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2,3-Dihydro-6,7-dimethyl-2-phenyl-4*H*-1-benzopyran-4-one

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Abstract

In the title molecule, $C_{17}H_{16}O_2$, the dihydropyran ring adopts a sofa conformation. The benzopyran ring is distorted from planarity and the phenyl ring forms a dihedral angle of 53.86 (6)° with the dihydropyran ring. In the crystal the benzopyran rings are stacked along the a direction with a repeat distance of 3.814 (3) Å.

Comment

The present work is part of a project concerned with the synthesis of flavanone analogues for antimalarial compounds. The title compound is one example to be investigated and the knowledge of the three dimensional structure of the molecule could be useful for the understanding of the synthesis and for predicting the conformation of other flavanone analogs.

The bond lengths and bond angles observed in the structure are comparable with those observed in the related compound 2,3-dihydro-5-hydroxy-6,7-dimethoxy- 2-phenyl-4*H*-1-benzopyran-4-one (Chantrapromma *et al.*, 1997). The dihydropyran ring adopts a sofa conformation with the asymmetry parameter $\Delta C_s(C2) = 0.032$ (1) (Nardelli, 1995); the deviation of C2 from the O1—C9—C10—C4—C3 plane is −0.650 (2) Å. The pyran ring is distorted from coplanarity with the benzene ring and they form a dihedral angle of 7.54 (5)°. The dihedral angle between the dihydropyran and phenyl rings is 53.86 (6)°. In the crystal the benzopyran rings are stacked along the 'a' direction with a repeat distance of 3.814 (3) Å. The carbonyl oxygen is involved in a short C—H···O intermolecular contact (Table 3).

Experimental

Condensation of 4,5-dimethyl-2-hydroxyacetophenone with benzaldehyde by a known procedure (Jain, Lal & Seshadri, 1968) furnished 4',5'-dimethyl-2'-hydroxychalcone. Cyclization of the corresponding chalcone with 5% H_2SO_4 in ethanol, at 70°C for 1 h, gave the title compound, after recrystallization with methanol, as colourless crystals, m.p. 81–82°C.

Refinement

The structure was solved by direct methods. All H-atoms were located from a difference Fourier map and refined isotropically.

Computing details

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS* (Siemens, 1994); data reduction: *XSCANS* (Siemens, 1994); program(s) used to solve structure: *SHELXTL*PC (Sheldrick, 1990); program(s) used to refine structure: *SHELXL93*

(Sheldrick, 1993); molecular graphics: *SHELXTL*PC (Sheldrick, 1990); software used to prepare material for publication: *SHELXL93* (Sheldrick, 1993) *PARST* (Nardelli, 1995).

2,3-Dihydro-6,7-dimethyl-2-phenyl-4*H*-1-benzopyran-4-one

Crystal data

$C_{17}H_{16}O_2$
$M_r = 252.31$
Triclinic, $P\bar{1}$
$a = 5.057\ (2)$ Å
$b = 11.046\ (2)$ Å
$c = 12.531\ (2)$ Å
$\alpha = 88.525\ (14)°$
$\beta = 87.03\ (2)°$
$\gamma = 79.29\ (2)°$
$V = 686.8\ (3)$ Å^3
$Z = 2$
Mo $K\alpha$
$\mu = 0.08$ mm^{-1}
$T = 293\ (2)$ K
0.66 × 0.38 × 0.30 mm

Data collection

Siemens P4 diffractometer
Absorption correction: none
4223 measured reflections
3165 independent reflections
1960 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.022$
3 standard reflections
every 97 reflections
intensity decay: <3%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.166$
$S = 1.02$
3165 reflections
237 parameters
All H-atom parameters refined
$\Delta\rho_{\rm max} = 0.17$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.21$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °)

O1—C9	1.371 (2)	C3—C4	1.506 (2)
O1—C2	1.449 (2)	C4—O11	1.217 (2)
C2—C3	1.523 (2)	C4—C10	1.473 (2)
C9—O1—C2—C3	−54.4 (2)	C2—O1—C9—C10	24.1 (2)
O1—C2—C3—C4	56.5 (2)	O1—C9—C10—C4	5.2 (2)
C2—C3—C4—C10	−28.9 (2)	C3—C4—C10—C9	−1.5 (2)

Table 2
Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O11$^{\rm i}$	0.98 (2)	2.59 (2)	3.314 (2)	131 (1)

Symmetry codes: (i) $-x, -y, -z+1$.

