

Fig. 2. Stereoscopic ORTEP packing drawing. The dotted and dashed lines denote the hydrogen bonds and electrostatic interactions, respectively.

slightly larger than the sum of van der Waals radii (3.25 Å). The conformation of the hydroxyethyl side chain is similar to that found in thiamin compounds with a close S...O interaction:  $\varphi_{5\alpha} = \text{S}(1) - \text{C}(5) - \text{C}(5\alpha) - \text{C}(5\beta) = 90.5^\circ$  and  $\varphi_{5\beta} = \text{C}(5) - \text{C}(5\alpha) - \text{C}(5\beta) - \text{O}(5\gamma) = -60.8^\circ$ .

The chloride ions play an important role in the crystal packing (Fig. 2). Besides the  $\text{C}(2) - \text{H} \cdots \text{Cl}^-$  hydrogen bond and the  $\text{S}(1) \cdots \text{Cl}^-$  electrostatic interaction, the  $\text{Cl}^-$  ion is hydrogen bonded to  $\text{O}(5\gamma)$  [ $\text{O}(5\gamma) \cdots \text{Cl}^-$  3.071 (2),  $\text{H}(5\gamma) \cdots \text{Cl}^-$  2.30 (4) Å,

$\text{O}(5\gamma) - \text{H}(5\gamma) \cdots \text{Cl}^-$  175 (4)°]. Two molecules related by a center of symmetry at  $(\frac{1}{2}, \frac{1}{2}, 0)$  are dimerized via a pair of the  $\text{C}(2) - \text{H} \cdots \text{Cl}^- \cdots \text{H} - \text{O}(5\gamma)$  hydrogen-bonding chains. These dimers are linked by strong  $\text{S}(1) \cdots \text{Cl}^-$  electrostatic interactions to form a molecular chain along the *a* axis. There are only van der Waals interactions between the molecular chains.

This work was supported by Non-Directed Research Fund, Korea Research Foundation (1990).

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*Acta Cryst.* (1993). C49, 70–72

## Structure of a Chalcone

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(Received 16 September 1991; accepted 8 June 1992)

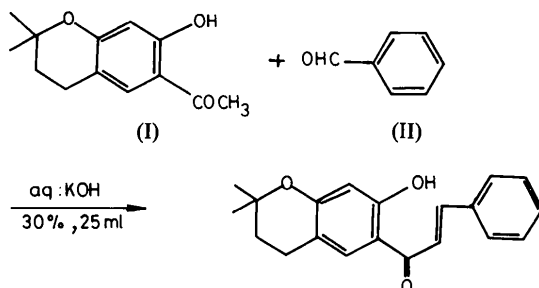
**Abstract.** 1-(7-Hydroxy-2,2-dimethylchroman-6-yl)-3-phenyl-2-propen-1-one,  $\text{C}_{20}\text{H}_{20}\text{O}_3$ ,  $M_r = 308.38$ ,  $P2_1/a$ ,  $a = 12.002$  (2),  $b = 11.013$  (2),  $c = 12.652$  (3) Å,  $\beta = 106.24$  (3)°,  $V = 1605.6$  Å<sup>3</sup>,  $Z = 4$ ,  $D_m = 1.26$  (2),  $D_x = 1.275$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $\mu = 0.079$  mm<sup>-1</sup>,  $F(000) = 656$ ,  $T = 293$  K,  $R = 0.037$ ,  $wR = 0.034$  for 892 reflections with  $I > 3\sigma(I)$ . There is an internal hydrogen bond  $\text{O} - \text{H} \cdots \text{O}$  between the carbonyl O atom in the cinnamoyl

group and the hydroxy group of the chroman moiety. The conformational analysis shows that the phenyl ring is *trans* with respect to the double bond linking it with the chroman group. The pyran ring is in the prevalent half-chair conformation.

**Introduction.** Analytical studies have shown that chalcones are the precursors of isoflavones, which are the pigments of many plants. The title compound, 6-cinnamoyl-7-hydroxy-2,2-dimethylchroman, which is a chalcone, was prepared by treating

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6-acetyl-7-hydroxy-2,2-dimethylchroman (I) with aryl aldehyde (II) under alkaline conditions (Suresh, Rukmani Iyer & Iyer, 1985). The crystal structure analysis was performed to determine the conformation of the C=C and C=O bonds bridging the two fragments of the molecule.



**Experimental.** The X-ray diffraction data were collected on an Enraf-Nonius CAD-4 diffractometer using the  $\omega$ - $2\theta$  scan mode. The unit-cell parameters were refined on the basis of 25  $2\theta$  values in the range 20–35°. No significant change was observed in the intensities of check reflections monitored every 100 reflections. Lp correction but absorption neglected. The crystal data and the X-ray data-collection parameters are given in Table 1. Refinement was on  $F$ .

