The condensed aromatic ring is planar within experimental error, $\sum(\Delta/\sigma)^2 = 7\cdot852$ vs. $7\cdot810$ max. for three degrees of freedom. A description of the orientation of the methoxy groups can be found in the relevant torsion angles: C(5)—C(6)—O(11)—C(12) —6 (1), C(6)—C(7)—O(13)—C(14) 92 (1) and C(7)—C(8)—O(15)—C(16) —67 (1)°. It is noteworthy that the C(6) substituent is nearly coplanar while the C(7) substituent adopts a normal conformation relative to the aromatic ring already mentioned (Bellard, Elliot & McDonald, 1982). On the other hand, the plane of the C(8) methoxy group makes an angle of 113·3 (5)° with the condensed aromatic ring (Miravitlles, Solans, Bladé-Font, Germain & Declercq, 1982).

The heterocyclic fragment of the molecule exhibits a half-chair conformation. The C(1)—C(9)—C(10)—C(4) angle is equal to —4 (1)° and the remaining torsion angles in the heterocyclic ring are consistent with the proposed conformation (Urbaniak, Jaskólski, Rozwadowska & Kosturkiewicz, 1986; Ammon, Prasad, Barnhart, Syal, El-Sayed & Wassel, 1987). The sum of valence angles around the N atom is 338 (10)° showing its considerable pyramidization. The structural data confirm the early configurational assignments (Dominguez, Badia, Castedo & Dominguez, 1988) and show that the solid-state conformation has the C(1) and C(3) substituents in pseudoaxial and equatorial positions respectively, torsion angles C(17)—C(1)—C(9)—C(10) 106 (1), H(1)—C(1)—C(9)—C(10) 139 (1), C(18)—C(3)—C(4)—H(4a) 70 (1), C(18)—C(3)—C(4)—H(4b) —50 (1), H(3)—C(3)—C(4)—H(4a) —46 (1), H(3)—C(3)—C(4)—H(4b) —166 (1)°.

Bond lengths and angles in the heterocyclic ring do not deviate significantly from standard values reported for other compounds containing a similar ring system (Pavkovic, Glowinski, Feng & Brown, 1981, and

references therein; Ammon, Prasad, Barnhart, Syal, El-Sayed & Wassel, 1987).

In the aryl group at C(3), the methoxy groups are approximately coplanar with the benzene nucleus, as can be seen from the torsion angles C(20)—C(21)—O(22)—C(23) 4 (1), C(27)—C(24)—O(25)—C(26) —1 (1)°. This behaviour is typical of *o*-dimethoxybenzene derivatives (Caillet, 1982; Bellard, Elliot & McDonald, 1982; Pavkovic, Glowinski, Feng & Brown, 1981). The aryl group at C(3) is in a perpendicular position with respect to the isoquinoline system.

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Structures of Four *cis* and *trans* Tricyclic Mono- and Dithioacetals

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Abstract. 1,10-*trans*-1,2-*trans*-2,7-*X*-Tricyclo[8.4.0.0^{2,7}]tetradecane: $X = trans$-6-thia-8-oxa, $C_{12}H_{20}OS$ (I), $X = trans$-6-oxa-8-thia, $C_{12}H_{20}OS$ (II), $X = trans$-6,8-dithia, $C_{12}H_{20}S_2$ (III), $X = cis$-6,8-dithia, $C_{12}H_{20}S_2$ (IV). $M_r = 212\cdot4$ (I) (II), $228\cdot4$ (III) (IV). Monoclinic, $P2_1/c$ (I) (II) (III); orthorhombic, $P2_12_12_1$ (IV). $Z = 4$, $a = 5\cdot769$ (3), $b = 12\cdot634$ (4), $c = 15\cdot921$ (12) Å, $\beta = 94\cdot61$ (5)°, $V = 1156\cdot6$ (8) Å^3, final $R = 0\cdot069$ for 1167 observed reflections (I); $a = 12\cdot459$ (5), $b = 10\cdot001$ (2), $c = 9\cdot331$ (3) Å, $\beta =$

