two. We note that the conformations at the C(1)—C(2), C(1)—C(6) and C(1)—C(12) bonds are similar which is different from the closely related α,α-bis(*p*-chlorophenyl)-3-pyridinemethanol (parinol) (Kennard, Smith & Palm, 1981). For clarity, Newman projections along the three bonds in fenarimol are shown in Fig. 2. While in parinol the two phenyl rings are nearly symmetrically oriented with respect to the heterocyclic ring plane, this is not the case in fenarimol.

Short H-bond interactions (Taylor & Kennard, 1982) are intramolecular Cl(1)···H(O1) [2·37 (8) Å] and intermolecular O(1)···H(C16) [2·48 (8) Å] and N(2)···H(C5) [2·54 (8) Å]; all other packing distances are in the normal range.

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References

BUCHENAUER, H. (1977). *Pestic. Biochem. Physiol.* **7**, 309–320.

CRUICKSHANK, D. W. J. (1970). *Crystallographic Computing*, edited by F. R. AHMED, pp. 187–197. Copenhagen: Munksgaard.

DOMENICANO, A., MURRAY-RUST, P. & VACIAGO, A. (1983). *Acta Cryst.* B**39**, 457–468.

HAMILTON, W. C. (1959). *Acta Cryst.* **18**, 609–610.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

KENNARD, C. H. L., SMITH, G. & PALM, T. B. (1981). *Acta Cryst.* B**37**, 1796–1798.

MAIN, P., LESSINGER, L., WOOLFSON, M. M., GERMAIN, G. & DECLERCQ, J.-P. (1976). *MULTAN76. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

MERCER, E. I. (1984). *Pestic. Sci.* **15**, 133–155.

MOTHERWELL, W. D. S. (1978). *PLUTO78*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

NARDELLI, M. (1983). *Comput. Chem.* **7**, 95–98.

SUTTON, L. E. (1958). Editor. *Tables of Interatomic Distances and Configuration in Molecules and Ions.* Spec. Publ. No. 11. London: The Chemical Society.

TAYLOR, R. & KENNARD, O. (1982). *J. Am. Chem. Soc.* **104**, 5063–5070.

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Structure of Dimethyl 2,3,4,5-Tetrahydro-2-oxo-5α-(3,4,5-trimethoxyphenyl)-3α,4β-furandicarboxylate

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Abstract. $C_{17}H_{20}O_9$, $M_r = 368·3$, triclinic, $P\bar{1}$, $a = 5·687\ (4)$, $b = 12·367\ (3)$, $c = 12·830\ (3)$ Å, $\alpha = 106·24\ (2)$, $\beta = 92·34\ (5)$, $\gamma = 90·39\ (5)°$, $V = 865·5$ Å^3, $Z = 2$, $D_x = 1·41$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0·73$ cm^{-1}, $F(000) = 388$, $T = 293$ K, final $R = 0·091$ for 1482 observed $[F_o \geq 5\sigma(F_o)]$ reflections. The observed structure confirms the stereochemistry as the all-*trans* isomer. There is no crystallographically imposed symmetry. High thermal motion was noted for one of the methoxycarbonyl groups, but a disorder model could not be developed.

Introduction. In the course of our work on the total synthesis of naturally occurring, antimitotic lignan lactones, we have determined the X-ray structure of the title compound. The assignment of substituent stereochemistry on a γ-lactone moiety by ^{1}H NMR coupling-constant analysis is sometimes problematic. Vicinal hydrogen coupling constants for *trans*-substituted compounds have generally been larger than those of the *cis* isomer; however, several exceptions have been noted (Bystrom, Hogberg & Norin, 1981; Fristad & Peterson, 1985). The preparation of the title compound provided two isomers that we felt were simply epimeric at the 3-position due to ^{1}H NMR chemical shifts and coupling constants. As our projected conversion of this mixture to a *cis*-fused dicinnamic acid dilactone necessitated a *trans* disposition of the 4-methoxycarbonyl and 5-aryl groups, we felt compelled to establish firmly this stereorelationship for the major isomer at an early stage.

