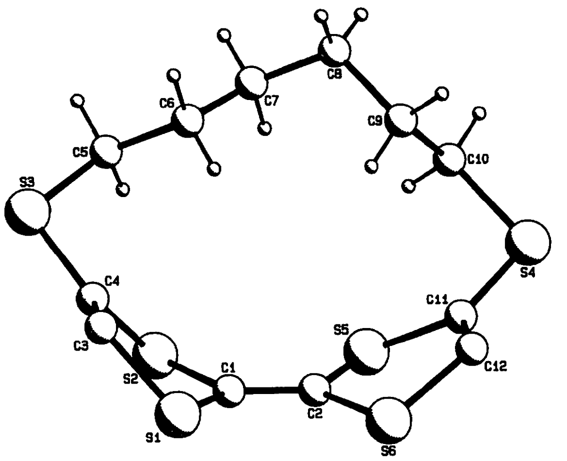


Fig. 2. Side view of the molecule (*PLUTO*; Motherwell & Clegg, 1978). The *p*-tolyl substituents have been omitted for clarity.

in the TTF molecule [1·729 (2) to 1·732 Å] (Cooper, Kenny, Edmonds, Nagel, Wudl & Coppens, 1971). The average S—C_{sp^3} bond length of 1·821 (4) Å is in agreement with the sum of single-bond radii (1·812 Å) (Pauling, 1960) as well as with the previously reported value [1·802 (4) Å] for 3,4′-dimethyl-3′4-bis(methylthio)-2,2′,5,5′-tetrathiafulvalene (Ouahab & Batail, 1985).

References

Bertho, F., Robert, A., Batail, P. & Robin, P. (1990). *Tetrahedron*, **46**, 433–444.

Bertho-Thoraval, F. (1988). Thèse Doctorat, Univ. Rennes 1, France.

Cooper, W. F., Kenny, N. C., Edmond, W. J., Nagel, A., Wudl, F. & Coppens, P. (1971). *J. Chem. Soc. Chem. Commun.* pp. 889–890.

Frenz, B. A. (1983). *Enraf–Nonius Structure Determination Package*; *SDP Users Guide*. Version of 6 January 1983. Enraf–Nonius, Delft, The Netherlands.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1982). *MULTAN*11/82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

Ouahab, L. & Batail, P. (1985). *Acta Cryst.* C**41**, 928–929.

Pauling, L. (1960). *The Nature of the Chemical Bond*. Ithaca: Cornell Univ. Press.

Staab, H. A., Ippen, J., Tao-pen, C., Krieger, C. & Starker, B. (1980). *Angew. Chem. Int. Ed. Engl.* **19**, 66–67.

Acta Cryst. (1991). C**47**, 1111–1113

Structure of 6,7-Dimethoxy-2,2-dimethyl-2*H*-chromene, a Natural Precocene

By J. Zukerman-Schpector, C. A. De Simone, E. E. Castellano and G. Oliva

Instituto de Física e Química de São Carlos, Universidade de São Paulo, Caixa Postal 369,
13560 *São Carlos – SP, Brazil*

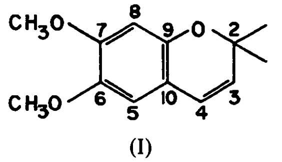
and J. Tercio, B. Ferreira and V. Catani

Departamento de Química, Universidade Federal de São Carlos, Caixa Postal 676,
13560 *São Carlos – SP, Brazil*

(*Received* 1 *October* 1990; *accepted* 23 *October* 1990)

Abstract. $C_{13}H_{16}O_3$, $M_r = 220·3$, orthorhombic, $Pca2_1$, $a = 14·358$ (2), $b = 9·297$ (1), $c = 9·011$ (1) Å, $V = 1202·9$ (4) Å^3, $Z = 4$, $D_x = 1·216$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0·80$ mm^{-1}, $F(000) = 472$, $T = 298$ K, $R = 0·036$ for 866 observed reflections. The heterocyclic ring is in a distorted sofa conformation, and the C—O bonds are inequivalent: C(2)—O(1) = 1·463 (4) Å and C(9)—O(1) = 1·379 (4) Å.

Experimental. Crystals of compound (I) were obtained from *n*-hexane at 277 K. The data collection and refinement parameters are summarized in Table 1.