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94·96 (2)°, $V = 1158·4$ (3) Å^3, final $R = 0·033$ for 2645 observed reflections (II); $a = 16·784$ (2), $b = 5·521$ (1), $c = 14·868$ (2) Å, $\beta = 116·28$ (1)°, $V = 1235·2$ (5) Å^3, final $R = 0·031$ for 2995 observed reflections (III); $a = 14·261$ (2), $b = 15·302$ (4), $c = 5·394$ (2) Å, $V = 1177·0$ (1) Å^3, final $R = 0·032$ for 836 observed reflections (IV). $D_x = 1·22$ Mg m^{-3}, $\mu = 0·23$ mm^{-1} (I) (II); $D_x = 1·23$ Mg m^{-3}, $\mu = 0·39$ mm^{-1} (III); $D_x = 1·29$ Mg m^{-3}, $\mu = 0·39$ mm^{-1} (IV); $F(000) = 464$ (I) (II), $F(000) = 496$ (III) (IV); λ(Mo $K\bar{\alpha}$) = 0·71069 Å, room temperature. Monothioacetals, compounds (I) and (II), adopt the 'all-*trans*' configuration. Compounds (III) and (IV) are respectively the *trans* and *cis* isomers expected from the dithioacetal equilibration mixture. The structures confirm the description given for the equilibration of rigid *cis* and *trans* tricyclic mono- and dithioacetals. Nevertheless, no evidence of the contribution of the anomeric effect in that process can be given from the examination of C–S bond lengths. In particular, the C–S bond lengths are not significantly different in the *trans* (III) and *cis* (IV) dithioacetals.

Introduction. The synthesis and equilibration of conformationally rigid *cis* and *trans* tricyclic mono- and dithioacetals was recently reported (Deslongchamps & Guay, 1985) and the intervention of the stereoelectronic (anomeric) effect on the equilibrations of thioacetals was invoked to account for the relative populations of *cis* and *trans* isomers. The magnitude of the anomeric effect on changing from O to S has been investigated by *ab initio* calculations (Vishveshwara & Rao, 1982) and a relative decrease was postulated. Nevertheless, it was concluded from the equilibration studies (Deslongchamps & Guay, 1985) that the anomeric effect of the S atom is of the same order of magnitude as that for the O atom. Crystal structure investigations needed to be undertaken to evaluate the modifications of C–S bond lengths resulting from the anomeric effects.

Experimental. Crystals 0·25 × 0·15 × 0·30 mm, Enraf–Nonius CAD-4 (Mo $K\bar{\alpha}$). Lattice parameters determined from 25 reflections with $2\theta \le 26°$. $\omega/2\theta$ scan, $2\theta \le 52°$; one standard reflection monitored every 80 reflections.

(I) $-7 \le h \le 7$, $0 \le k \le 15$, $0 \le l \le 19$, 2252 unique measured reflections, 1085 observed with $I \ge 2·0\sigma(I)$, $R = 0·069$, $wR = 0·071$, max. Δ/σ (for non-H atoms) $= 0·42$, max. and min. electron densities in final difference map 0·31 and $-0·44$ e Å^{-3}.

(II) $-15 \le h \le 15$, $-12 \le k \le 12$, $0 \le l \le 11$, 4313 unique measured reflections, 2645 observed with $I \ge 2·0\sigma(I)$, $R = 0·033$, $wR = 0·030$, max. Δ/σ (for non-H atoms) $= 0·012$, max. and min. electron densities in final difference map 0·25 and $-0·34$ e Å^{-3}.

(III) $-20 \le h \le 20$, $-6 \le k \le 0$, $0 \le l \le 18$, 4842 unique measured reflections, 2995 observed with

$I \ge 2·0\sigma(I)$, $R = 0·031$, $wR = 0·027$, max. Δ/σ (for non-H atoms) $= 0·22$, max. and min. electron densities in final difference map 0·24 and $-0·35$ e Å^{-3}.

