). Figs. 1 and 2 show the atomic numbering of the respective structures.

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

(I)

O(1)—C(2)	1·390 (4)	C(1′)—C(2′)	1·387 (5)
O(1)—C(6*A*)	1·377 (4)	C(1′)—C(6′)	1·385 (5)
C(2)—C(2*M*)	1·477 (5)	C(2′)—C(3′)	1·388 (5)
C(2)—C(3)	1·358 (5)	C(3′)—C(4′)	1·387 (6)
C(3)—C(3*A*)	1·412 (5)	C(4′)—C(5′)	1·376 (5)
C(3*A*)—C(4)	1·512 (4)	C(5′)—C(6′)	1·394 (5)
C(3*A*)—C(6*A*)	1·347 (5)	C(1″)—C(2″)	1·386 (5)
C(4)—C(5)	1·593 (5)	C(1″)—C(6″)	1·397 (5)
C(4)—C(1′)	1·520 (5)	C(2″)—C(3″)	1·388 (5)
C(5)—C(6)	1·551 (5)	C(3″)—C(4″)	1·385 (5)
C(5)—C(1″)	1·516 (4)	C(4″)—C(5″)	1·383 (6)
C(6)—O(6)	1·218 (4)	C(5″)—C(6″)	1·385 (5)
C(6)—C(6*A*)	1·429 (5)		

C(2)—O(1)—C(6*A*)	104·0 (2)	O(1)—C(6*A*)—C(6)	133·0 (3)
O(1)—C(2)—C(2*M*)	115·6 (3)	C(3*A*)—C(6*A*)—C(6)	115·2 (3)
O(1)—C(2)—C(3)	111·2 (3)	C(4)—C(1′)—C(2′)	118·7 (3)
C(2*M*)—C(2)—C(3)	133·1 (3)	C(4)—C(1′)—C(6′)	122·1 (3)
C(2)—C(3)—C(3*A*)	106·2 (3)	C(2′)—C(1′)—C(6′)	119·1 (3)
C(3)—C(3*A*)—C(4)	141·2 (3)	C(1′)—C(2′)—C(3′)	120·7 (3)
C(3)—C(3*A*)—C(6*A*)	106·8 (3)	C(2′)—C(3′)—C(4′)	119·9 (3)
C(4)—C(3*A*)—C(6*A*)	112·0 (3)	C(3′)—C(4′)—C(5′)	119·6 (4)
C(3*A*)—C(4)—C(5)	101·5 (2)	C(4′)—C(5′)—C(6′)	120·5 (3)
C(3*A*)—C(4)—C(1′)	113·7 (3)	C(1′)—C(6′)—C(5′)	120·1 (3)
C(5)—C(4)—C(1′)	116·8 (3)	C(5)—C(1″)—C(2″)	121·8 (3)
C(4)—C(5)—C(6)	106·8 (3)	C(5)—C(1″)—C(6″)	119·9 (3)
C(4)—C(5)—C(1″)	116·9 (3)	C(2″)—C(1″)—C(6″)	118·4 (3)
C(6)—C(5)—C(1″)	112·9 (3)	C(1″)—C(2″)—C(3″)	120·8 (3)
C(5)—C(6)—O(6)	125·0 (3)	C(2″)—C(3″)—C(4″)	120·3 (4)
C(5)—C(6)—C(6*A*)	104·3 (3)	C(3″)—C(4″)—C(5″)	119·5 (3)
O(6)—C(6)—C(6*A*)	130·7 (3)	C(4″)—C(5″)—C(6″)	120·1 (3)
O(1)—C(6*A*)—C(3*A*)	111·8 (3)	C(1″)—C(6″)—C(5″)	120·9 (3)

(II)