(I)

The structure was solved using standard direct methods and difference Fourier synthesis techniques. In the final cycles of full-matrix least-squares

0108-2701/91/051111-03$03.00

Table 1. *Crystallographic summary for* (I)

Data collection^{i,ii}	
Mode	ω–2θ
Scan rate (° min^{-1})	2·8–10·0
θ range (°)	0–25
Range of *hkl*	$0 \le h \le 10$, $0 \le k \le 11$, $-2 \le l \le 17$
Total reflections measured	1304
Unique reflections	1113
$R_{\rm int}$	0·013
Crystal dimensions approx. (mm)	0·40 × 0·38 × 0·35
Structure determination^{iii}	
Reflections used [$I > 3\sigma(I)$]	866
Number of variables	145
R, *wR*	0·036, 0·041
Max. shift/e.s.d.	0·004
Max., min. density in final difference map (e Å^{-3})	0·12, −0·12
S	1·62

Notes: (i) Unit-cell parameters by least-squares refinement of the setting angles of 25 reflections with $11 < \theta < 24°$. (ii) Enraf–Nonius CAD-4 diffractometer with a graphite monochromator. Two standard reflections (006, 0012), measured every hour, showed no significant variation. (iii) Function minimized was $\sum w(|F_o| - |F_c|)^2$, where $w^{-1} = [\sigma^2(F_o) + 0{\cdot}0005F_o^2]$.

refinement on *F*'s all non-H atoms were treated anisotropically. H atoms included, as fixed contributors, at positions found in difference synthesis, all with a common isotropic temperature factor that refined to $U = 0{\cdot}078\,(3)$ Å^2. Scattering factors for non-H atoms were taken from Cromer & Mann (1968) with corrections for anomalous dispersion taken from Cromer & Liberman (1970), for H atoms were taken from Stewart, Davidson & Simpson (1965). Programs used: *SHELX*76 (Sheldrick, 1976) and *ORTEP* (Johnson, 1965). Most of the calculations were performed on a VAX 11/780 computer of the Instituto de Física e Química de São Carlos.

Atomic coordinates are listed in Table 2,* bond lengths and angles are listed in Table 3. The shortest intermolecular distance is O(3)···C(13)($\frac{1}{2}-x$, y, $\frac{1}{2}+z$) = 3·131 (5) Å. Fig. 1 is a stereoscopic projection of the molecule.

Table 2. *Final atomic coordinates and equivalent isotropic temperature factors* (Å^2) *with e.s.d.'s in parentheses*

$$B_{\rm eq} = (4/3)\textstyle\sum_i\sum_j \beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	$B_{\rm iso}$
O(1)	−0·1292 (1)	0·8597 (2)	0·5	4·60 (8)
O(2)	0·2036 (1)	0·6349 (2)	0·6991 (3)	4·94 (8)
O(3)	0·0581 (1)	0·6278 (2)	0·8675 (3)	4·93 (8)
C(2)	−0·1505 (2)	0·8673 (3)	0·3414 (4)	4·2 (1)
C(3)	−0·0647 (3)	0·9096 (3)	0·2562 (4)	4·4 (1)
C(4)	0·0199 (2)	0·8783 (3)	0·3042 (4)	4·2 (1)
C(5)	0·1163 (2)	0·7491 (3)	0·4987 (3)	3·64 (9)
C(6)	0·1236 (2)	0·6920 (3)	0·6382 (4)	3·6 (1)
C(7)	0·0450 (2)	0·6882 (3)	0·7310 (4)	3·8 (1)
C(8)	−0·0391 (2)	0·7410 (3)	0·6807 (4)	3·7 (1)
C(9)	−0·0450 (2)	0·7993 (3)	0·5391 (4)	3·7 (1)
C(10)	0·0312 (2)	0·8041 (3)	0·4470 (3)	3·5 (1)
C(11)	−0·1843 (2)	0·7214 (3)	0·2940 (4)	5·2 (1)
C(12)	−0·2267 (3)	0·9759 (3)	0·3289 (5)	6·1 (1)
C(13)	0·2826 (2)	0·6248 (4)	0·6048 (5)	5·8 (1)
C(14)	−0·0190 (3)	0·6192 (4)	0·9635 (4)	5·8 (1)

Table 3. *Interatomic bond distances* (Å) *and angles* (°)