(IV) $0 \le h \le 18$, $0 \le k \le 19$, $0 \le l \le 6$, 1509 unique measured reflections, 836 observed with $I \ge 2·0\sigma(I)$, $R = 0·032$, $wR = 0·029$, max. Δ/σ (for non-H atoms) $= 0·29$, max. and min. electron densities in final difference map 0·19 and $-0·17$ e Å^{-3}.

For all the structures, H atoms calculated. All non-H atoms refined with anisotropic temperature factors. $\sum w(\Delta F)^2$ minimized, $w^{-1} = \sigma^2(F_o)$. Intensities corrected

Table 1. *Final coordinates* ($\times 10^4$) *and* B_{eq} *values with e.s.d.'s in parentheses*

	x	y	z	$B_{eq}(\text{Å}^2)$
(I)				
C(1)	−2019 (11)	6863 (5)	1061 (4)	3·64
C(2)	13 (12)	7348 (5)	629 (4)	3·87
C(3)	−153 (13)	7125 (6)	−330 (4)	4·75
C(4)	1932 (14)	7560 (6)	−751 (4)	5·73
C(5)	2300 (14)	8740 (6)	−657 (5)	6·14
S(6)	2785 (4)	9129 (2)	429 (1)	4·94
C(7)	252 (12)	8510 (5)	808 (4)	4·01
O(8)	437 (8)	8748 (4)	1690 (3)	4·76
C(9)	−1576 (13)	8370 (6)	2063 (4)	4·68
C(10)	−1928 (12)	7231 (6)	1985 (4)	4·23
C(11)	−4024 (13)	6831 (7)	2406 (4)	5·42
C(12)	−4414 (14)	5646 (7)	2315 (5)	6·53
C(13)	−4391 (14)	5275 (6)	1417 (5)	6·09
C(14)	−2295 (13)	5701 (6)	992 (4)	5·29
(II)				
C(1)	7871 (1)	8777 (2)	6174 (2)	2·83
C(2)	7556 (1)	10235 (2)	6457 (2)	3·06
C(3)	8126 (1)	11235 (2)	5547 (2)	3·90
C(4)	7726 (2)	12661 (2)	5760 (2)	4·68
C(5)	6516 (2)	12705 (2)	5483 (2)	5·27
O(6)	6039 (1)	11795 (1)	6431 (2)	5·12
C(7)	6346 (1)	10454 (2)	6171 (2)	3·62
S(8)	5566 (0)	9477 (1)	7340 (1)	4·98
C(9)	6049 (1)	7884 (2)	6773 (2)	4·41
C(10)	7271 (1)	7753 (2)	7035 (2)	3·32
C(11)	7608 (1)	6321 (2)	6713 (2)	4·10
C(12)	8818 (2)	6114 (2)	6991 (2)	4·41
C(13)	9428 (1)	7122 (2)	6155 (2)	4·45
C(14)	9091 (1)	8551 (2)	6451 (2)	3·72
(III)				
C(1)	2852 (1)	−1024 (3)	2740 (1)	3·81
C(2)	2129 (1)	952 (3)	2385 (1)	3·95
C(3)	1499 (1)	683 (3)	1265 (1)	4·96
C(4)	815 (1)	2692 (3)	830 (1)	5·75
C(5)	196 (1)	2869 (4)	1323 (1)	6·26
S(6)	795 (0)	3423 (1)	2649 (0)	6·58
C(7)	1577 (1)	935 (3)	2976 (1)	4·44
S(8)	2221 (0)	1294 (1)	4315 (0)	6·52
C(9)	2980 (1)	−1163 (4)	4505 (1)	5·65
C(10)	3478 (1)	−913 (3)	3865 (1)	4·28
C(11)	4200 (1)	−2865 (3)	4178 (1)	5·42
C(12)	4736 (1)	−2698 (4)	3580 (1)	6·37
C(13)	4133 (1)	−2856 (4)	2477 (1)	6·62
C(14)	3408 (1)	−913 (4)	2149 (1)	5·59
(IV)				
C(1)	3702 (4)	2215 (4)	1239 (11)	2·60
C(2)	3737 (4)	2908 (4)	3295 (11)	2·72
C(3)	4597 (5)	3529 (4)	3130 (13)	3·53
C(4)	4619 (4)	4088 (4)	807 (16)	3·61
C(5)	3794 (5)	4725 (4)	695 (15)	3·98
S(6)	2673 (1)	4155 (1)	722 (4)	3·27
C(7)	2845 (4)	3478 (4)	3463 (12)	3·12
S(8)	1802 (1)	2847 (1)	4034 (4)	4·22
C(9)	1927 (4)	2070 (4)	1536 (13)	4·21
C(10)	2862 (4)	1575 (4)	1532 (12)	2·92
C(11)	2856 (4)	901 (4)	−587 (16)	3·79
C(12)	3761 (5)	381 (4)	−616 (16)	4·62
C(13)	4590 (5)	978 (5)	−903 (16)	4·41
C(14)	4618 (4)	1673 (4)	1135 (15)	3·76

