

## References

- Allen, F. H., Kennard, O. & Taylor, R. (1983). *Acc. Chem. Res.* **16**, 146–153.
- Edsall, J. T., Flory, P. J., Kendrew, J. C., Liquori, A. M., Nemethy, G., Ramachandran, G. N. & Scheraga, H. A. (1966). *J. Mol. Biol.* **15**, 399–407.
- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
- Ramanadham, M. & Chidambaram, R. (1978). *Advances in Crystallography, Amino Acids: Systematics of Molecular Structure, Conformation and Hydrogen Bonding*, pp. 81–103. New Delhi: Oxford and IBH Publishing.
- Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures*. Univ. of Göttingen, Germany.
- Sheldrick, G. M. (1991). *SHELXTL-Plus*. Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Sheldrick, G. M. (1993). *SHELXL93. Program for Crystal Structure Refinement*. Univ. of Göttingen, Germany.
- Siemens (1992). *XSCANS*. Version 2.0. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

*Acta Cryst.* (1995). **C51**, 2720–2722

## Xanthyletin†

ENRIQUE RUDIÑO-PIÑERA, GABRIELA JUÁREZ-MARTÍNEZ, KALIYAMOORTHY PANNEERSELVAM AND MANUEL SORIANO-GARCÍA\*

*Instituto de Química, Circuito Exterior, Ciudad Universitaria, Delegación Coyoacán, México DF 04510, México*

ANA LUISA ANAYA AND CLARA GARCÍA-SANTANA

*Instituto de Fisiología Celular, Circuito Exterior, Ciudad Universitaria, Delegación Coyoacán, México DF 04510, México*

RACHEL MATA AND PERLA SÁNCHEZ

*Facultad de Química, Circuito Exterior, Ciudad Universitaria, Delegación Coyoacán, México DF 04510, México*

(Received 12 May 1995; accepted 7 July 1995)

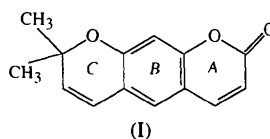
## Abstract

The present X-ray diffraction study establishes the molecular structure of the title compound, 8,8-dimethyl-2*H*,8*H*-benzo[1,2-*b*:5,4-*b'*]dipyran-2-one, C<sub>14</sub>H<sub>12</sub>O<sub>3</sub>. The molecule consists of three six-membered rings. Ring C has a screw-boat conformation, whereas rings A and B are planar. The crystal structure is stabilized by van der Waals forces and there are three C—H···O intermolecular interactions.

† Contribution No. 1326 of the Instituto de Química, UNAM.

## Comment

Xanthyletin, (I), was isolated from the roots of *Stauranthus perforatus* (*Rutaceae*) and collected in Felipe Carrillo Puerto, Quintana Roo, México. The roots are used locally for the treatment of rheumatism. The preparation of crystals of (I) is described in the *Experimental* section.



A view of the title molecule with the atom-numbering scheme is shown in Fig. 1. The molecule consists of three six-membered rings A (C1–C4, C12, O1), B (C4–C6, C10–C12) and C (C6–C9, O2, C10). Bond lengths and angles are quite similar to those of related compounds such as amyrolin (Kato, 1970). Rings A and

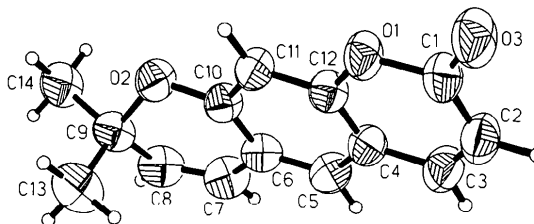


Fig. 1. The molecular structure of the title compound showing the atom-labelling scheme and 50% probability displacement ellipsoids.

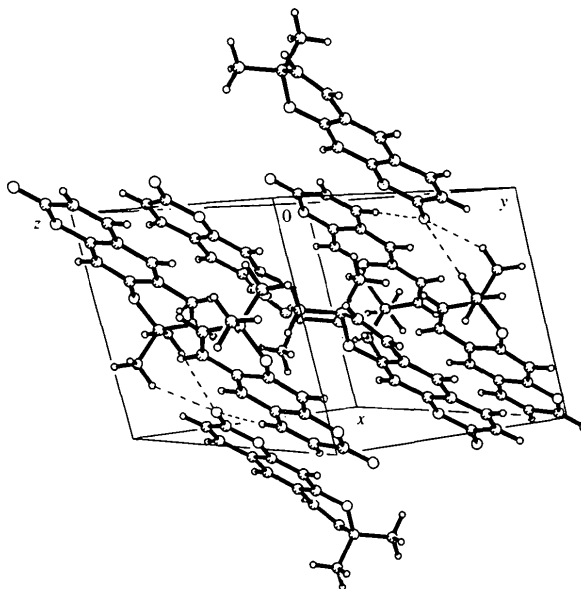


Fig. 2. A unit-cell drawing of the packing arrangement with dashed lines indicating C—H···O intermolecular interactions.

