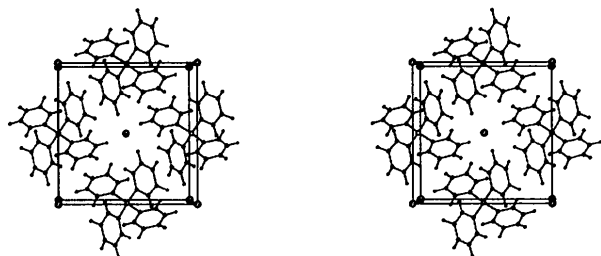
Fig. 5. Unit-cell packing diagram for (2) as viewed down the *c* axis.Fig. 6. Unit-cell packing diagram for (3) as viewed down the *a* axis.

For (3), yellow crystals from methylene chloride/hexane (0.26 × 0.30 × 0.36 mm); lattice parameters from least-squares fit of 25 reflections ( $20 < 2\theta < 25^\circ$ ); empirical absorption correction (6 reflections, 216 data);  $2\theta_{\max} = 48^\circ$  ( $h = +15, k = +15, l = +9$ ); standard reflections 222, 123, 321 ( $\leq 1\%$ ). 675 reflections collected, 667 independent ( $R_{\text{int}} = 2.7\%$ ), 624 observed with  $F_o > 5\sigma(F_o)$ , 43 unobserved reflections. Least-squares refinement on 81 parameters; all non-H atoms

anisotropic, H atoms were refined (C—H = 0.96 Å).  $R_F = 3.68\%$ ,  $wR_F = 5.63\%$ ,  $S = 1.28$ ,  $w^{-1} = \sigma^2(F_o) + gF_o^2$ ,  $g = 0.001$ ;  $(\Delta/\sigma)_{\max} = 0.044$ ;  $\Delta\rho_{\max} = 0.55$ ,  $\Delta\rho_{\min} = -0.28 \text{ e } \text{Å}^{-3}$ .

Atomic coordinates and equivalent isotropic thermal parameters for (1), (2), and (3) are given in Tables 1, 2 and 3, respectively.\* Bond lengths and angles for (1) are given in Table 4, for (2) in Table 5, and for (3) in Table 6. Phenyl-ring torsional angles for (1), (2) and (3) are given in Table 7. Figs. 1, 2 and 3 show the labeled molecular structures for (1), (2) and (3), respectively. Unit-cell packing diagrams for (1), (2) and (3) are shown in Figs. 4, 5 and 6, respectively.

**Related literature.** To our knowledge, there has been only one previous report of the structure of a tetraphenyl cation halide of a group-15 element: tetraphenylphosphonium dibromiodide (Muller, 1979).

\* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51738 (78 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Structure of 8-Methoxy-5-methyl-3,4-*cis*-diphenylisochroman-1-one

BY UPALI SIRIWARDANE, LORI CRENSHAW, SUBHASH P. KHANAPURE AND EDWARD R. BIEHL\*

*Department of Chemistry, Southern Methodist University, Dallas, Texas 75275, USA*

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**Abstract.**  $\text{C}_{23}\text{H}_{20}\text{O}_3$ ,  $M_r = 344.4$ , monoclinic,  $P2_1/c$ ,  $a = 6.254$  (6),  $b = 18.266$  (3),  $c = 15.536$  (3) Å,  $\beta = 93.35$  (1)°,  $V = 1771.7$  (4) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.29 \text{ g cm}^{-3}$ ,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 0.79 \text{ cm}^{-1}$ ,  $F(000) = 728$ ,  $T = 295 \text{ K}$ , final  $R = 0.044$  for 1023 observed reflections. The bond distances are C=O = 1.196 (11), O—C (cyclic, av.) = 1.405 (10), O—C (benz.) = 1.364 (8), O—CH<sub>3</sub> = 1.417 (7), C—C (benz.,

av) = 1.382 (12), C—C (aliph.—aliph.) = 1.535 (6) and C—C (benz.—aliph., av.) = 1.512 (9) Å. The X-ray structure shows the half-boat conformation of the six-membered lactone ring and confirms the *cis* disposition of the 3- and 4-phenyl groups.

**Experimental.** The title compound (1) was prepared by an intramolecular addition of the lithio salt obtained by the reaction of 6-benzyl- $\alpha$ -lithio-5-methyl-*o*-anisonitrile and benzaldehyde at 195 K to room temperature (18 h)

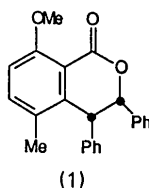
\* To whom correspondence should be addressed.

