ing e.s.d.'s are the largest of the 'external' and 'internal' standard deviations (Topping, 1960).

The calculations were performed on the ENCORE 91 computer of the Centro di Studio per la Strutturistica Diffrattometrica del CNR (Parma). Data collection: local programs. Cell refinement: *LQPARM* (Nardelli & Mangia, 1984). Data reduction: local programs. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1986). Program(s) used to refine structure: *SHELXL*92 (Sheldrick, 1992). Molecular graphics: *ORTEP* (Johnson, 1965). Software used to prepare material for publication: *PARST* (Nardelli, 1983*a*); *PARSTCIF* (Nardelli, 1991).

The authors are indebted to Professor G. M. Sheldrick who kindly made his program *SHELXL*92 available to them at the GAMMA-TEST checking stage. Financial support from the European Community Commission under contract No. SC1000657 is gratefully acknowledged.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71327 (82 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL1053]

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Acta Cryst. (1993). C**49**, 2027–2030

Structure of a Cyclohexanone Derivative

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(*Received* 29 *October* 1992; *accepted* 13 *May* 1993)

Abstract

The crystal structure of 4-hydroxy-4,*N*,*N*′-trimethyl-2-(3-nitrophenyl)-6-oxo-1,3-cyclohexanedicarboxamide has been determined. The cyclohexane ring exhibits a chair conformation. The phenyl ring is planar and is approximately perpendicular to the cyclohexanone ring. The 3-nitro substituent on the phenyl ring is twisted about 10° out of the ring plane. The amide carbonyl groups are oriented in different directions with respect to the cyclohexanone ring. These orientations of the carboxamide groups facilitate the formation of an intramolecular O—H···O hydrogen bond. The molecules are packed such that chains are formed along the *b* axis. These chains are held together by N—H···O hydrogen bonds.

Comment

The title compound was prepared by stirring an ethanol solution of *N*-methylacetoacetamide with aromatic aldehyde in the presence of a catalytic amount of piperidine at room temperature (Shetty, 1987). Since it possesses potential pharmacological and pesticidal activity and is used as a key intermediate in the preparation of phenothiazines (Sadanandam & Leelavathi, 1991), it was considered interesting to establish its conformational details by an X-ray diffraction study.

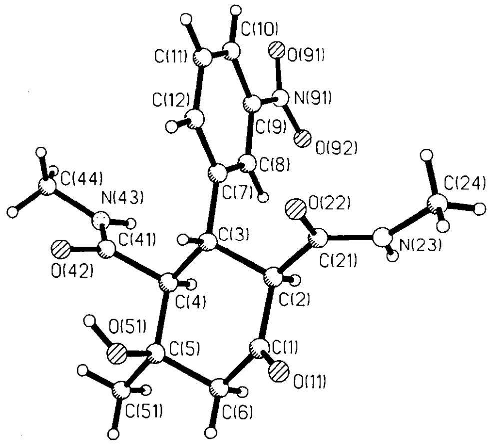

Fig. 1. A perspective view of the molecule with atom labelling.

The cyclohexanone ring is in a chair conformation. This is in agreement with the observations of Mootz & Berking (1969), Groth (1972), Bocelli (1981) and Spek, Duisenberg, van den Heuvel, Boer Rookhuizen & Bosch (1990). In fact, Bocelli (1981) has reported data for 22 substituted cyclohexanone structures, retrieved from the Cambridge Structural Database, of which 18 possess a chair conformation of the cyclohexanone ring. The mean value of the endocyclic torsion angles [56.4 (4)°] of the cyclohexanone ring shows that the title compound is slightly more puckered than cyclohexanone [54.1 (3)° (Suwinska & Gerdil, 1987)]. The values of the puckering parameters Q, φ and θ (Cremer & Pople, 1975) of 0.577 Å, −88.8 and 176.7°, respectively, compare well with the values reported by Spek, Duisenberg, van den Heuvel, Boer Rookhuizen & Bosch (1990).