*B* are planar, with a maximum deviation of 0.01 Å for both rings. According to the puckering parameters [ $\varphi_2 = 26(3)$ ,  $\theta_2 = 114(2)^\circ$  and  $Q = 0.345(3)$  Å], ring *C* has a screw-boat conformation (Boeyens, 1978). The interplanar angles between the pairs of rings *A/C* and *B/C* are 13(3) and 10(3)°, respectively.

The crystal structure (Fig. 2) is stabilized by van der Waals forces. There are three C—H...O intermolecular interactions (Desiraju, 1991) within the range 3.3–3.6 Å and their dimensions are given in Table 3.

## Experimental

The air-dried milled material (2.62 kg) was extracted exhaustively by maceration at room temperature with CHCl<sub>3</sub>/MeOH (1:1). This solution, upon evaporation under low pressure, yielded 232.8 g of extract. The crude extract was chromatographed over 1 kg of silica gel in a gravity column using a gradient of hexane/EtOAc/MeOH. 2.0 g of xanthyletin was crystallized from combined fractions 40–41 (eluted with hexane/EtOAc 85:15) and recrystallized from benzene/EtOAc solution.

### Crystal data

C<sub>14</sub>H<sub>12</sub>O<sub>3</sub>  
*M<sub>r</sub>* = 228.24  
 Monoclinic  
*P*2<sub>1</sub>/*c*  
*a* = 8.830 (1) Å  
*b* = 11.658 (2) Å  
*c* = 11.492 (2) Å  
 $\beta$  = 104.25 (1)°  
*V* = 1146.6 (3) Å<sup>3</sup>  
*Z* = 4  
*D<sub>x</sub>* = 1.322 Mg m<sup>-3</sup>

### Data collection

Siemens P4 diffractometer  
 $\theta/2\theta$  scans  
 Absorption correction:  
 none  
 2083 measured reflections  
 1524 independent reflections  
 1313 observed reflections  
 [*I* > 2 $\sigma$ (*I*)]  
*R*<sub>int</sub> = 0.0231

### Refinement

Refinement on *F*<sup>2</sup>  
*R*(*F*) = 0.0440  
*wR*(*F*<sup>2</sup>) = 0.1259  
*S* = 1.129  
 1524 reflections  
 202 parameters  
 All H atoms were refined isotropically

Cu K $\alpha$  radiation  
 $\lambda$  = 1.54178 Å  
 Cell parameters from 25 reflections  
 $\theta$  = 15–30°  
 $\mu$  = 0.760 mm<sup>-1</sup>  
*T* = 293 (2) K  
 Trigonal  
 0.3 × 0.25 × 0.2 mm  
 Colourless

$\theta_{\max}$  = 56.61°  
*h* = -9 → 9  
*k* = -1 → 12  
*l* = -1 → 12  
 3 standard reflections monitored every 100 reflections  
 intensity decay: 3%

$w = 1/[\sigma^2(F_o^2) + (0.0782P)^2 + 0.1280P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.023$   
 $\Delta\rho_{\max} = 0.137$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.202$  e Å<sup>-3</sup>  
 Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å<sup>2</sup>)