Experimental. Crystals (m.p. 380–382 K) were obtained by slow evaporation of an ethyl acetate–petroleum ether solution of the title compound. The X-ray structure was in full agreement with IR, ^{1}H and ^{13}C NMR and analytical data. The compound was

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Table 1. *Final fractional coordinates and equivalent isotropic thermal parameters for* $C_{17}H_{20}O_9$

$B_{eq} = \frac{4}{3}(a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + ab\cos\gamma\beta_{12} + ac\cos\beta\beta_{13} + bc\cos\alpha\beta_{23})$.

	x	y	z	B_{eq}(Å^2)
O(1)	0·187 (1)	0·9180 (5)	0·4027 (5)	2·76
O(2)	0·303 (1)	1·0905 (5)	0·4988 (5)	3·19
O(3)	0·803 (1)	1·0386 (5)	0·2771 (6)	3·26
O(4)	0·605 (1)	1·1900 (5)	0·3644 (5)	3·27
O(5)	0·528 (2)	0·7878 (7)	0·0738 (6)	5·13
O(6)	0·258 (2)	0·9167 (8)	0·0831 (6)	5·16
O(7)	−0·106 (1)	0·4598 (5)	0·1430 (5)	2·82
O(8)	0·206 (1)	0·3879 (5)	0·2686 (5)	2·57
O(9)	0·537 (1)	0·5265 (5)	0·3891 (5)	2·75
C(1)	0·293 (2)	1·0191 (8)	0·4147 (8)	2·83
C(2)	0·390 (2)	1·0211 (7)	0·3056 (7)	2·25
C(3)	0·408 (2)	0·8934 (7)	0·2484 (7)	2·28
C(4)	0·186 (2)	0·8502 (7)	0·2889 (7)	2·60
C(5)	0·199 (2)	0·7253 (7)	0·2856 (7)	2·02
C(6)	0·033 (2)	0·6536 (7)	0·2162 (7)	2·03
C(7)	0·047 (2)	0·5380 (7)	0·2104 (7)	2·08
C(8)	0·212 (2)	0·4995 (7)	0·2688 (7)	1·90
C(9)	0·378 (2)	0·5731 (7)	0·3366 (7)	1·93
C(10)	0·371 (2)	0·6878 (7)	0·3432 (7)	2·10
C(11)	0·634 (2)	1·0809 (7)	0·3155 (7)	2·34
C(12)	0·823 (2)	1·2579 (8)	0·380 (1)	4·10
C(13)	0·408 (2)	0·8605 (9)	0·1254 (8)	3·17
C(14)	0·223 (3)	0·883 (1)	−0·0389 (9)	8·02
C(15)	−0·275 (2)	0·4947 (8)	0·0786 (8)	3·04
C(16)	0·340 (2)	0·3156 (8)	0·1860 (9)	3·62
C(17)	0·719 (2)	0·5973 (8)	0·4566 (7)	2·80