The 3-nitro substituent on the phenyl ring is twisted by about 10° out of the plane of this ring [C(8)—C(9)—N(91)—O(91) = 170.0 (6) and C(8)—C(9)—N(91)—O(92) = −8.8 (9)°]. The phenyl ring itself is nearly planar [$\sum(\Delta/\sigma)^2 = 10.13$], with its orientation perpendicular to the cyclohexanone ring, as can be seen from the torsion angles C(2)—C(3)—C(7)—C(8) and C(4)—C(3)—C(7)—C(8) of 63.9 (5) and −60.2 (4), respectively. The conformation of the nitrophenyl ring relative to the cyclohexanone ring is constrained perhaps by the presence of the carboxamide groups at atoms C(2) and C(4) of the cyclohexanone ring.

There are differences in the spatial arrangement of the carbonyl groups at the C(2) and C(4) positions of the cyclohexanone ring. The two carbonyl groups are twisted in different directions, the torsion angles C(5)—C(4)—C(41)—O(42) and C(1)—C(2)—C(21)—O(22) being 49.2 (5) and −79.7 (4)°, respectively. The carbonyl group at C(4) is +synclinal and that at C(2) is −synclinal with respect to the C(5)—C(4) and C(1)—C(2) bonds, respectively. It is interesting to note that this +synclinal orientation of the group at C(4) facilitates the formation of an intramolecular hydrogen bond [O(42)···O(51) = 2.632 (3), H(51)···O(42) = 1.896 (4) Å and O(51)—H(51)···O(42) = 145.2 (3)°].

The carboxamide substituents at C(2) and C(4) are both equatorial to the cyclohexanone ring. Whereas the orientation of the 5-methyl group is equatorial [torsion angle C(3)—C(4)—C(5)—C(51) = −177.7 (3)°], that of the 5-hydroxy group is axial to the cyclohexanone ring [torsion angle C(3)—C(4)—C(5)—O(51) = 60.2 (4)°].

The temperature factors of the O atoms of the nitro group are quite high, probably because of the greater thermal motion of these terminal atoms. A similar effect has been reported by Chen, Masnovi, Baker, Krafcik & Towns (1992).

The molecules form chains along the *b* axis (Fig. 2). These chains are held together by the hydrogen bonds N(23)···O(51)($-x+\frac{1}{2}$, $y-\frac{1}{2}$, $-z+\frac{1}{2}+1$) 2.875 (4) and N(43)···O(42)($-x+\frac{1}{2}+1$, $y-\frac{1}{2}$, $-z+\frac{1}{2}+1$) 2.798 (5) Å.

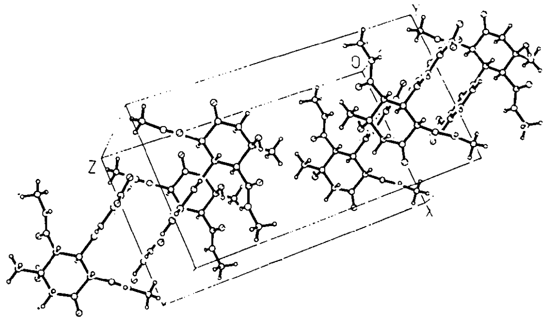

Fig. 2. A perspective view of the packing of the molecules in the unit cell.

Experimental

Crystal data

$C_{17}H_{21}N_3O_6$
M_r = 363.4
Monoclinic
$P2_1/n$
a = 10.196 (2) Å
b = 9.233 (2) Å
c = 20.068 (4) Å
β = 90.33 (1)°
V = 1889.2 (7) Å^3
Z = 4
D_x = 1.278 Mg m^{-3}
D_m = 1.268 (3) Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71063 Å
Cell parameters from 25 reflections
θ = 7–15°
μ = 0.092 mm^{-1}
T = 293 (1) K
Cubes
0.18 × 0.15 × 0.15 mm
Colourless
Crystal source: recrystallization from methanol

Data collection

Siemens *R*3*m*/*V* diffractometer

$R_{\rm int}$ = 0.074
$\theta_{\rm max}$ = 45°

$\omega/2\theta$ scans
Absorption correction: none
2844 measured reflections
2485 independent reflections
1730 observed reflections $[I \geq 3\sigma(I)]$

$h = -11 \rightarrow 11$
$k = 0 \rightarrow 10$
$l = 0 \rightarrow 21$
2 standard reflections monitored every 98 reflections intensity variation: <1%

Refinement

Refinement on F
Final $R = 0.056$
$wR = 0.053$
$S = 1.92$
1730 reflections
235 parameters
$w = 1/[\sigma^2(F) + 0.0015|F|^2]$