O(1)—C(2)	1·463 (4)	O(1)—C(9)	1·379 (4)
O(2)—C(6)	1·379 (4)	O(2)—C(13)	1·420 (4)
O(3)—C(7)	1·365 (4)	O(3)—C(14)	1·407 (5)
C(2)—C(3)	1·504 (5)	C(2)—C(11)	1·503 (5)
C(2)—C(12)	1·493 (5)	C(3)—C(4)	1·322 (5)
C(4)—C(10)	1·469 (5)	C(5)—C(6)	1·369 (5)
C(5)—C(10)	1·404 (4)	C(6)—C(7)	1·405 (4)
C(7)—C(8)	1·380 (4)	C(8)—C(9)	1·389 (5)
C(9)—C(10)	1·374 (4)		
C(2)—O(1)—C(9)	116·9 (2)	C(6)—O(2)—C(13)	116·9 (2)
C(7)—O(3)—C(14)	118·0 (3)	O(1)—C(2)—C(3)	109·9 (3)
O(1)—C(2)—C(11)	107·6 (3)	O(1)—C(2)—C(12)	105·0 (3)
C(3)—C(2)—C(11)	110·8 (3)	C(3)—C(2)—C(12)	112·6 (3)
C(11)—C(2)—C(12)	110·6 (3)	C(2)—C(3)—C(4)	121·9 (3)
C(3)—C(4)—C(10)	119·4 (3)	C(6)—C(5)—C(10)	120·8 (3)
O(2)—C(6)—C(5)	125·3 (3)	O(2)—C(6)—C(7)	115·0 (3)
C(5)—C(6)—C(7)	119·7 (3)	O(3)—C(7)—C(6)	115·8 (3)
O(3)—C(7)—C(8)	124·2 (3)	C(6)—C(7)—C(8)	119·9 (3)
C(7)—C(8)—C(9)	119·6 (3)	O(1)—C(9)—C(8)	116·6 (3)
O(1)—C(9)—C(10)	122·0 (3)	C(8)—C(9)—C(10)	121·3 (3)
C(4)—C(10)—C(5)	123·9 (3)	C(4)—C(10)—C(9)	117·2 (3)
C(5)—C(10)—C(9)	118·7 (3)		

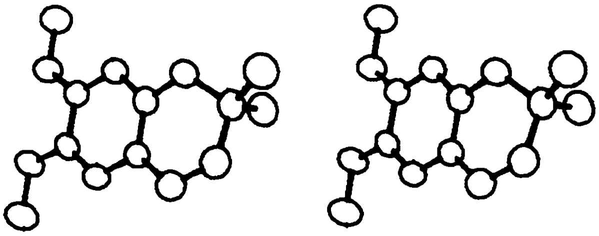


Fig. 1. Stereoscopic projection of the molecule.

Related literature. The C—O bonds in the heterocyclic ring are different [C(2)—O(1) = 1·463 (4), C(9)—O(1) = 1·379 (4) Å] owing to the effect of conjugation on the C(9) side. The conformation of the heterocycle is a distorted sofa; C(3) and C(4) are coplanar, to within experimental accuracy, with the C(5)—C(10) benzene ring, whereas C(2) is 0·418 (3) Å above and O(1) is only 0·116 (2) Å below that plane. The C(9)—O(1)—C(2)—C(3) torsion angle is −40·8 (3)°. The O(1)—C(2)—C(3) angle of 109·9 (3)° is closely similar to those found by Flippen, Karle & Karle (1970), Spek, Kojić-Prodić & Labadie (1984), Rao, Seshadri & Rao (1987) and Valente, Eggleston & Schomaker (1987).

This work has received partial support from CAPES, CNPq, FAPESP and FINEP.

---

* 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 53679 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Cromer, D. T. & Liberman, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.

Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* **A24**, 321–324.

Flippen, J. L., Karle, J. & Karle, I. L. (1970). *J. Am. Chem. Soc.* **92**, 3749–3754.

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

Rao, B. H. L., Seshadri, T. P. & Rao, L. M. (1987). *Acta Cryst.* **C43**, 495–497.

Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Spek, A. L., Kojić-Prodić, B. & Labadie, R. P. (1984). *Acta Cryst.* **C40**, 2068–2071.

Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Valente, E. J., Eggleston, D. S. & Schomaker, V. (1987). *Acta Cryst.* **C43**, 533–536.