Table 1. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic displacement coefficients ( $\text{\AA}^2 \times 10^3$ )

Equivalent isotropic  $U$  defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor.

	$x$	$y$	$z$	$U_{eq}$
O(1)	2417 (9)	4872 (2)	6679 (2)	41 (2)
O(2)	5255 (9)	4250 (2)	7128 (2)	56 (2)
O(3)	4590 (8)	3168 (2)	8208 (2)	63 (2)
C(1)	409 (10)	4561 (3)	8219 (3)	36 (2)
C(2)	-647 (10)	4378 (3)	8956 (3)	42 (2)
C(3)	108 (11)	3782 (3)	9436 (3)	53 (3)
C(4)	1825 (12)	3373 (3)	9208 (3)	53 (3)
C(5)	2881 (11)	3552 (3)	8478 (3)	44 (3)
C(6)	2185 (10)	4156 (3)	7982 (3)	38 (2)
C(7)	-362 (9)	5189 (2)	7644 (3)	38 (2)
C(8)	124 (13)	4983 (3)	6718 (3)	38 (3)
C(9)	3436 (15)	4404 (3)	7244 (3)	42 (3)
C(10)	-529 (13)	5535 (3)	6029 (3)	38 (3)
C(11)	-2632 (13)	5536 (3)	5701 (3)	45 (3)
C(12)	-3315 (10)	6020 (3)	5051 (3)	47 (3)
C(13)	-1861 (15)	6509 (3)	4742 (3)	51 (3)
C(14)	216 (14)	6524 (3)	5078 (4)	55 (3)
C(15)	882 (10)	6037 (3)	5719 (3)	47 (2)
C(16)	546 (13)	5932 (3)	7917 (3)	38 (3)
C(17)	-654 (11)	6556 (3)	7772 (3)	64 (3)
C(18)	187 (17)	7245 (4)	7964 (4)	86 (4)
C(19)	2228 (17)	7313 (4)	8319 (4)	83 (4)
C(20)	3419 (11)	6701 (4)	8484 (3)	64 (3)
C(21)	2582 (12)	6011 (3)	8287 (3)	49 (3)
C(22)	-2560 (10)	4795 (3)	9228 (3)	57 (3)
C(23)	5437 (10)	2594 (3)	8737 (4)	78 (3)

followed by acidic work-up (Crenshaw, Khanapure, Siriwardane & Biehl, 1988).



Crystals of (1) are colorless rectangular plates, unit-cell parameters by least-squares fit of 25 reflections in the range  $10 < 2\theta < 25^\circ$ , crystal dimensions  $0.30 \times 0.20 \times 0.10$  mm, space group  $P2_1/c$  from systematic absences ( $0k0$ ,  $k$  odd,  $h0l$ ,  $l$  odd); automatic Nicolet  $R3m/V$  diffractometer, graphite-monochromated Mo  $K\alpha$  radiation,  $\theta/2\theta$  scan mode (scan rate  $3.0\text{--}15.0^\circ \text{ min}^{-1}$ , depending on intensity), 1876 measured reflections, 1516 independent reflections in the range  $3 < 2\theta < 45^\circ$ ,  $R_{int} = 0.023$ ,  $hkl$  range  $h - 3 \rightarrow 3$ ,  $k 0 \rightarrow 19$ ,  $l 0 \rightarrow 16$ , 1023 observed reflections with  $I > 3.0\sigma(I)$ ,  $\sigma(I)$  from counting statistics; three standard reflections remeasured after every 100 reflections did not show any significant change in intensity (1.0%) during data collection; Lorentz-polarization correction, no absorption or extinction corrections. Structure solved by direct methods (*SHELXTL-Plus*, Sheldrick, 1988) and subsequent difference Fourier methods. Hydrogen positions were calculated. Primary, tertiary and phenyl hydrogen were included in riding

Table 2. Selected bond lengths ( $\text{\AA}$ ), bond angles ( $^\circ$ ) and torsion angles ( $^\circ$ )