$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.201$ e Å^{-3}
$\Delta\rho_{\min} = -0.152$ e Å^{-3}
Atomic scattering factors from *SHELXTL-Plus* (Sheldrick, 1990)

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$$U_{\rm eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
C(1)	0.2315 (3)	−0.0989 (4)	0.7409 (2)	0.036 (1)
O(11)	0.1409 (2)	−0.0135 (3)	0.7439 (1)	0.050 (1)
C(2)	0.3007 (3)	−0.1550 (4)	0.8027 (2)	0.034 (1)
C(21)	0.2341 (4)	−0.0997 (5)	0.8651 (2)	0.040 (1)
O(22)	0.2537 (2)	0.0233 (3)	0.8864 (1)	0.054 (1)
N(23)	0.1519 (3)	−0.1934 (4)	0.8933 (1)	0.054 (1)
C(24)	0.0739 (5)	−0.1571 (6)	0.9511 (2)	0.094 (2)
C(3)	0.4461 (3)	−0.1080 (4)	0.7998 (2)	0.037 (1)
C(4)	0.5090 (3)	−0.1643 (4)	0.7347 (2)	0.036 (1)
C(41)	0.6498 (4)	−0.1122 (4)	0.7320 (2)	0.045 (2)
O(42)	0.6743 (2)	0.0172 (3)	0.7190 (2)	0.068 (1)
N(43)	0.7448 (3)	−0.2044 (4)	0.7442 (2)	0.059 (1)
C(44)	0.8810 (4)	−0.1593 (5)	0.7470 (3)	0.090 (2)
C(5)	0.4333 (3)	−0.1113 (4)	0.6719 (2)	0.042 (1)
O(51)	0.4351 (2)	0.0447 (3)	0.6706 (1)	0.055 (1)
C(51)	0.4925 (4)	−0.1722 (5)	0.6084 (2)	0.066 (2)
C(6)	0.2884 (3)	−0.1512 (4)	0.6768 (2)	0.043 (1)
C(7)	0.5212 (4)	−0.1551 (5)	0.8615 (2)	0.050 (2)
C(8)	0.5392 (4)	−0.2994 (6)	0.8770 (2)	0.062 (2)
C(9)	0.6118 (5)	−0.3341 (8)	0.9333 (3)	0.088 (3)
N(91)	0.6315 (6)	−0.4902 (7)	0.9483 (3)	0.146 (4)
O(91)	0.7106 (6)	−0.5193 (7)	0.9925 (3)	0.216 (4)
O(92)	0.5742 (5)	−0.5786 (7)	0.9177 (4)	0.176 (4)
C(10)	0.6638 (6)	−0.2286 (8)	0.9750 (3)	0.118 (4)
C(11)	0.6427 (6)	−0.0897 (7)	0.9593 (3)	0.121 (3)
C(12)	0.5735 (4)	−0.0507 (6)	0.9035 (2)	0.082 (2)

Table 2. *Bond distances* (Å), *bond angles* (°) *and selected torsion angles* (°)