Table 2. *Intramolecular bond lengths* (Å) *and angles* (°), *with e.s.d.'s in parentheses*

	(I)	(II)	(III)	(IV)
O(8)–C(7)	1·432 (8)			
S(8)–C(7)		1·810 (2)	1·807 (1)	1·800 (6)
C(7)–O(6)		1·421 (2)		
C(7)–S(6)	1·804 (7)		1·811 (2)	1·822 (7)
C(7)–C(2)	1·499 (9)	1·523 (2)	1·535 (3)	1·546 (8)
S(6)–C(5)	1·798 (8)		1·798 (2)	1·822 (7)
O(6)–C(5)		1·433 (3)		
C(5)–C(4)	1·512 (11)	1·509 (3)	1·517 (3)	1·528 (9)
C(4)–C(3)	1·526 (11)	1·530 (3)	1·519 (2)	1·518 (11)
C(3)–C(2)	1·547 (9)	1·527 (3)	1·534 (2)	1·554 (9)
C(2)–C(1)	1·534 (10)	1·539 (2)	1·541 (2)	1·535 (9)
C(1)–C(14)	1·480 (10)	1·536 (2)	1·519 (2)	1·548 (9)
C(1)–C(10)	1·539 (9)	1·535 (2)	1·534 (2)	1·555 (9)
C(14)–C(13)	1·530 (11)	1·522 (3)	1·531 (3)	1·530 (10)
C(13)–C(12)	1·505 (11)	1·518 (3)	1·540 (3)	1·503 (9)
C(12)–C(11)	1·519 (12)	1·522 (3)	1·523 (3)	1·516 (9)
C(11)–C(10)	1·515 (10)	1·530 (3)	1·533 (2)	1·540 (10)
C(10)–C(9)	1·457 (10)	1·527 (2)	1·525 (3)	1·534 (9)
C(9)–O(8)	1·428 (9)			
C(9)–S(8)		1·799 (2)	1·796 (2)	1·806 (7)
O(8)–C(7)–C(2)	113·0 (5)			
S(8)–C(7)–C(2)		113·0 (1)	114·3 (1)	112·8 (4)
O(8)–C(7)–S(6)	103·7 (4)			
S(8)–C(7)–O(6)		103·9 (1)		
S(8)–C(7)–S(6)			102·8 (1)	109·4 (3)
C(2)–C(7)–S(6)	115·1 (5)		113·0 (1)	112·6 (4)
C(2)–C(7)–O(6)		112·5 (1)		
C(7)–S(6)–C(5)	97·7 (3)		98·8 (1)	99·2 (3)
C(7)–O(6)–C(5)		111·2 (1)		
S(6)–C(5)–C(4)	112·0 (5)		111·8 (1)	111·8 (4)
O(6)–C(5)–C(4)		109·7 (1)		
C(5)–C(4)–C(3)	114·9 (6)	109·6 (1)	113·0 (2)	112·1 (6)
C(4)–C(3)–C(2)	112·4 (6)	111·5 (1)	114·9 (1)	114·1 (5)
C(3)–C(2)–C(1)	112·5 (5)	112·8 (1)	111·3 (1)	114·0 (5)
C(7)–C(2)–C(1)	111·5 (6)	111·7 (1)	112·5 (1)	113·9 (5)
C(2)–C(1)–C(14)	116·4 (6)	111·9 (1)	111·1 (1)	111·6 (5)
C(2)–C(1)–C(10)	110·0 (5)	113·5 (1)	113·6 (1)	112·7 (5)
C(14)–C(1)–C(10)	111·4 (6)	109·4 (1)	109·0 (1)	108·5 (5)
C(1)–C(14)–C(13)	113·6 (6)	113·1 (1)	112·8 (1)	112·1 (6)
C(14)–C(13)–C(12)	112·2 (6)	111·9 (2)	111·3 (2)	111·7 (6)
C(13)–C(12)–C(11)	112·6 (6)	110·7 (1)	110·4 (1)	110·6 (5)
C(12)–C(11)–C(10)	113·7 (6)	112·2 (1)	111·9 (2)	110·8 (6)
C(11)–C(10)–C(9)	113·6 (6)	109·6 (1)	108·9 (1)	109·1 (5)
C(11)–C(10)–C(1)	110·9 (6)	111·5 (1)	111·8 (1)	110·5 (5)
C(1)–C(10)–C(9)	111·9 (6)	112·5 (1)	112·2 (1)	111·1 (5)
C(10)–C(9)–O(8)	114·1 (6)			
C(10)–C(9)–S(8)		112·5 (1)	112·3 (1)	114·3 (4)
C(9)–O(8)–C(7)	109·7 (5)			
C(9)–S(8)–C(7)		95·2 (1)	97·3 (1)	98·3 (3)

