N and C(methyl) atoms of DMM, the disorder was accounted for by increase of thermal tensor U, and for each of the DMM ring C atoms by use of two fractional atoms, each with occupancy 0.5. Computations were carried out on a Cyber 170/760 with the *XRAY* (1976) system and local crystallographic programs.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71739 (24 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SH1060]

References

Becker, P. J. & Coppens, P. (1974). *Acta Cryst.* **A30**, 148–153.
Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* **A24**, 321–324.
Helmholdt, R. B. & Vos, A. (1977). *Acta Cryst.* **A33**, 456–465.
Kobayashi, H. (1974). *Bull. Chem. Soc. Jpn*, **47**, 1346–1352.
Kobayashi, H., Marumo, F. & Saito, Y. (1971). *Acta Cryst.* **B27**, 373–378.
Steurer, W., Visser, R. J. J., van Smaalen, S. & de Boer, J. L. (1987). *Acta Cryst.* **B43**, 567–574.
Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
Visser, R. J. J., Bouwmeester, H. J. M., de Boer, J. L. & Vos, A. (1990). *Acta Cryst.* **C46**, 852–856.
Visser, R. J. J., de Boer, J. L. & Vos, A. (1990). *Acta Cryst.* **C46**, 869–871.
Visser, R. J. J., van Heemstra, T. W. L. & de Boer, J. L. (1982). *Mol. Cryst. Liq. Cryst.* **85**, 265–269.
XRAY (1976). Dutch version of *The XRAY System of Crystallographic Programs.* Technical Report TR-192. Computer Science Center, Univ. of Maryland, College Park, Maryland, USA.

Acta Cryst. (1994). **C50**, 1141–1143

Indan-2,2-dicarboxylic Acid, $C_{11}H_{10}O_4$

J. Gabriel Garcia* and Joel D. Enas

Lawrence Berkeley Laboratory, UC-Berkeley, Berkeley, CA 94720, USA

Frank R. Fronczek

Department of Chemistry, Louisiana State University, Baton Rouge, Louisiana 70803-1804, USA

(*Received* 22 *April* 1993; *accepted* 29 *October* 1993)

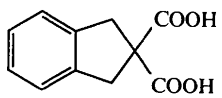
Abstract

The cyclopentene ring has an envelope conformation with the C atom carrying the carboxyl group lying 0.355 (2) Å out of the best plane of the other four C atoms. The cyclopentene torsion angle along the fusion of both rings has a magnitude of −2.5 (3)°. The carboxyl groups adopt synperiplanar conformations with C—C—C—O torsion angles of 4.5 (3) and −20.3 (3)°. The C=C distance at the ring fusion is 1.379 (3) Å. The C=O distances are 1.207 (3) and 1.215 (3) Å.

Comment

As part of an ongoing structural study of the intermediates in the synthesis of atipamezole (Garcia & Enas, 1993), indan-2,2-dicarboxylic acid was required. The title compound was prepared by condensing *α,α′*-dibromo-*o*-xylene with diethyl malonate and sodium ethoxide followed by basic hydrolysis (Carlson, Quina, Zarnegar & Whitten, 1975).

The carboxyl groups adopt synperiplanar conformations (Klyne & Prelog, 1960) with torsion angles O1—C10—C2—C3 of 4.5 (3)° and O3—C11—C2—C1 of −20.3 (3)°. Structural data for cyclobutane-1,1-dicarboxylic acid (Santarsiero, 1990), cyclopropane-1,1-dicarboxylic acid (Meester, Schenk & MacGillavry, 1971), 2,2-dimethyl-4,5-dinitroindan (Garcia, Enas & Fronczek, 1993*a*), 2-ethyl-2-nitroindan-1,3-dione (Garcia, Enas, Chang & Fronzek, 1993) and 2-acylindan-1-one (Garcia, Enas & Fronczek, 1993*b*) are in agreement with those of the title compound, (I).



(I)

The cyclopentene ring adopts an envelope conformation [torsion angles ω_1 (C1—C8—C9—C3), ω_2 (C8—C9—C3—C2), ω_3 (C9—C3—C2—C1), ω_4 (C3—C2—C1—C8) and ω_5 (C2—C1—C8—C9) of −2.5 (3), −11.7 (3), 20.5 (2), −21.9 (2) and 15.6 (3)°, respectively] that is slightly distorted from the ideal envelope conformation of cyclopentene [torsion angles $\omega_1 = 0$, $\omega_2 = -15$, $\omega_3 = 24$, $\omega_4 = -24$ and $\omega_5 = 15°$ (Bartlett, Kimura, Nakayama & Watson, 1979)].