C(1)—O(11)	1.216 (4)	C(1)—C(2)	1.514 (5)
C(1)—C(6)	1.496 (5)	C(2)—C(21)	1.515 (5)
C(2)—C(3)	1.546 (4)	C(21)—O(22)	1.230 (5)
C(21)—N(23)	1.333 (5)	N(23)—C(24)	1.449 (6)
C(3)—C(4)	1.549 (5)	C(3)—C(7)	1.516 (5)
C(4)—C(41)	1.515 (5)	C(4)—C(5)	1.553 (5)
C(41)—O(42)	1.245 (5)	C(41)—N(43)	1.312 (5)
N(43)—C(44)	1.450 (5)	C(5)—O(51)	1.441 (5)
C(5)—C(51)	1.520 (5)	C(5)—C(6)	1.526 (5)
C(7)—C(8)	1.380 (7)	C(7)—C(12)	1.385 (7)
C(8)—C(9)	1.385 (7)	C(9)—N(91)	1.485 (10)
C(9)—C(10)	1.387 (9)	N(91)—O(91)	1.225 (8)
N(91)—O(92)	1.176 (9)	C(10)—C(11)	1.338 (9)
C(11)—C(12)	1.367 (7)		
C(2)—C(1)—C(6)	114.4 (3)	O(11)—C(1)—C(6)	123.4 (3)
O(11)—C(1)—C(2)	122.1 (3)	C(1)—C(2)—C(3)	108.3 (3)
C(1)—C(2)—C(21)	110.6 (3)	C(21)—C(2)—C(3)	111.8 (3)
C(2)—C(21)—N(23)	114.7 (3)	C(2)—C(21)—O(22)	121.7 (3)
O(22)—C(21)—N(23)	123.5 (4)	C(21)—N(23)—C(24)	122.7 (4)
C(2)—C(3)—C(7)	111.7 (3)	C(2)—C(3)—C(4)	109.8 (3)
C(4)—C(3)—C(7)	112.5 (3)	C(3)—C(4)—C(5)	111.9 (3)
C(3)—C(4)—C(41)	108.6 (3)	C(41)—C(4)—C(5)	109.7 (3)
C(4)—C(41)—N(43)	119.1 (3)	C(4)—C(41)—O(42)	120.2 (3)
O(42)—C(41)—N(43)	120.6 (4)	C(41)—N(43)—C(44)	121.8 (3)
C(4)—C(5)—C(6)	110.4 (3)	C(4)—C(5)—C(51)	111.4 (3)
C(4)—C(5)—O(51)	108.9 (3)	C(51)—C(5)—C(6)	110.7 (3)
O(51)—C(5)—C(6)	104.7 (3)	O(51)—C(5)—C(51)	110.4 (3)
C(1)—C(6)—C(5)	111.0 (3)	C(3)—C(7)—C(12)	119.2 (4)
C(3)—C(7)—C(8)	121.9 (4)	C(8)—C(7)—C(12)	119.0 (4)
C(7)—C(8)—C(9)	118.5 (5)	C(8)—C(9)—C(10)	122.0 (5)
C(8)—C(9)—N(91)	117.4 (5)	N(91)—C(9)—C(10)	120.5 (5)
C(9)—N(91)—O(92)	120.1 (6)	C(9)—N(91)—O(91)	116.6 (6)
O(91)—N(91)—O(92)	123.3 (7)	C(9)—C(10)—C(11)	118.1 (6)
C(10)—C(11)—C(12)	121.8 (6)	C(7)—C(12)—C(11)	120.6 (5)
C(2)—C(1)—C(6)—C(5)	−57.3 (4)		
C(1)—C(2)—C(3)—C(4)	−56.7 (3)		
C(21)—C(2)—C(3)—C(4)	−178.9 (3)		
C(2)—C(3)—C(7)—C(8)	63.9 (5)		
C(2)—C(3)—C(4)—C(41)	178.0 (3)		
C(4)—C(3)—C(7)—C(8)	−60.2 (4)		
C(3)—C(4)—C(5)—O(51)	60.2 (4)		
C(3)—C(4)—C(5)—C(6)	−54.2 (3)		
C(4)—C(5)—C(6)—C(1)	52.9 (4)		
C(8)—C(9)—N(91)—O(91)	170.0 (6)		
C(6)—C(1)—C(2)—C(3)	58.7 (4)		
C(1)—C(2)—C(21)—O(22)	−79.7 (4)		
C(3)—C(2)—C(21)—O(22)	41.2 (5)		
C(2)—C(3)—C(7)—C(12)	−116.1 (4)		
C(2)—C(3)—C(4)—C(5)	56.7 (4)		
C(4)—C(3)—C(7)—C(12)	119.8 (4)		
C(3)—C(4)—C(5)—C(51)	−177.7 (3)		
C(3)—C(4)—C(41)—O(42)	−73.4 (4)		
C(7)—C(8)—C(9)—N(91)	−179.0 (5)		
C(8)—C(9)—N(91)—O(92)	−8.8 (9)		

A $\Delta\rho$ map showed the positions of all H-atoms but they were placed in idealized positions and included in the least-squares refinement with fixed isotropic temperature factors. *SHELXTL-Plus* (Sheldrick, 1990) was used for the structure solution and refinement and also to produce the figures. Geometrical parameters were calculated using the program *PARST* (Nardelli, 1983).