**Discussion.** The final atomic coordinates are given in Table 2.\* An ORTEP (Johnson, 1965) plot of the molecule is given in Fig. 1. The carbonyl O atom O(3) of the cinnamoyl group and the hydroxy group O(1) of the chroman moiety are involved in an O—H...O internal hydrogen bond [O(2)...O(3) = 2.563, H(O2)...O(3) = 1.743 (55) Å, O(2)—H(O2)...O(3) = 152.38 (32)°], forming a six-membered ring consisting of O(2), H(O2), O(3), C(13), C(6) and C(7) as in 2',4'-dihydrochalcone (Fronczek, Tanrisever & Fischer, 1987). The phenyl ring is *trans* with respect to the C(14)—C(15) bond as shown by the torsion angle  $\tau$  [C(13)—C(14)—C(15)—C(16)] = 179.4 (4)°. The pyran ring adopts a half-chair conformation, a prevalent feature in chalcones [e.g. 2,3-dihydro-1H-naphtho[2,1-*b*]pyran-1-one (Ravikumar, Rajan, Ponnuswamy & Trotter, 1986); 3,4-dihydro-2H,5H-pyrano[3,2-*c*][1]benzopyran-5-ones (Valente, Santarsiero & Schomaker, 1979)], the distortion from the ideal being characterized by the asymmetry parameter  $\Delta C_{(2-3)} = 10.2$  (5)° (Duax & Norton, 1975).

\* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, bond lengths, bond angles, torsion angles and least-squares-planes calculations have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55516 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CD0080]

Table 1. *Crystal data and X-ray structure-determination parameters*

Crystal system	Monoclinic
Space group	$P2_1/a$
Crystal dimensions (mm)	$0.20 \times 0.30 \times 0.35$
Index range of unique data ( $h, k, l$ )	$\pm 11, 12, 15$
Reflections monitored ( $hkl$ )	142, 160
Number of reflections measured	1208
Number of reflections with $I > 3\sigma(I)$	892
$2\theta_{\max}$ (°)	62
$R_{\text{int}}$	0.050
$R_w$	0.048
$(\Delta/\sigma)_{\max}$	0.024
Max. and min. peak heights in final difference map ( $e \text{ \AA}^{-3}$ )	0.201/−0.195
$R$	0.037
$wR^*$	0.034

Programs used: SHELXS86 (Sheldrick, 1986) for solution and SHELXL76 (Sheldrick, 1976) for refinement. Scattering factors: SHELXL76 and SHELXS86. Computer used: Siemens 7580-E.

$$*w = k\{\sigma(F_o)\}^2 + g\{I(F_o)\}^{-1}, k = 1.00000, g = 0.000045.$$

Table 2. *Positional parameters ( $\times 10^4$ ) and equivalent isotropic thermal parameters ( $\text{\AA}^2 \times 10^3$ ) for non-H atoms with e.s.d.'s in parentheses*

	$x$	$y$	$z$	$U_{\text{eq}}$
O(1)	3693 (3)	4129 (3)	5713 (2)	46 (2)
O(2)	4851 (3)	1038 (3)	8299 (2)	44 (2)
O(3)	3597 (3)	0558 (3)	9585 (3)	51 (2)
C(2)	2851 (4)	5033 (5)	5093 (4)	44 (3)
C(3)	2340 (4)	5732 (5)	5883 (4)	54 (3)
C(4)	1748 (5)	4901 (5)	6528 (4)	57 (3)
C(5)	2434 (4)	3163 (4)	7883 (4)	34 (3)
C(6)	3189 (4)	2223 (4)	8371 (3)	32 (3)
C(7)	4097 (4)	1948 (4)	7902 (4)	33 (3)
C(8)	4234 (4)	2613 (4)	7021 (4)	37 (3)
C(9)	3467 (4)	3531 (4)	6570 (3)	35 (3)
C(10)	2548 (4)	3837 (4)	7003 (4)	36 (3)
C(11)	3571 (5)	5860 (5)	4594 (4)	67 (3)
C(12)	1956 (5)	4346 (5)	4227 (4)	71 (3)
C(13)	3023 (4)	1504 (5)	9286 (4)	38 (3)
C(14)	2172 (4)	1912 (4)	9857 (4)	40 (3)
C(15)	1756 (4)	1151 (4)	10469 (4)	41 (3)
C(16)	0916 (4)	1451 (5)	11084 (3)	35 (3)
C(17)	0724 (4)	2639 (4)	11357 (4)	44 (3)
C(18)	−0072 (5)	2866 (5)	11934 (4)	60 (3)
C(19)	−0704 (5)	1950 (6)	12225 (4)	55 (3)
C(20)	−0519 (5)	0772 (5)	11952 (4)	52 (3)
C(21)	0294 (4)	0526 (4)	11399 (4)	41 (3)