O(1)–C(8)	1.453 (10)	O(1)–C(9)	1.357 (7)
O(2)–C(9)	1.196 (11)	O(3)–C(5)	1.364 (8)
O(3)–C(23)	1.417 (7)	C(1)–C(7)	1.515 (6)
C(2)–C(22)	1.500 (9)	C(6)–C(9)	1.495 (8)
C(7)–C(8)	1.535 (6)	C(7)–C(16)	1.523 (7)
C(8)–C(10)	1.509 (7)		
C(8)–O(1)–C(9)	119.3 (5)	C(5)–O(3)–C(23)	118.4 (4)
C(2)–C(1)–C(7)	121.0 (5)	C(6)–C(1)–C(7)	118.3 (4)
C(1)–C(2)–C(22)	122.3 (5)	C(3)–C(2)–C(22)	119.9 (5)
O(3)–C(5)–C(6)	123.4 (5)	O(3)–C(5)–C(6)	117.4 (5)
C(1)–C(6)–C(9)	120.0 (5)	C(5)–C(6)–C(9)	120.0 (6)
C(1)–C(7)–C(8)	107.1 (4)	C(1)–C(7)–C(16)	114.2 (4)
C(8)–C(7)–C(16)	112.9 (4)	O(1)–C(8)–C(7)	108.8 (4)
O(1)–C(8)–C(10)	106.9 (5)	C(7)–C(8)–C(10)	116.2 (4)
O(1)–C(9)–O(2)	117.8 (6)	O(1)–C(9)–C(6)	116.1 (7)
O(2)–C(9)–C(6)	126.1 (5)	C(8)–C(10)–C(11)	118.3 (6)
C(8)–C(10)–C(15)	122.6 (6)	C(7)–C(16)–C(17)	120.1 (6)
C(7)–C(16)–C(21)	122.1 (5)	C(17)–C(16)–C(21)	117.8 (5)
C(9)–O(1)–C(8)–C(7)	52.8 (5)	C(9)–O(1)–C(8)–C(10)	179.0 (4)
C(8)–O(1)–C(9)–O(2)	170.5 (5)	C(8)–O(1)–C(9)–C(6)	-12.2 (6)
C(23)–O(3)–C(5)–C(6)	-5.2 (8)	C(23)–O(3)–C(5)–C(6)	174.7 (5)
C(6)–C(1)–C(2)–C(22)	-179.7 (5)	C(7)–C(1)–C(2)–C(3)	178.0 (5)
C(7)–C(1)–C(2)–C(22)	-1.0 (8)	C(2)–C(1)–C(6)–C(5)	1.5 (8)
C(2)–C(1)–C(6)–C(9)	-173.6 (5)	C(7)–C(1)–C(6)–C(5)	-177.2 (5)
C(7)–C(1)–C(6)–C(9)	7.7 (7)	C(2)–C(1)–C(7)–C(8)	-147.2 (5)
C(2)–C(1)–C(7)–C(8)	87.0 (7)	C(6)–C(1)–C(7)–C(8)	31.4 (7)
C(6)–C(1)–C(7)–C(16)	-94.3 (6)	O(3)–C(5)–C(6)–C(9)	-6.2 (8)
C(4)–C(5)–C(6)–C(9)	173.7 (5)	C(1)–C(6)–C(9)–O(1)	-19.9 (7)
C(1)–C(6)–C(9)–O(2)	157.2 (6)	C(5)–C(6)–C(9)–O(1)	165.0 (5)
C(1)–C(7)–C(8)–C(10)	179.7 (6)	C(16)–C(7)–C(8)–O(1)	66.9 (5)
C(16)–C(7)–C(8)–C(10)	-53.8 (8)	C(1)–C(7)–C(16)–C(17)	-148.1 (5)
C(1)–C(7)–C(16)–C(21)	34.0 (7)	C(8)–C(7)–C(16)–C(17)	89.2 (7)
C(8)–C(7)–C(16)–C(21)	-88.6 (6)	O(1)–C(8)–C(10)–C(11)	155.5 (4)
O(1)–C(8)–C(10)–C(15)	-24.7 (6)	C(7)–C(8)–C(10)–C(11)	-82.7 (6)
C(7)–C(16)–C(17)–C(18)	-175.8 (5)	C(7)–C(16)–C(21)–C(20)	176.2 (5)

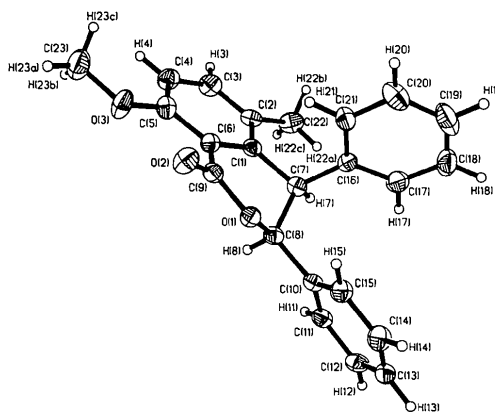


Fig. 1. ORTEP (Johnson, 1965) drawing of the molecule. Thermal ellipsoids are scaled to enclose 30% probability. H atoms are represented as spheres of arbitrary radii.