Acknowledgements

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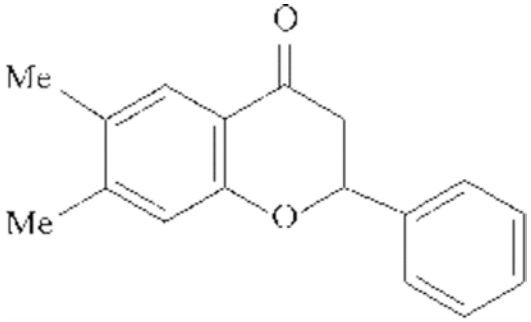
Scheme 1



supplementary materials

2,3-Dihydro-6,7-dimethyl-2-phenyl-4*H*-1-benzopyran-4-one

Crystal data

$C_{17}H_{16}O_2$
$M_r = 252.31$
Triclinic, $P\overline{1}$
$a = 5.057$ (2) Å
$b = 11.046$ (2) Å
$c = 12.531$ (2) Å
$\alpha = 88.525$ (14)°
$\beta = 87.03$ (2)°
$\gamma = 79.29$ (2)°
$V = 686.8$ (3) $Å^3$

$Z = 2$
$F_{000} = 268$
$D_x = 1.220$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 40 reflections
$\theta = 6.1–12.5°$
$\mu = 0.08$ mm^{-1}
$T = 293$ (2) K
Parallelepiped, colourless
0.66 × 0.38 × 0.30 mm

Data collection

Siemens P4 diffractometer
Radiation source: fine-focus sealed tube
Monochromator: graphite
$T = 293(2)$ K
$\theta/2\theta$ scans
Absorption correction: none
4223 measured reflections
3165 independent reflections
1960 reflections with $I > 2\sigma(I)$