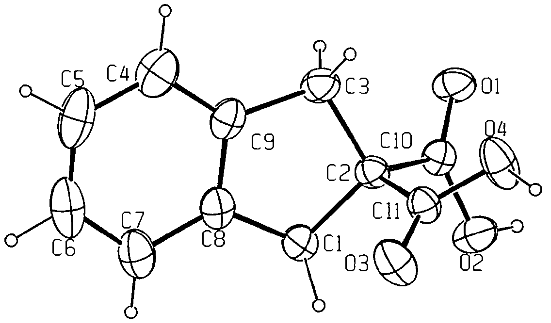


Fig. 1. *ORTEP* drawing (Johnson, 1965) of the title molecule representing heavy atoms as 40% probability ellipsoids and H atoms as circles of arbitrary radii.

Acta Crystallographica Section C
ISSN 0108-2701 © 1994

Both carboxyl groups engage in normal hydrogen-bonded dimer formation about centers of symmetry. The carboxyl group involving O1 and O2 related by the inversion center at the origin has an O···O distance of 2.669 (2) Å and an angle about H of 172 (3)°. That involving O3 and O4 related by an inversion center at $\frac{1}{2},\frac{1}{2},0$ has an O···O distance of 2.656 (3) Å and an angle about H of 173 (3)°. Thus, molecules are arranged in hydrogen-bonded chains in the [$\bar{1}$10] direction as illustrated in Fig. 2.

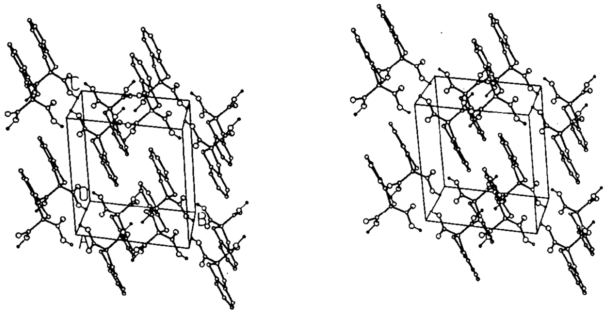


Fig. 2. Stereoview of the unit cell illustrating the hydrogen bonding. H atoms not involved in hydrogen bonding are omitted.

Experimental

Crystal data

$C_{11}H_{10}O_4$
$M_r = 206.2$
Triclinic
$P\bar{1}$
$a = 5.8127$ (4) Å
$b = 9.3602$ (7) Å
$c = 9.5880$ (5) Å
$\alpha = 98.172$ (5)°
$\beta = 106.609$ (5)°
$\gamma = 90.770$ (6)°
$V = 494.0$ (2) Å^3
$Z = 2$
$D_x = 1.384$ Mg m^{-3}
M.p. = 454-456 K (sub.)

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 8$-12°
$\mu = 0.10$ mm^{-1}
$T = 294$ K
Lath
0.55 × 0.10 × 0.05 mm
Colorless
Crystal source: crystallized from ethanol

Data collection

Enraf-Nonius CAD-4 diffractometer
ω-2θ scans
Absorption correction: none
1729 measured reflections
1729 independent reflections
1219 observed reflections [$I > \sigma(I)$]

$\theta_{max} = 25°$
$h = 0 \rightarrow 6$
$k = -11 \rightarrow 11$
$l = -11 \rightarrow 10$
3 standard reflections
frequency: 166 min
intensity variation: 2.4%

Refinement

Refinement on F
$R = 0.045$
$wR = 0.044$
$S = 1.453$
1219 reflections
177 parameters
All H-atom parameters refined
$w = 4F_o^2/[\sigma^2(I) + (0.02F_o^2)^2]$
$(\Delta/\sigma)_{max} = 0.02$