$$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i\cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> <sub>eq</sub>
C1	-0.0350 (2)	0.2841 (2)	0.2747 (2)	0.0570 (5)
C2	-0.0234 (2)	0.4056 (2)	0.2618 (2)	0.0672 (6)
C3	0.0559 (2)	0.4524 (2)	0.1888 (2)	0.0647 (6)
C4	0.1361 (2)	0.3811 (2)	0.1218 (2)	0.0510 (5)
C5	0.2252 (2)	0.4223 (2)	0.0451 (2)	0.0571 (5)
C6	0.3077 (2)	0.3499 (2)	-0.0112 (2)	0.0530 (5)
C7	0.4013 (3)	0.3866 (2)	-0.0928 (2)	0.0660 (6)
C8	0.4972 (3)	0.3142 (2)	-0.1258 (2)	0.0662 (6)
C9	0.5192 (2)	0.1947 (2)	-0.0775 (2)	0.0577 (5)
C10	0.3000 (2)	0.2317 (2)	0.0095 (2)	0.0497 (5)
C11	0.2094 (2)	0.1877 (2)	0.0812 (2)	0.0519 (5)
C12	0.1299 (2)	0.2632 (2)	0.1369 (2)	0.0481 (5)
C13	0.6517 (3)	0.1893 (3)	0.0347 (2)	0.0800 (8)
C14	0.5421 (3)	0.1092 (2)	-0.1705 (2)	0.0698 (7)
O1	0.0444 (1)	0.2163 (1)	0.2107 (1)	0.0556 (4)
O2	0.3760 (2)	0.1552 (1)	-0.0460 (1)	0.0606 (4)
O3	-0.1080 (2)	0.2337 (1)	0.3355 (1)	0.0720 (5)

Table 2. Selected geometric parameters (Å, °)

C1—O3	1.213 (2)	C7—C8	1.316 (3)
C1—O1	1.382 (2)	C8—C9	1.494 (3)
C1—C2	1.430 (3)	C9—O2	1.473 (2)
C2—C3	1.335 (3)	C9—C14	1.510 (3)
C3—C4	1.433 (3)	C9—C13	1.514 (3)
C4—C12	1.388 (3)	C10—O2	1.365 (2)
C4—C5	1.403 (3)	C10—C11	1.381 (2)
C5—C6	1.377 (3)	C11—C12	1.379 (2)
C6—C10	1.403 (3)	C12—O1	1.380 (2)
C6—C7	1.458 (3)		
O3—C1—O1	116.1 (2)	O2—C9—C14	103.9 (2)
O3—C1—C2	127.0 (2)	C8—C9—C14	112.4 (2)
O1—C1—C2	116.9 (2)	O2—C9—C13	107.9 (2)
C3—C2—C1	122.2 (2)	C8—C9—C13	111.0 (2)
C2—C3—C4	120.4 (2)	C14—C9—C13	111.4 (2)
C12—C4—C5	117.6 (2)	O2—C10—C11	117.3 (2)
C12—C4—C3	117.8 (2)	O2—C10—C6	120.8 (2)
C5—C4—C3	124.5 (2)	C11—C10—C6	121.7 (2)
C6—C5—C4	121.9 (2)	C10—C11—C12	118.4 (2)
C5—C6—C10	118.0 (2)	O1—C12—C11	116.8 (2)
C5—C6—C7	124.8 (2)	O1—C12—C4	120.9 (2)
C10—C6—C7	117.2 (2)	C11—C12—C4	122.3 (2)
C8—C7—C6	120.2 (2)	C12—O1—C1	121.7 (2)
C7—C8—C9	121.7 (2)	C10—O2—C9	117.5 (1)
O2—C9—C8	110.0 (2)		

Table 3. Hydrogen-bonding geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C3—H3...O3'	0.94 (3)	2.39 (3)	3.333 (3)	176 (2)
C13—H131...O3''	0.99 (3)	2.74 (3)	3.597 (3)	145 (2)
C14—H142...O3'''	0.99 (2)	2.69 (2)	3.578 (3)	149 (2)

Symmetry codes: (i)  $-x, \frac{1}{2} + y, \frac{1}{2} - z$ ; (ii)  $1 + x, \frac{1}{2} - y, z - \frac{1}{2}$ .

Data collection: P4 diffractometer software. Cell refinement: XSCANS (Siemens, 1992). Data reduction: XSCANS. Program(s) used to solve structure: SHELXS86 (Sheldrick, 1985). Program(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Molecular graphics: SHELXTL-Plus (Sheldrick, 1990). Software used to prepare material for publication: SHELXL93.