$R_{int} = 0.022$
$\theta_{max} = 27.5°$
$\theta_{min} = 1.9°$
$h = -1\rightarrow6$
$k = -14\rightarrow14$
$l = -16\rightarrow16$
3 standard reflections
every 97 reflections
intensity decay: <3%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.166$
$S = 1.02$
3165 reflections
237 parameters
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
Calculated $w = 1/[\sigma^2(F_o^2) + (0.0965P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$?
$\Delta\rho_{max} = 0.17$ e $Å^{-3}$
$\Delta\rho_{min} = -0.21$ e $Å^{-3}$
Extinction correction: SHELXL, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0103 (66)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement on F^2 for ALL reflections. Weighted *R*-factors *wR* and all goodnesses of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The observed criterion of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating observed *R*-factor *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and *R*-factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	$U_{\rm iso}$*/$U_{\rm eq}$
O1	0.0845 (2)	0.08285 (10)	0.20093 (8)	0.0537 (3)
C2	0.1899 (3)	−0.01638 (14)	0.27374 (12)	0.0473 (4)
C3	0.2967 (4)	0.0361 (2)	0.37056 (13)	0.0533 (4)
C4	0.0831 (3)	0.1300 (2)	0.42595 (13)	0.0530 (4)
C5	−0.3296 (4)	0.2895 (2)	0.3951 (2)	0.0579 (5)
C6	−0.5122 (4)	0.3587 (2)	0.3300 (2)	0.0611 (5)
C7	−0.4884 (4)	0.3364 (2)	0.2193 (2)	0.0604 (5)
C8	−0.2862 (4)	0.2439 (2)	0.17969 (15)	0.0567 (4)
C9	−0.1058 (3)	0.17395 (14)	0.24670 (12)	0.0475 (4)
C10	−0.1218 (3)	0.19699 (14)	0.35579 (12)	0.0489 (4)
O11	0.0854 (3)	0.15097 (13)	0.52068 (10)	0.0742 (4)
C12	0.4076 (3)	−0.10495 (15)	0.21576 (12)	0.0510 (4)
C13	0.5606 (4)	−0.0687 (2)	0.1309 (2)	0.0684 (5)
C14	0.7739 (5)	−0.1507 (2)	0.0837 (2)	0.0845 (7)
C15	0.8318 (5)	−0.2701 (2)	0.1213 (2)	0.0850 (7)
C16	0.6799 (6)	−0.3069 (2)	0.2058 (2)	0.0927 (8)
C17	0.4703 (5)	−0.2253 (2)	0.2529 (2)	0.0784 (6)
C18	−0.7370 (5)	0.4552 (2)	0.3778 (3)	0.0869 (8)
C19	−0.6798 (6)	0.4104 (2)	0.1431 (3)	0.0872 (8)
H2	0.038 (3)	−0.0557 (14)	0.2968 (12)	0.043 (4)*
H3A	0.453 (4)	0.0787 (16)	0.3476 (14)	0.060 (5)*
H3B	0.364 (4)	−0.0329 (19)	0.4164 (17)	0.069 (5)*
H5	−0.343 (4)	0.3012 (19)	0.4680 (19)	0.074 (6)*
H8	−0.262 (4)	0.2262 (18)	0.1041 (17)	0.070 (6)*
H13	0.509 (5)	0.016 (2)	0.1053 (18)	0.088 (7)*
H14	0.894 (5)	−0.118 (3)	0.024 (2)	0.113 (9)*
H15	0.977 (6)	−0.320 (3)	0.089 (2)	0.105 (8)*
H16	0.737 (6)	−0.391 (3)	0.237 (2)	0.125 (10)*
H17	0.354 (5)	−0.245 (2)	0.308 (2)	0.090 (8)*
H18A	−0.723 (7)	0.540 (3)	0.354 (3)	0.139 (11)*
H19A	−0.859 (7)	0.402 (3)	0.156 (3)	0.132 (12)*
H19B	−0.669 (6)	0.490 (3)	0.145 (2)	0.114 (10)*
H19C	−0.616 (6)	0.387 (3)	0.061 (3)	0.126 (10)*
H18B	−0.918 (6)	0.439 (3)	0.357 (2)	0.110 (8)*
H18C	−0.740 (9)	0.457 (4)	0.453 (4)	0.186 (18)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0555 (7)	0.0558 (6)	0.0421 (6)	0.0086 (5)	0.0011 (5)	0.0015 (5)
C2	0.0477 (8)	0.0479 (8)	0.0441 (8)	−0.0035 (7)	−0.0033 (7)	0.0039 (6)
C3	0.0504 (9)	0.0595 (10)	0.0503 (9)	−0.0093 (8)	−0.0085 (7)	0.0007 (7)
C4	0.0579 (10)	0.0578 (9)	0.0456 (8)	−0.0174 (8)	0.0022 (7)	−0.0037 (7)
C5	0.0595 (10)	0.0536 (9)	0.0607 (10)	−0.0123 (8)	0.0084 (9)	−0.0144 (8)
C6	0.0505 (9)	0.0480 (9)	0.0835 (12)	−0.0067 (8)	0.0077 (9)	−0.0129 (9)
C7	0.0506 (10)	0.0472 (9)	0.0806 (12)	−0.0013 (8)	−0.0057 (9)	−0.0005 (8)
C8	0.0571 (10)	0.0539 (9)	0.0554 (9)	−0.0006 (8)	−0.0028 (8)	0.0006 (8)
C9	0.0442 (8)	0.0471 (8)	0.0491 (8)	−0.0046 (7)	0.0039 (7)	−0.0013 (7)
C10	0.0473 (8)	0.0486 (8)	0.0512 (8)	−0.0109 (7)	0.0055 (7)	−0.0050 (7)
O11	0.0884 (10)	0.0875 (9)	0.0448 (6)	−0.0104 (8)	−0.0025 (6)	−0.0101 (6)
C12	0.0503 (9)	0.0515 (9)	0.0486 (8)	−0.0007 (7)	−0.0095 (7)	−0.0028 (7)
C13	0.0726 (12)	0.0608 (11)	0.0612 (10)	0.0119 (10)	0.0074 (10)	−0.0003 (9)
C14	0.0809 (15)	0.0861 (15)	0.0721 (12)	0.0173 (12)	0.0154 (12)	−0.0050 (11)
C15	0.0783 (14)	0.0789 (14)	0.0827 (15)	0.0284 (12)	−0.0087 (12)	−0.0183 (12)
C16	0.101 (2)	0.0561 (12)	0.108 (2)	0.0204 (12)	−0.013 (2)	0.0032 (12)
C17	0.0853 (15)	0.0558 (11)	0.0850 (14)	0.0067 (11)	0.0041 (13)	0.0097 (10)
C18	0.0666 (14)	0.0596 (13)	0.129 (2)	0.0025 (11)	0.0146 (15)	−0.0285 (14)
C19	0.075 (2)	0.0631 (14)	0.115 (2)	0.0128 (12)	−0.0245 (15)	0.0050 (13)