model with fixed isotropic  $U^s$ . Final full-matrix least-squares refinement (*SHELXTL-Plus*) converged to  $R = 0.044$ ,  $wR = 0.049$ ,  $S = 1.54$  for 235 parameters;  $w = 1/[\sigma^2(F) + 0.0009F^2]$ ,  $\sum w(|F_o| - |F_c|)^2$  minimized;  $(\Delta/\sigma)_{max} = 0.012$ ,  $\Delta\rho_{max, min} = 0.24, -0.27 \text{ e \AA}^{-3}$  in final difference Fourier map. The puckering parameters (Cremer & Pople, 1975) for the six-membered lactone ring in the half-boat conformation are  $q_2 = 0.472$  (6),  $\varphi_2 = 1.9$  (8),  $q_3 = 0.351$  (6),

$Q = 0.588(6)$  and  $\theta = 126.6(6)$ . Atomic scattering factors for C, H and O were those stored in *SHELXTL-Plus* which were taken from *International Tables for X-ray Crystallography* (1974). The final atomic parameters are given in Table 1,\* selected bond lengths, angles and torsion angles are given in Table 2. The identification of the atoms and the configuration are shown in the *ORTEP* (Johnson, 1965) drawing of Fig. 1.

**Related literature.** Recently we observed (Khanapure, Crenshaw, Reddy & Biehl, 1988) that the reactions of diaryl-2-cyano- $\alpha$ -lithiomethanes with benzaldehydes are highly diastereoselective which is in accord with Cram's rule (Cram & Wilson, 1963). The lithio salts formed in these reactions undergo further intramolecular addition to nitrile to give isochroman-1-ones upon acidic work-up. The lithiation of alkylbenzonitriles and subsequent addition of the resulting anion to alkylbenzonitrile itself to give ketones is known (Kaiser & Petty, 1976). The one-pot high-yield synthesis of *cis*-3,4-diarylisochroman-1-one is of significance due to the presence of the 3-arylisochroman-1-one moiety in several natural products such as hydrangenol and

phylloolulcin (Watanabe, Sahara, Furukawa, Billedeau & Snieckus, 1982). Several other methods involving *ortho*-directed metallation of benzamides and reaction of aldehydes with *ortho*-toluenes have also been used (Regan & Staunton, 1987) in the synthesis of 3,4-dialkylisochroman-1-ones.

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\* Lists of anisotropic temperature factors, bond lengths, bond angles and torsion angles, H-atom parameters, and structure factors have been deposited with British Library Document Supply Centre as Supplementary Publication No. SUP 51800 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Structure of 3-Benzamido-6,8-dibenzoyl-1,3,5,6,8-pentaazabicyclo[3.2.2]nonane

BY CLIFFORD GEORGE

Laboratory for the Structure of Matter, Naval Research Laboratory, Washington, DC 20375, USA

(Received 2 December 1988; accepted 18 January 1989)

**Abstract.**  $C_{25}H_{23}N_6O_3$ ,  $M_r = 455.50$ , monoclinic,  $P2_1/c$ ,  $a = 11.325(3)$ ,  $b = 18.161(4)$ ,  $c = 11.952(5)$  Å,  $\beta = 112.01(3)^\circ$ ,  $V = 2280.3(10)$  Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.327$  g cm<sup>-3</sup>,  $\lambda(\text{Cu K}\alpha) = 1.54178$  Å,  $\mu = 7.01$  cm<sup>-1</sup>,  $F(000) = 956$ ,  $T = 295$  K, final  $R = 0.072$ ,  $wR = 0.061$  for 1269 independent reflections. The benzoyl-substituted ring nitrogens are near planar while the benzamido-substituted nitrogen is pyramidal. The bonds to the remaining unsubstituted ring nitrogens, N(1) and N(5), are eclipsed with respect to each other. A Newman projection down the N(1)–N(5) axis shows a maximum of 4° deviation for the three pairs of

eclipsed bonds. Intermolecular hydrogen bonding occurs between the secondary amine and one of the benzoyl oxygens.

**Experimental.** A clear colorless  $0.05 \times 0.08 \times 0.15$  mm data crystal, crystallized from methanol. Synthesized by G. Kumar and J. Boyer of the University of Illinois at Chicago. Automated Nicolet *R3m* diffractometer with incident-beam monochromator, 20 centered reflections within  $25 \leq 2\theta \leq 50^\circ$  used for determining lattice parameters.  $[(\sin\theta)/\lambda]_{\max} = 0.53$  Å<sup>-1</sup>, range of  $hkl$ :  $0 \leq h \leq 10$ ,  $0 \leq k \leq 19$ ,