$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.16$ e Å^{-3}
Extinction correction: $(1 + gI_c)^{-1}$ applied to F_c
Extinction coefficient: $g = 1.3$ (3) × 10^{-6}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
O1	1.0170 (3)	0.1072 (2)	0.8816 (2)	3.55 (4)
O2	0.6949 (3)	0.0634 (2)	0.9537 (2)	3.82 (4)
O3	0.3794 (3)	0.3897 (2)	0.8450 (2)	4.04 (4)
O4	0.7558 (3)	0.3961 (2)	0.9879 (2)	4.41 (4)
C1	0.4184 (4)	0.1283 (2)	0.6745 (2)	2.72 (5)
C2	0.6462 (4)	0.2172 (2)	0.7769 (2)	2.40 (5)
C3	0.7664 (4)	0.2804 (4)	0.6719 (3)	3.25 (5)
C4	0.5603 (5)	0.3363 (3)	0.4077 (3)	4.29 (6)
C5	0.3530 (6)	0.3153 (3)	0.2888 (3)	5.24 (8)
C6	0.1561 (6)	0.2371 (3)	0.2947 (3)	5.12 (7)
C7	0.1611 (5)	0.1728 (3)	0.4156 (3)	3.91 (6)
C8	0.3685 (4)	0.1914 (3)	0.5345 (2)	2.99 (5)
C9	0.5648 (4)	0.2736 (3)	0.5310 (2)	3.04 (5)
C10	0.8094 (4)	0.1240 (2)	0.8765 (2)	2.57 (5)
C11	0.5780 (4)	0.3419 (2)	0.8741 (2)	2.70 (5)

Table 2. *Selected geometric parameters* (Å, °)

O1—C10	1.207 (3)	C2—C11	1.518 (3)
O2—C10	1.305 (3)	C3—C9	1.507 (3)
O3—C11	1.215 (3)	C4—C5	1.391 (3)
O4—C11	1.303 (2)	C4—C9	1.386 (4)
C1—C2	1.551 (3)	C5—C6	1.370 (5)
C1—C8	1.497 (3)	C6—C7	1.373 (4)
C2—C3	1.553 (4)	C7—C8	1.391 (3)
C2—C10	1.522 (3)	C8—C9	1.379 (3)
C2—C1—C8	103.8 (2)	C1—C8—C7	128.3 (2)
C1—C2—C3	105.2 (2)	C1—C8—C9	111.2 (2)
C1—C2—C10	111.2 (2)	C7—C8—C9	120.4 (2)
C1—C2—C11	110.7 (2)	C3—C9—C4	128.3 (2)
C3—C2—C10	113.4 (2)	C3—C9—C8	111.2 (2)
C3—C2—C11	108.4 (2)	C4—C9—C8	120.5 (2)
C10—C2—C11	108.0 (2)	O1—C10—O2	124.7 (2)
C2—C3—C9	103.6 (2)	O1—C10—C2	124.1 (2)
C5—C4—C9	118.5 (3)	O2—C10—C2	111.3 (2)
C4—C5—C6	120.7 (3)	O3—C11—O4	123.6 (2)
C5—C6—C7	121.0 (2)	O3—C11—C2	123.0 (2)
C6—C7—C8	118.8 (3)	O4—C11—C2	113.4 (2)
C7—C8—C9—C4	−1.4 (4)	C10—C2—C11—O3	−142.2 (2)
C10—C2—C11—O4	40.5 (3)	C11—C2—C10—O1	−115.6 (2)
C11—C2—C10—O2	65.3 (2)		

Programs used include *MolEN* (Fair, 1990), *RANTAN* (Yao, 1981) and *ORTEP* (Johnson, 1965).

We thank DOE for support of this research through grant No. DE-AC03-76SF00098.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and geometry, least-squares-planes data and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71829 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR1076]

References

Bartlett, P. D., Kimura, M., Nakayama, J. & Watson, W. H. (1979). *J. Am. Chem. Soc.* **101**, 6332–6337.
Carlson, G. L. B., Quina, F. H., Zarnegar, B. M. & Whitten, D. G. (1975). *J. Am. Chem. Soc.* **97**, 347–354.
Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis.* Enraf–Nonius, Delft, The Netherlands.
Garcia, J. G. & Enas, J. D. (1993). Unpublished results.
Garcia, J. G., Enas, J. D., Chang, E. & Fronczek, F. R. (1993). *Acta Cryst.* **C49**, 1401–1402.
Garcia, J. G., Enas, J. D. & Fronczek, F. R. (1993*a*). *Acta Cryst.* Submitted.
Garcia, J. G., Enas, J. D. & Fronczek, F. R. (1993*b*). *Acta Cryst.* **C49**, 1823–1824.
Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
Klyne, W. & Prelog, V. (1960). *Experientia*, **16**, 521–523.
Meester, M. A. M., Schenk, H. & MacGillavry, C. H. (1971). *Acta Cryst.* **B27**, 630–634.
Santarsiero, B. D. (1990). *J. Chem. Phys.* **92**, 2794–3797.
Yao, J.-X. (1981). *Acta Cryst.* **A37**, 642–644.