Geometric parameters (Å, °)

O1—C9	1.371 (2)	C7—C8	1.386 (2)
O1—C2	1.449 (2)	C7—C19	1.509 (3)
C2—C12	1.500 (2)	C8—C9	1.385 (2)
C2—C3	1.523 (2)	C9—C10	1.392 (2)
C3—C4	1.506 (2)	C12—C13	1.377 (3)
C4—O11	1.217 (2)	C12—C17	1.382 (3)
C4—C10	1.473 (2)	C13—C14	1.390 (3)
C5—C6	1.373 (3)	C14—C15	1.374 (3)
C5—C10	1.401 (2)	C15—C16	1.373 (4)
C6—C7	1.411 (3)	C16—C17	1.376 (3)
C6—C18	1.516 (3)		
C9—O1—C2	114.34 (11)	C9—C8—C7	121.2 (2)
O1—C2—C12	108.90 (12)	O1—C9—C8	117.25 (14)
O1—C2—C3	109.73 (13)	O1—C9—C10	122.50 (14)
C12—C2—C3	111.20 (14)	C8—C9—C10	120.25 (15)
C4—C3—C2	111.53 (14)	C9—C10—C5	118.0 (2)
O11—C4—C10	122.9 (2)	C9—C10—C4	120.43 (14)
O11—C4—C3	122.3 (2)	C5—C10—C4	121.5 (2)
C10—C4—C3	114.79 (14)	C13—C12—C17	118.5 (2)
C6—C5—C10	122.5 (2)	C13—C12—C2	122.27 (15)
C5—C6—C7	118.7 (2)	C17—C12—C2	119.1 (2)
C5—C6—C18	120.0 (2)	C12—C13—C14	121.0 (2)

C7—C6—C18	121.3 (2)	C15—C14—C13	119.7 (2)
C8—C7—C6	119.3 (2)	C16—C15—C14	119.6 (2)
C8—C7—C19	119.3 (2)	C15—C16—C17	120.6 (2)
C6—C7—C19	121.4 (2)	C16—C17—C12	120.6 (2)
C9—O1—C2—C12	−176.33 (13)	O1—C9—C10—C4	5.2 (2)
C9—O1—C2—C3	−54.4 (2)	C8—C9—C10—C4	−174.85 (15)
O1—C2—C3—C4	56.5 (2)	C6—C5—C10—C9	−1.2 (3)
C12—C2—C3—C4	177.08 (13)	C6—C5—C10—C4	175.6 (2)
C2—C3—C4—O11	153.2 (2)	O11—C4—C10—C9	176.3 (2)
C2—C3—C4—C10	−28.9 (2)	C3—C4—C10—C9	−1.5 (2)
C10—C5—C6—C7	−0.5 (3)	O11—C4—C10—C5	−0.4 (3)
C10—C5—C6—C18	178.4 (2)	C3—C4—C10—C5	−178.3 (2)
C5—C6—C7—C8	1.4 (3)	O1—C2—C12—C13	27.7 (2)
C18—C6—C7—C8	−177.5 (2)	C3—C2—C12—C13	−93.3 (2)
C5—C6—C7—C19	−179.1 (2)	O1—C2—C12—C17	−156.8 (2)
C18—C6—C7—C19	2.1 (3)	C3—C2—C12—C17	82.1 (2)
C6—C7—C8—C9	−0.6 (3)	C17—C12—C13—C14	−0.2 (3)
C19—C7—C8—C9	179.9 (2)	C2—C12—C13—C14	175.2 (2)
C2—O1—C9—C8	−155.80 (15)	C12—C13—C14—C15	0.7 (4)
C2—O1—C9—C10	24.1 (2)	C13—C14—C15—C16	−0.7 (4)
C7—C8—C9—O1	178.76 (15)	C14—C15—C16—C17	0.2 (4)
C7—C8—C9—C10	−1.2 (3)	C15—C16—C17—C12	0.3 (4)
O1—C9—C10—C5	−177.88 (14)	C13—C12—C17—C16	−0.3 (3)
C8—C9—C10—C5	2.0 (2)	C2—C12—C17—C16	−175.9 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O11$^{\text{i}}$	0.98 (2)	2.59 (2)	3.314 (2)	131 (1)
C13—H13···O1	0.97 (2)	2.40 (2)	2.780 (3)	102 (2)

Symmetry codes: (i) −*x*, −*y*, −*z*+1.