Table 2. *Bond distances* (Å) *and angles* (°) *for* $C_{17}H_{20}O_9$

O(1)–C(1)	1·35 (1)	O(1)–C(4)	1·47 (1)
O(2)–C(1)	1·19 (1)	O(3)–C(11)	1·16 (1)
O(4)–C(11)	1·33 (1)	O(4)–C(12)	1·47 (1)
O(5)–C(13)	1·19 (1)	O(6)–C(13)	1·30 (1)
O(6)–C(14)	1·51 (1)	O(7)–C(7)	1·38 (1)
O(7)–C(15)	1·39 (1)	O(8)–C(8)	1·38 (1)
O(8)–C(16)	1·43 (1)	O(9)–C(9)	1·33 (1)
O(9)–C(17)	1·44 (1)	C(1)–C(2)	1·53 (1)
C(2)–C(3)	1·55 (1)	C(2)–C(11)	1·55 (1)
C(3)–C(4)	1·53 (1)	C(3)–C(13)	1·52 (1)
C(4)–C(5)	1·53 (1)	C(5)–C(6)	1·40 (1)
C(5)–C(10)	1·36 (1)	C(6)–C(7)	1·41 (1)
C(7)–C(8)	1·35 (1)	C(8)–C(9)	1·40 (1)
C(9)–C(10)	1·40 (1)		
C(1)–O(1)–C(4)	110·3 (7)	C(11)–O(4)–C(12)	113·6 (8)
C(13)–O(6)–C(14)	118 (1)	C(7)–O(7)–C(15)	119·6 (7)
C(8)–O(8)–C(16)	114·2 (7)	C(9)–O(9)–C(17)	118·9 (7)
O(1)–C(1)–O(2)	122·2 (8)	O(1)–C(1)–C(2)	108·7 (8)
O(2)–C(1)–C(2)	129·1 (8)	C(1)–C(2)–C(3)	101·1 (7)
C(1)–C(2)–C(11)	113·9 (7)	C(3)–C(2)–C(11)	110·9 (8)
C(2)–C(3)–C(4)	99·7 (7)	C(2)–C(3)–C(13)	115·8 (8)
C(4)–C(3)–C(13)	111·6 (8)	O(1)–C(4)–C(3)	102·4 (6)
O(1)–C(4)–C(5)	108·7 (7)	C(3)–C(4)–C(5)	112·7 (8)
C(4)–C(5)–C(6)	115·8 (8)	C(4)–C(5)–C(10)	121·2 (8)
C(6)–C(5)–C(10)	122·9 (8)	C(5)–C(6)–C(7)	116·5 (8)
O(7)–C(7)–C(6)	121·1 (9)	O(7)–C(7)–C(8)	117·4 (8)
C(6)–C(7)–C(8)	121·5 (8)	O(8)–C(8)–C(7)	119·7 (8)
O(8)–C(8)–C(9)	119·3 (8)	C(7)–C(8)–C(9)	120·8 (8)
O(9)–C(9)–C(8)	116·2 (7)	O(9)–C(9)–C(10)	124·7 (8)
C(8)–C(9)–C(10)	119·1 (8)	C(5)–C(10)–C(9)	119·2 (8)
O(3)–C(11)–O(4)	127·4 (9)	O(3)–C(11)–C(2)	124·8 (8)
O(4)–C(11)–C(2)	107·5 (9)	O(5)–C(13)–O(6)	124 (1)
O(5)–C(13)–C(3)	123 (1)	O(6)–C(13)–C(3)	113 (1)

prepared by a procedure described elsewhere (Surjasasmita 1987; Peterson, Do & Surjasasmita, 1988). D_m not determined. Crystal 0·20 × 0·25 × 0·60 mm. Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$. Cell constants from setting angles of 25 reflections ($\theta > 19°$). Correction for Lorentz–polarization effect, no correction for absorption. $\theta_{\max} = 42°$; h 0 to 5, k −12 to 12, l −12 to 12. Standard reflections measured every 3600 s of data collection time: 201, 070, 009. Variation = ±2%. 1836 reflections measured, 1482 independent observed reflections [$F_o \geq 5\sigma(F_o)$]. Structure solved utilizing *MULTAN* (Germain, Main & Woolfson, 1971) direct-methods program. Geometrically constrained H atoms were placed 0·95 Å from the bonded C atom with a fixed isotropic thermal parameter $B = 5{\cdot}5$ Å^2 and allowed to ride on that atom. High thermal motion was noted for C(13), C(14), O(5) and O(6) although it was most pronounced for C(14). A disorder model could not be developed considering the rigidity of this group, and the effect is most probably thermal in nature. The methyl H atoms, except those for C(14), were located from a difference Fourier map and included with fixed contributions ($B = 5{\cdot}5$ Å^2). An unfortunate consequence of the high thermal motion and the inability to locate all H atoms for the title compound comprising only light atoms, is a rather high R value of 0·091. Scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974); structure refined with *SHELX*76 (Sheldrick, 1976). $\sum w(|F_o| - |F_c|)^2$ minimized, weights = unit, 235 parameters varied. $R = 0{\cdot}091$, $wR = 0{\cdot}100$, $S = 1{\cdot}74$. Δ/σ in final least-squares refinement cycle < 0·01, $\Delta\rho < 0{\cdot}4$ e Å^{-3} in final difference map.

Discussion. Fractional coordinates and B_{eq} values are given in Table 1,* distances and angles in Table 2. A

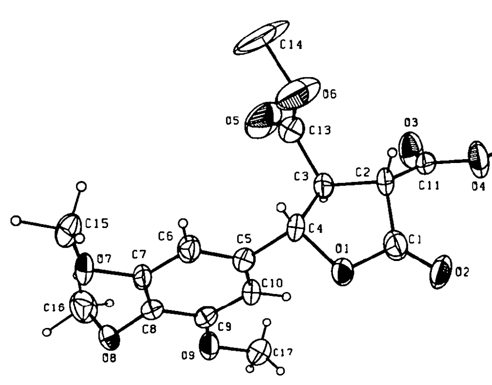

Fig. 1. Thermal-ellipsoid plot of the title compound showing the arbitrary atom-numbering scheme.