KP acknowledges the CONACYT (Cátedra Patrimonial Nivel II) for fellowship assistance. The authors thank the Instituto de Biotecnología, UNAM, for data collection and Miss T. Hernández for growing the crystals.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: KA1139). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

## References

- Boeyens, J. C. A. (1978). *J. Cryst. Mol. Struct.* **8**, 317–320.  
 Desiraju, G. R. (1991). *Acc. Chem. Res.* **24**, 290–296.  
 Kato, V. K. (1970). *Acta Cryst.* **B26**, 2022–2029.  
 Sheldrick, G. M. (1985). *SHELXS86. Crystallographic Computing 3*, edited by G. M. Sheldrick, C. Krüger & R. Goddard, pp. 175–189. Oxford Univ. Press.  
 Sheldrick, G. M. (1990). *SHELXTL-Plus Structure Determination Software Programs*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.  
 Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. Univ. of Göttingen, Germany.  
 Siemens (1992). *XSCANS. X-ray Single Crystal Analysis System*. Version 2.0. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

*Acta Cryst.* (1995). **C51**, 2722–2723

## *N*-Methyl-1,1,4-triphenyl-1,2-dihydronaphthalene-2,3-dicarboximide

FUMIO TODA AND KOICHI TANAKA

*Department of Applied Chemistry, Faculty of Engineering, Ehime University, Matsuyama, Ehime 790, Japan*

ZAFRA STEIN AND ISRAEL GOLDBERG

*School of Chemistry, Sackler Faculty of Exact Sciences, Tel-Aviv University, Ramat-Aviv, 69978 Tel-Aviv, Israel*

(Received 11 May 1995; accepted 7 July 1995)

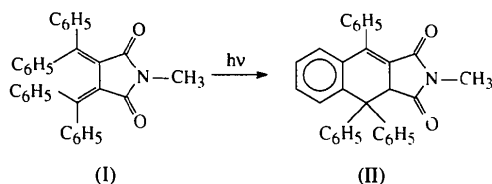
## Abstract

The stereochemistry of the molecular product, C<sub>31</sub>H<sub>23</sub>NO<sub>2</sub>, obtained by photoirradiation of 3,4-bis(diphenylmethylene)-*N*-methylsuccinimide in the solid state, together with the position of the chiral center in the molecule, have been confirmed by X-ray crystal structure analysis.

## Comment

The structure of the title compound, *N*-methyl-1,1,4-triphenyl-1,2-dihydronaphthalene-2,3-dicarboximide, (II), was studied in order to confirm the chemical identity

and stereochemistry of the product obtained by photoirradiation of powdered 3,4-bis(diphenylmethylene)-*N*-methylsuccinimide, (I), in the solid state. In particular, a question was raised with regard to the position of the double bond within the ring system of (II), in view of a possible 1,3-shift isomerization (*i.e.* movement of the chiral center from C5 to C22). It was also of interest to correlate the topological features of the crystalline reactant and product associated with this reaction.



The topochemistry of (I) is characterized by both a helical conformation (due to severe steric hindrance between the phenyl groups) and the close proximity of unsaturated fragments, which facilitates an intramolecular photocyclization reaction in the solid state. Studies of the structure and solid-state photochemistry of chiral and racemic crystalline polymorphs of (I) have been reported elsewhere (Toda, Tanaka, Stein & Goldberg, 1995). It has been shown that irradiation of the chiral crystalline form of (I) yields optically active (II), while irradiation of racemic (I) leads to the formation of racemic crystals of (II). We report here on the detailed structure of racemic (II). Suitable crystals of the optically pure enantiomorph of (II) could not be obtained.

The present analysis indicates clearly that all C—C distances within the five-membered ring correspond to single bonds, with values in the range 1.481(4)–1.514(4) Å. The double bond is located adjacent to the

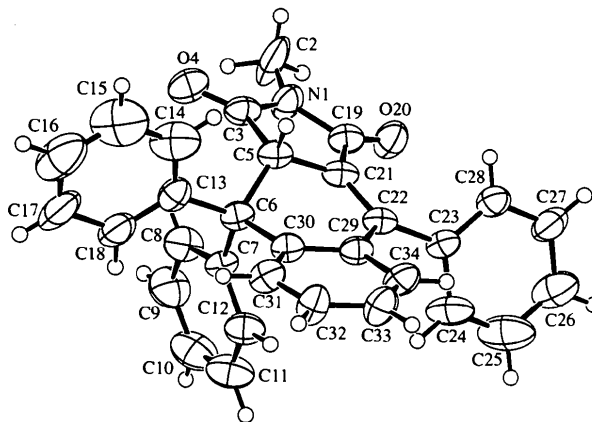


Fig. 1. The molecular structure of compound (II) showing 50% probability displacement ellipsoids and the labelling of the non-H atoms.