O(1)—C(5)	1·380 (2)	C(3)—C(1*B*)	1·474 (2)
O(1)—C(2)	1·393 (2)	C(3)—C(2)	1·353 (2)
C(5)—N	1·389 (2)	C(1*B*)—C(2*B*)	1·395 (2)
C(5)—C(4)	1·363 (2)	C(1*B*)—C(6*B*)	1·391 (3)
N—C(*M*1)	1·454 (2)	C(2*B*)—C(3*B*)	1·388 (2)
N—C(*M*2)	1·466 (2)	C(3*B*)—C(4*B*)	1·380 (3)
C(4)—C(1*A*)	1·477 (2)	C(4*B*)—C(5*B*)	1·379 (3)
C(4)—C(3)	1·444 (3)	C(5*B*)—C(6*B*)	1·386 (2)
C(1*A*)—C(2*A*)	1·395 (2)	C(2)—C(2′)	1·443 (2)
C(1*A*)—C(6*A*)	1·393 (1)	C(2′)—O(2′)	1·375 (2)
C(2*A*)—C(3*A*)	1·384 (2)	C(2′)—C(1′)	1·342 (2)
C(3*A*)—C(4*A*)	1·382 (2)	O(2′)—C(3′)	1·379 (2)
C(4*A*)—C(5*A*)	1·391 (3)	C(1′)—C(4′)	1·424 (3)
C(5*A*)—C(6*A*)	1·387 (2)	C(4′)—C(3′)	1·334 (2)

C(5)—O(1)—C(2)	106·1 (1)	C(1*B*)—C(3)—C(2)	127·7 (2)
O(1)—C(5)—N	116·0 (1)	C(3)—C(1*B*)—C(2*B*)	121·1 (2)
O(1)—C(5)—C(4)	110·6 (1)	C(3)—C(1*B*)—C(6*B*)	120·1 (1)
N—C(5)—C(4)	133·3 (2)	C(2*B*)—C(1*B*)—C(6*B*)	118·7 (1)
C(5)—N—C(*M*1)	116·5 (1)	C(1*B*)—C(2*B*)—C(3*B*)	120·2 (2)
C(5)—N—C(*M*2)	113·9 (1)	C(2*B*)—C(3*B*)—C(4*B*)	120·5 (2)
C(*M*1)—N—C(*M*2)	113·1 (2)	C(3*B*)—C(4*B*)—C(5*B*)	119·6 (2)
C(5)—C(4)—C(1*A*)	128·0 (2)	C(4*B*)—C(5*B*)—C(6*B*)	120·5 (2)
C(5)—C(4)—C(3)	106·1 (1)	C(1*B*)—C(6*B*)—C(5*B*)	120·5 (1)
C(1*A*)—C(4)—C(3)	126·0 (1)	O(1)—C(2)—C(3)	110·2 (1)
C(4)—C(1*A*)—C(2*A*)	121·3 (1)	O(1)—C(2)—C(2′)	113·7 (1)
C(4)—C(1*A*)—C(6*A*)	120·1 (1)	C(3)—C(2)—C(2′)	136·1 (1)
C(2*A*)—C(1*A*)—C(6*A*)	118·5 (1)	C(2)—C(2′)—O(2′)	116·5 (1)
C(1*A*)—C(2*A*)—C(3*A*)	120·5 (1)	C(2)—C(2′)—C(1′)	133·4 (2)
C(2*A*)—C(3*A*)—C(4*A*)	120·6 (2)	O(2′)—C(2′)—C(1′)	110·2 (2)
C(3*A*)—C(4*A*)—C(5*A*)	119·5 (2)	C(2′)—O(2′)—C(3′)	105·7 (1)
C(4*A*)—C(5*A*)—C(6*A*)	120·0 (1)	C(2′)—C(1′)—C(4′)	106·9 (1)
C(1*A*)—C(6*A*)—C(5*A*)	120·9 (2)	C(1′)—C(4′)—C(3′)	106·6 (2)
C(4)—C(3)—C(1*B*)	125·3 (1)	O(2′)—C(3′)—C(4′)	110·7 (2)
C(4)—C(3)—C(2)	106·9 (1)		