Thanks are due to Dr Y. S. Sadanandam and Dr (Mrs) Meera Shetty, Organic Chemistry Division, IICT, Hyderabad, for supplying the compound.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and bond distances and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71324 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB1055]

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Acta Cryst. (1993). C**49**, 2030–2032

2-(2,3-Dimethoxyphenyl)-6-hydroxy-4*H*-1-benzopyran-4-one (6-Hydroxy-2′,3′-dimethoxyflavone)

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(*Received* 25 *January* 1993; *accepted* 20 *May* 1993)

Abstract

The structure of the new flavone reported here has a general shape defined by a phenyl ring rotated by 62.2 (1)° from the plane of the γ-benzopyranone portion. The two methoxy groups have the expected conformations: out of plane for the methoxy group at C2′ and in plane for the other at C3′. The major influence on the packing appears to be the O6—H6···O4 intermolecular hydrogen bond.

Comment

Fig. 1 represents a perspective view of the asymmetric unit with the atom-numbering scheme. A search of the January 1980 version of the Cambridge Structural Database (Allen, Kennard & Taylor, 1983) revealed that this is the first report of a 2′,3′-dimethoxylated fragment for flavones.

The molecule can be described in two different regions: γ-benzopyrone atoms, including the O atoms of the hydroxyl and carbonyl groups, lie in one plane and the phenyl atoms lie in another plane. The angle between these two planes is 62.2 (1)°. The methoxy group at C(2′) is oriented out of the plane of the phenyl ring with a torsion angle of C2′1—O2′—C2′—C3′ = 70.5°. The methoxy group at C3′ lies in the phenyl plane; C3′1—O3′—C3′—C4′ = 1.1°.

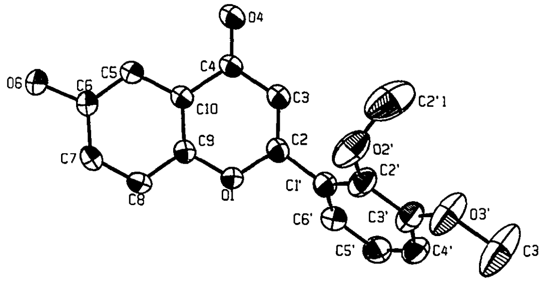

Fig. 1. The structure of the molecule with the atom-numbering scheme. The thermal ellipsoids are drawn at 50% probability.

The O—H6···O4 intermolecular bonds appear to have an important effect on the packing of the molecule. Each molecule hydrogen bonds to another molecule related by $-0.5 + x$, $1.5 - y$, z and with a molecule related by $0.5 + x$, $1.5 - y$, z. The molecules are arranged in such a way that all the hydroxyl and carbonyl groups participate in hydrogen bonding, thus inducing a gathering of hydrophobic dimethoxy groups on the opposite side (Fig. 2). It may also be noted that one molecule hydrogen bonds to two other molecules, giving infinite chains rather than associated pairs of molecules.

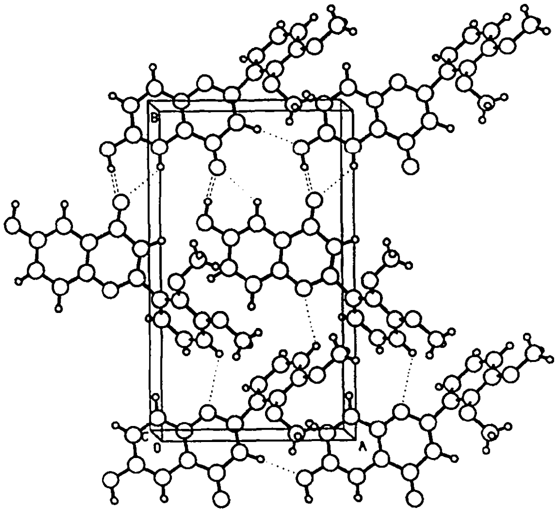

Fig. 2. The crystal packing viewed down the *c* axis.

Similar flavones with the same γ-benzopyrone fragment are 6-hydroxyflavone (Seetharaman & Rajan, 1992) and 6-hydroxy-2′,3′,4′-trimethoxyflavone (Wallet, 1993), which have torsion angles of 9.8 and 19.1°, respectively. Bond lengths and angles are comparable but the torsion angles are different, the smaller value being for 6-hydroxyflavone with no substituent on the phenyl ring; the title compound is more twisted than either of these compounds.

Acta Crystallographica Section C
ISSN 0108-2701 ©1993