Table 3. *Selected torsion angles* (°) *including the two heteroatoms with e.s.d.'s in parentheses*

|	(I)	(II)	(III)	(IV)
O(8)–C(7)–S(6)–C(5)	-0·8 (6)			
S(8)–C(7)–O(6)–C(5)		5·3 (1)		
S(8)–C(7)–S(6)–C(5)			0·4 (2)	-1·9 (3)
C(9)–O(8)–C(7)–S(6)	-4·5 (4)			
C(9)–S(8)–C(7)–O(6)		-2·0 (1)		
C(9)–S(8)–C(7)–S(6)			-2·6 (4)	73·5 (4)
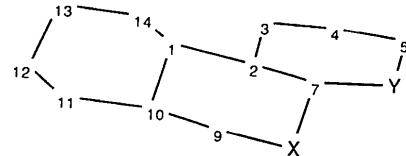
Fig. 1. Numbering scheme: (I) $X = O$, $Y = S$; (II) $X = S$, $Y = O$; (III) $X = S$, $Y = S$; (IV) $X = S$, $Y = S$.
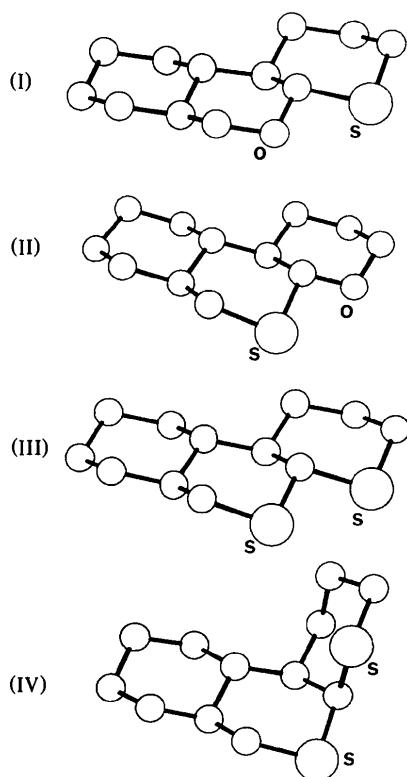
Fig. 2. Perspective views of the title compounds using *PLUTO* (Motherwell & Clegg, 1978).

for Lp, secondary extinction and absorption corrections not used, atomic scattering factors from *XTAL*83. Structures solved by direct methods. Computer programs used for all structures: *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), *XTAL*83 (Stewart & Hall, 1985) and *PLUTO* (Motherwell & Clegg, 1978).