Acta Cryst. (1994). **C50**, 1143–1145

A Chromotropic Bi(benzofuranonyl)

Yukie Mori, Ikuko Kubota, Yuka Watanabe
and Koko Maeda*

*Department of Chemistry, Faculty of Science,
Ochanomizu University, Otsuka, Bunkyo-ku,
Tokyo 112, Japan*

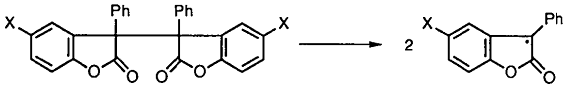
(*Received* 15 *June* 1993; *accepted* 1 *November* 1993)

Abstract

The structure of a chromotropic compound, 5,5′-dichloro-3,3′-diphenyl-3,3′-bi(1-benzofuran)-2,2′-(3*H*,3′*H*)-dione (I), $C_{28}H_{16}Cl_2O_4$, has been determined. The C(3)—C(3′) bond, which undergoes homolysis by mechanical energy such as rubbing, is lengthened to 1.624 (4) Å compared with the average hexa-substituted C_{sp^3}—C_{sp^3} bond length. In the crystal, the molecule exists in a nonsymmetric *gauche* conformation.

Comment

3,3′-Diphenyl-3,3′-bi(1-benzofuran)-2,2′(3*H*,3′*H*)-dione (II) was reported to exhibit piezochromism† in the solid state (Löwenbein & Schmitt, 1927). Recently it was revealed that this mechanochromic phenomenon was responsible for the reversible cleavage of the C(3)—C(3′) bond to give blue-coloured benzofuranonyl radicals (Watanabe, 1987). There have been few reports on organic mechanochromic compounds and investigations from the structural chemistry viewpoint are very limited (Ohkanda, Mori, Maeda & Osawa, 1992) since these compounds were obtained only as powdery solids.



(I) $X = Cl$
(II) $X = H$

Compound (I), the 5-chloro derivative of (II), also exhibited mechanochromism and gave a single crystal suitable for X-ray structure analysis. The C(3)—C(3′) bond is lengthened to 1.624 (4) Å compared with the average length (1.588 Å) of hexa-substituted C_{sp^3}—C_{sp^3} bonds (Allen *et al.*, 1987). This bond elongation seems to be caused by steric repulsion and through-bond interactions between the phenyl groups at the 3 and 3′ positions. In the crystal, the molecule is not in a centrosymmetric *anti* conformation but is instead in a nonsymmetric *gauche* conformation (Fig. 2). The corresponding bond angles around C(3) and C(3′) are somewhat different from each other, while bond distances and other angles of the two halves of the molecule coincide within experimental error.

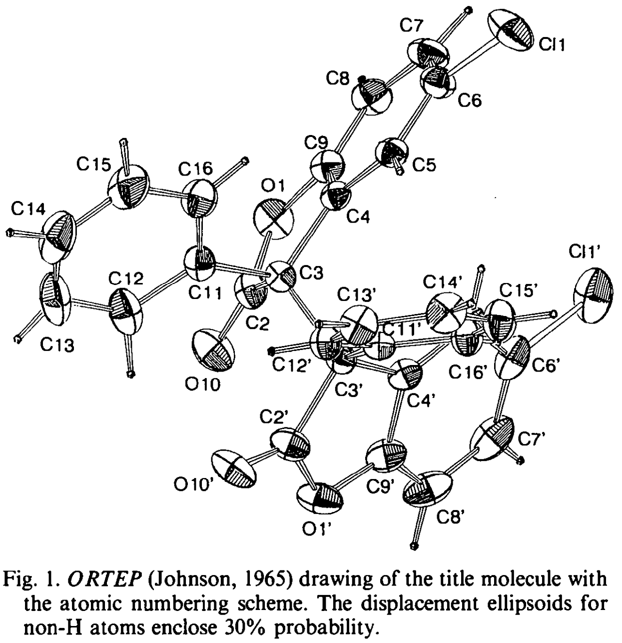


Fig. 1. *ORTEP* (Johnson, 1965) drawing of the title molecule with the atomic numbering scheme. The displacement ellipsoids for non-H atoms enclose 30% probability.

---

† We proposed a term 'mechanochromism' instead of piezochromism based on the fact that the mechanochemical reaction is caused by rubbing rather than high pressure (Ohkanda, Mori, Maeda & Osawa, 1992).