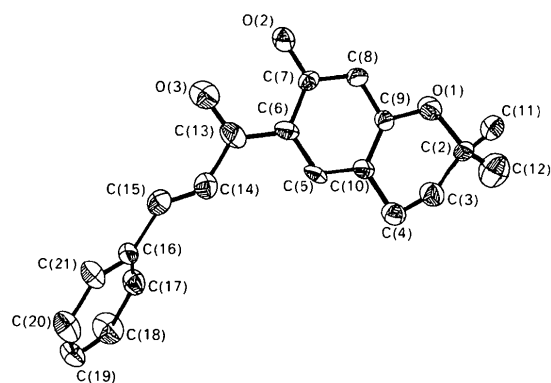


Fig. 1. ORTEP plot of the title molecule showing the thermal ellipsoids drawn at the 50% probability level.

The authors are grateful to Dr T. N. Guru Row, National Chemical Laboratory, Pune, for the use of the diffractometer. They thank the referees for their suggestions and comments.

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*Acta Cryst.* (1993). **C49**, 72–75

## Structure of 5,11,17,23-Tetra-*tert*-butyl-25,27-dicyanomethoxy-26,28-dimethoxycalix[4]arene

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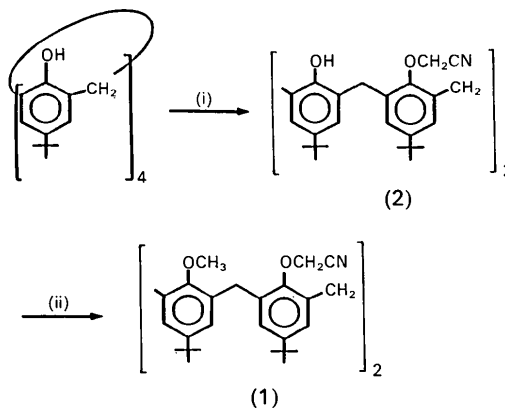
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(Received 17 February 1992; accepted 29 June 1992)

**Abstract.**  $\beta,\beta'$ -{5,11,17,23-Tetra-*tert*-butyl-26,28-dimethoxypentacyclo[19.3.1.1<sup>3,7</sup>.1<sup>9,13</sup>.1<sup>15,19</sup>]octacosal(25),3,5,7(28),9,11,13(27),15,17,19(26),21,23-dodecaene-25,27-diylidioxy}diacetonitrile, C<sub>50</sub>H<sub>62</sub>N<sub>2</sub>O<sub>4</sub>,  $M_r = 755.1$ , monoclinic,  $C2/c$ ,  $a = 14.659(1)$ ,  $b = 16.591(1)$ ,  $c = 37.558(5)$  Å,  $\beta = 90.38(1)^\circ$ ,  $V = 9134.19$  Å<sup>3</sup>,  $Z = 8$ ,  $D_x = 1.098$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.7107$  Å,  $\mu = 0.64$  cm<sup>-1</sup>,  $F(000) = 3264.0$ ,  $T = 293$  K,  $wR = 0.060$  ( $w = 1$ ) for 4394 observed reflections with  $I > 3\sigma(I)$ . The X-ray structure shows that the compound adopts a partial-cone conformation in which the two anisole moieties are parallel to each other with the methoxy groups pointing outwards. The cyano groups are in *anti* positions.

**Introduction.** Calix[4]arenes are cyclic tetramers extracted from the condensation products of phenol and formaldehyde in basic media (Gutsche, 1989; Vicens & Bohmer, 1991). They may exist in four different conformations: cone, partial-cone, 1,2-alternate and 1,3-alternate (Gutsche, 1989; Vicens & Bohmer, 1991). In most cases, non-functionalized

calix[4]arenes as well as their *O*-alkyl derivatives display a cone conformation. Aiming at the preparation of mixed *O*-functionalized ligands derived from *p-tert*-butylcalix[4]arene, we synthesized the dimethoxydinitrile ligand (1), which was shown to exist in partial-cone conformation, as deduced from NMR experiments.



- (i) BrCH<sub>2</sub>CN, K<sub>2</sub>CO<sub>3</sub>, acetonitrile, reflux 25h  
(ii) CH<sub>3</sub>I, K<sub>2</sub>CO<sub>3</sub>, acetonitrile, reflux 8h

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