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* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, least-squares-planes results and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51094 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

trans stereorelationship between the 4-methoxycarbonyl and 5-aryl groups has been confirmed as seen in the *ORTEP* drawing (Johnson, 1976), Fig. 1.

The lactone ring exists in an envelope conformation with atoms O(1), C(1), C(2) and C(4) planar to within 0·023 Å and C(3) out of this plane by 0·65 Å. The O(1)—C(1)—C(2) bond angle is 108·7 (8)°, deviating somewhat from an unconstrained trigonal carbon center. The bond angles C(2)—C(3)—C(4) and C(1)—C(2)—C(3) are 99·7 (7) and 101·1 (7)°, respectively, and in turn reflect angle compression due to ring size from that usually associated with aliphatic sp^3-hybridized carbon. Bond distances are unaffected by ring geometry in this compound. The bond distance between C(2) and C(3) is 1·55 (1) Å and that between C(3) and C(4) is 1·53 (1) Å. The torsion angles H(1)[C(3)]—C(3)—C(4)—H(1)[C(4)] and H(1)[C(2)]—C(2)—C(3)—H(1)[C(3)] of −154·4 and +151·0°, respectively, support an all-*trans* disposition of substituents at these centers, and are consistent with the relatively large ^{1}H NMR vicinal coupling constants (observed 8·72 and 10·62 Hz, respectively) associated with the major stereoisomer formed by our reaction procedure. The similarly large vicinal hydrogen coupling constant observed for H(1)[C(4)] of the non-crystalline minor isomer is hence consistent with our initial structural assignment that these compounds are simply epimeric at the 3-position (Peterson *et al.*, 1988). Finally, as anticipated, the aromatic ring atoms C(5)—C(10) define a plane to within 0·011 Å.

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References

BYSTROM, S., HOGBERG, H. E. & NORIN, T. (1981). *Tetrahedron* **37**, 2249–2254.

FRISTAD, W. E. & PETERSON, J. R. (1985). *J. Org. Chem.* **50**, 10–18.

GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* A**27**, 368–376.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

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

PETERSON, J. R., DO, H. D. & SURJASASMITA, I. B. (1988). *Synth. Commun.* **18**(16). In the press.

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

SURJASASMITA, I. B. (1987). *Toward the Synthesis of Podophyllotoxin*. MS Thesis, Northern Illinois Univ., DeKalb, Illinois, USA.

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Anticancer-Agent Development: X-ray Structure of Dimethyl 2,3,4,5-Tetrahydro-3-(3,4-methylenedioxybenzoyl)-2-oxo-5β-(3,4,5-trimethoxyphenyl)-3α,4α-furandicarboxylate

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(*Received* 28 *April* 1988; *accepted* 1 *June* 1988)

Abstract. $C_{25}H_{24}O_{12}$, $M_r = 516{\cdot}5$, triclinic, $P\bar{1}$, $a = 7{\cdot}383\,(5)$, $b = 11{\cdot}843\,(7)$, $c = 14{\cdot}775\,(9)$ Å, $\alpha = 104{\cdot}95\,(9)$, $\beta = 100{\cdot}30\,(8)$, $\gamma = 103{\cdot}09\,(7)°$, $V = 1176$ Å^3, $Z = 2$, $D_x = 1{\cdot}46$ g cm^{-3}, $\lambda(\text{Mo }K\alpha) = 0{\cdot}71073$ Å, $\mu = 0{\cdot}74$ cm^{-1}, $F(000) = 540$, $T = 293$ K, final $R = 0{\cdot}047$ for 2733 observed $[F_o \geq 5\sigma(F_o)]$ reflections. The observed structure reveals that the *trans* disposition of the substituents at positions 4 and 5 of the heterocycle is maintained in the reaction sequence, and that the major stereocomponent in the product mixture possesses a *cis*-3,4-bis(methoxycarbonyl) relationship. There is no crystallographically imposed symmetry.

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