Acta Cryst. (1991). **C47**, 1113–1114

Structure of Phenazine

By K. Woźniak

Department of Chemistry, Warsaw University, Pasteura 1, 02-093 Warszawa, Poland

and B. Kariuki and W. Jones

Department of Chemistry, Cambridge University, Lensfield Road, Cambridge CB2 1EW, England

(*Received* 28 *August* 1990; *accepted* 23 *October* 1990)

Abstract. $C_{12}H_8N_2$, $M_r = 180{\cdot}21$, monoclinic, $P2_1/n$, $a = 7{\cdot}083\,(1)$, $b = 5{\cdot}072\,(1)$, $c = 12{\cdot}794\,(8)$ Å, $\beta = 102{\cdot}34\,(2)°$, $V = 449{\cdot}01\,(3)$ Å^3, $Z = 2$, $D_x = 1{\cdot}333$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}756$ cm^{-1}, $F(000) = 188$, $T = 291$ K, $R = 0{\cdot}0532$ for 2005 observed reflections. The structure consists of phenazine molecules oriented about a centre of symmetry. The molecule is planar within experimental error, and a pseudo C_{2v} axis is observed in the molecule.

Experimental. Crystals of phenazine were crystallized from acetonitrile. An Enraf–Nonius CAD-4 diffractometer was used with graphite-monochromatized Mo $K\alpha$ radiation. Crystal size was $0{\cdot}25 \times 0{\cdot}30 \times 0{\cdot}35$ mm. Unit-cell parameters were obtained by least-squares fit of the setting angles of 25 reflections in the range $3 \leq 2\theta \leq 18°$. The intensities of 2702 reflections were measured ($\sin\theta/\lambda \leq 0{\cdot}704$ Å^{-1}, $-9 \leq h \leq 9$, $0 \leq k \leq 7$, $0 \leq l \leq 18$, ω–2θ scan mode). No significant variation (< 3%) was found in the intensities of the intensity control reflections $3\bar{1}\bar{1}$ and 103. The data were corrected for Lorentz and polarization effects but no absorption correction was applied. 2005 reflections with $|F| \geq 34\sigma(F)$ were used in the calculations. The structure was solved with multisolution direct methods (*SHELXS86*; Sheldrick, 1990) and refined using full-matrix least-squares refinement (*SHELX76*; Sheldrick, 1976), minimizing $\sum w(|F_o| - |F_c|)^2$, $w = \{2{\cdot}7955/[\sigma^2(F) + 0{\cdot}000553F^2]\}$. The C and N atoms were refined with anisotropic, and H atoms with isotropic temperature factors, 81 parameters were varied. The refinement converged to $R = 0{\cdot}0532$, $wR = 0{\cdot}061$, $(\Delta/\sigma)_{\max} = 0{\cdot}002$, $(\Delta/\sigma)_{\text{mean}} = 0{\cdot}001$, $\Delta\rho_{\max} = 0{\cdot}52$, $\Delta\rho_{\min} = -0{\cdot}35$ e Å^{-3}. The

Table 1. *Fractional coordinates* (× 10^4) *and equivalent isotropic temperature coefficients* (× 10^4) *for non*-H *atoms*

$$U_{\text{eq}} = (1/3)\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}(Å^2)
N1	8035 (1)	550 (1)	9523 (1)	461 (3)
C2	9491 (1)	1851 (2)	9233 (1)	423 (2)
C3	8523 (1)	−1293 (2)	10284 (1)	415 (2)
C4	7050 (1)	−2758 (2)	10629 (1)	522 (3)
C5	9077 (1)	3834 (2)	8429 (1)	551 (3)
C6	7519 (1)	−4624 (2)	11395 (1)	572 (4)
C7	10525 (2)	5155 (2)	8124 (1)	590 (4)

Table 2. *Bond lengths* (Å) *and bond angles* (°)

C2—N1	1·342 (1)	C4—C3	1·426 (1)
C3—N1	1·341 (1)	C6—C4	1·352 (2)
C3—C2^{i}	1·438 (1)	C7—C5	1·351 (2)
C5—C2	1·424 (2)	C7—C6	1·416 (1)
N1—C2—C3^{i}	121·56 (10)	C3^{i}—C2—C5	118·75 (9)
N1—C2—C5	119·70 (7)	C2—C5—C7	120·49 (8)
C2—N1—C3	116·72 (7)	C3—C4—C6	120·44 (7)
N1—C3—C2^{i}	121·73 (9)	C4—C6—C7^{i}	120·94 (9)
N1—C3—C4	119·74 (7)	C5—C7—C6^{i}	120·85 (10)
C2^{i}—C3—C4	118·53 (10)		

Symmetry code: (i) $2 - x$, $-y$, $2 - z$.

0108-2701/91/051113-02$03.00