* Lists of structure factors, anisotropic thermal parameters, hydrogen coordinates, and C—H distances have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52603 (31 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

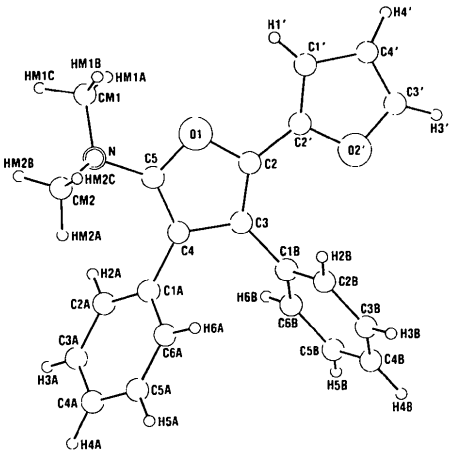

Fig. 2. Structure (II) with atom numbering.

Related literature. Details of the syntheses, reactions, and mechanisms are given by Yamashita, Toy, Watt & Muchmore (1988). The only other related compound reported that has a long C—C distance (1·58 Å) is a tetracyclone–dimethyl phosphonate adduct (Iball, Kaye & Miller, 1974). Another bifuran structure was published by Still & Romero (1986).

References

DOYLE, P. A. & TURNER, P. S. (1968). *Acta Cryst.* A**24**, 390–397.

DUCHAMP, D. J. (1977). *ACS Symp. Ser.* **46**, 98–121.

DUCHAMP, D. J. (1984*a*). *DIRECT. A Direct-Methods Program.* The Upjohn Company, Kalamazoo, MI, USA.

DUCHAMP, D. J. (1984*b*). *CRYM. A System of Crystallographic Programs.* The Upjohn Company, Kalamazoo, MI, USA.

IBALL, J., KAYE, P. & MILLER, J. A. (1974). *J. Chem. Soc. Perkin Trans.* 2, pp. 650–653.

STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

STILL, W. C. & ROMERO, A. G. (1986). *J. Am. Chem. Soc.* **108**, 2105–2106.

YAMASHITA, A., TOY, A., WATT, W. & MUCHMORE, C. R. (1988). *Tetrahedron Lett.* **28**, 3403–3406.

YAO JIA-XING (1981). *RANTAN*81. *A Computer Program for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univ. of York, England.

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6-Methoxy-2-(2,3,4-trimethoxyphenyl)-4*H*-1-benzopyran-4-one (6,2′,3′,4′-Tetramethoxyflavone)

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Abstract. $C_{19}H_{18}O_6$, $M_r = 342{\cdot}35$, monoclinic, $P2_1/a$, $a = 22{\cdot}341$ (9), $b = 16{\cdot}489$ (9), $c = 4{\cdot}477$ (1) Å, $\beta = 91{\cdot}35$ (3)°, $V = 1649$ (1) Å^3, $Z = 4$, $D_x = 1{\cdot}38$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}54178$ Å, $\mu = 8{\cdot}71$ cm^{-1}, $F(000) = 720$, $T = 291$ K, $R = 0{\cdot}064$ for 2560 observed reflections. The three rings are planar within experimental error (maximum deviation from mean planes less than 0·02 Å). The dihedral angle between the mean planes of rings *A* (O1—C2—C3—C4—C9—C10) and *B* (C5—C6—C7—C8—C9—C10) is 2 (1)°, and that between rings *A* and *C* (C1′—C2′—C3′—C4′—C5′—C6′) is 45 (1)°. The two methoxy groups bound to C2′ and C3′ have an out-of-plane conformation due to steric inhibition: C3′—C2′—O14—C15 = −108·7 (6), C4′—C3′—O16—C17 = −83·2 (6)°. The two other methoxy groups are only slightly out of plane with C5′—C4′—O18—C19 = 6·6 (6) and C7—C6—O12—C13 = 12·2 (6)°.

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