Discussion. Final atomic parameters are given in Table 1.* Bond lengths and angles are given in Table 2 and selected torsion angles in Table 3. The numbering scheme used is given in Fig. 1. Perspective views of the four title molecules are depicted in Fig. 2.

'All-chair' conformations are observed for all the compounds. Minor distortions are observed on torsion-

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and a complete list of torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51092 (133 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

angle values. The main differences must be attributed to the constraints induced by the heteroatoms.

The C—S bond lengths observed in (I), (II) and (III) are comparable within e.s.d.'s. This observation allows assignment of a value of 1·810 Å for C—S bonds unaffected by anomeric effects. Recent studies have presented crystallographic evidence for anomeric inter-

actions between O and N lone pairs and C—S bond lengths (Orrell & Wallis, 1984). In that study, a lengthening of the C—S bond to 1·880 (2) Å was attributed to the anomeric interaction between the N lone pair and the σ^* of the C—S. Nevertheless it was mentioned that this effect was combined with a conjugation of the S with the acrylic ester function that increased the polarization of the C—S bond.

A lengthening of the C—S bond up to 1·830 (5) Å was obtained for compounds in which an O lone pair was approximately antiperiplanar to the C—S bond (Soboleva, D'Yachenko, Atovmyan, Kharchenko & Klimenko, 1978). A corresponding shortening of the C—O bond to 1·425 (5) Å was also observed.

The particular *trans–cis* configuration of (IV) places a C—S bond in an axial orientation and susceptible to the anomeric effect. The torsion angle S(6)—C(7)—S(8)—C(9) of 73·4 (4)° observed in (IV) is such that the S lone pair of S(8) is antiperiplanar to the S(6)—C(7) bond. A resulting shortening of the S(8)—C(7) bond would be expected as well as an elongation of the S(6)—C(7) bond length. Observed values: 1·800 (6) and 1·822 (7) respectively are not significantly different from the 1·810 Å obtained for a

standard C—S bond length; the differences are of the order of the standard deviations. No significant intermolecular interactions have been observed: packing is due to van der Waals contacts.

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Structure of Isomammeigin – a New Phenylcoumarin from Guttiferae Species

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Abstract. $C_{25}H_{24}O_5$, $M_r = 404·47$, triclinic, $P\bar{1}$, $a = 9·487$ (2), $b = 9·617$ (5), $c = 12·397$ (3) Å, $\alpha = 77·28$ (3), $\beta = 70·04$ (2), $\gamma = 82·43$ (2)°, $V = 1035·1$ (7) Å^3, $Z = 2$, $D_x = 1·30$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ(Mo $K\alpha$) = 0·08 mm^{-1}, $F(000) = 428$, $T = 296$ K, $R = 0·060$ for 1329 reflections. A new phenylcoumarin isolated from *Kilmeyera pumila* Pohl is shown to be 5-hydroxy-6-isovaleryl-2,2-dimethyl-10-phenyl-2*H*,8*H*-benzo[1,2-*b*:3,4-*b'*]dipyran-8-one. There is an intramolecular O(2)···O(5) hydrogen bond of 2·445 (5) Å.

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Introduction. Chemical investigation of the fruit of *Kilmeyera pumila* Pohl (family Guttiferae) collected in Minas Gerais, Brazil, led us to the isolation of two phenylcoumarins (I) and (II). Compound (I), named mammeigin, had already been isolated from *Mammea americana* L. (Finnegan & Mueller, 1964) and its structure established on spectroscopic evidence and on chemical correlation (Finnegan & Mueller, 1965). It was pointed out in that work that, from spectroscopic data alone, structures (I) and (II) were possible alternatives for mammeigin. From IR and NMR data (de Abreu e Silva, 1987) structure (II) was proposed for the new compound, named isomammeigin. To verify this hypothesis and to establish its molecular con-