In summary, our structure results show that the metal ions are completely disordered in Al_2TiO_5. The differences between our room temperature and 600°C structures explain the anisotropic thermal expansion behavior of the single crystal. Furthermore, the importance of cleavage planes resulting from edge-shared octahedra in the hysteresis of the thermal expansion of the ceramic is pointed out.

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The Molecular Structure of 3′,5,5′,6-Tetramethoxyflavone, $C_{19}H_{18}O_6$*

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Sayre's relations were used to determine the structure of 3′,5,5′,6-tetramethoxyflavone. The flavone was extracted from the fruit of *Sargentia greggii*, a citrus plant found in Mexico. The space group is $P2_1/c$ and the cell dimensions are $a=7{\cdot}335\,(5)$, $b=11{\cdot}304\,(5)$, $c=20{\cdot}249\,(6)$ Å and $\beta=104{\cdot}5\,(4)°$. 2511 independent reflections were collected by counter methods. The observed density is 1·42 g.cm^{-3} which is consistent with four molecules per unit cell, $d_c=1{\cdot}40$ g.cm^{-3}. The model was refined by least-squares techniques to a conventional R value of 0·086 for 1249 of the largest intensities. The heterocyclic ring, which is fused to the benzene ring to form the γ-benzopyrone portion of the molecule, is slightly puckered and planes fitted to the two rings make an angle of 4°. The phenyl ring is planar and makes an angle of 28° with the γ-benzopyrone portion of the molecule. Three of the methoxyl groups are essentially coplanar with the benzene rings while the fourth is forced from the plane due to intramolecular crowding.

Introduction

A white crystalline compound was extracted from the fruit of *Sargentia greggii*. The citrus plant is known locally in Mexico as limoncello, chapote amarillo or naranjillo. The compound was identified as a flavone and was characterized by nuclear magnetic resonance and mass spectrometry. The highest mass peak (342) along with elemental analyses established the formula as $C_{19}H_{18}O_6$. Zeisel tests indicated the molecule contained four methoxyl groups. The positions of the three phenyl ring hydrogen atoms were established from the n.m.r., and this established the positions of two of the methoxyl groups. The assignment of the hydrogen atoms associated with γ-benzopyrone portion of the molecule was not unequivocal; however, the molecule tentatively was assumed to be 3′,5,5′,8-tetramethoxyisoflavone. A few crystals of the flavone were submitted to the FASTBIOS Laboratory by Dr Xorge Domínguez.

Experimental

A large crystal was ground into an ellipsoid with axes of 0·46 mm, 0·48 mm and 1·0 mm. The crystal was mounted with the a axis (major axis of the ellipsoid) coincident with the rotation axis. The unit cell was found to be monoclinic and the room temperature cell

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* Contribution No. 2 from FASTBIOS Laboratory, Texas Christian University.

Table 1. *Squares of the observed and calculated structure factors for* 3′,5,5′,6-*tetramethoxyflavone*

[illegible]

dimensions were determined from a-axis oscillation and $0kl$ Weissenberg photographs. Sodium chloride lines, $a = 5{\cdot}6402$ Å, were superimposed on the photographs as an internal standard. Errors in cell dimensions are standard deviations obtained from a calculation of cell edges for a number of reflections. The density was measured by the flotation technique using a mixture of benzene and carbon tetrachloride.

$C_{19}H_{18}O_6$, molecular weight 342·35. $a = 7{\cdot}335$ (5), $b = 11{\cdot}304$ (5), $c = 20{\cdot}249$ (6) Å; $\beta = 104{\cdot}5$ (4)°. Systematic absences: $h0l$, $h = 2n+1$; $0k0$, $k = 2n+1$. Space group: $P2_1/c$ (C^5_{2h}, No. 14). $Z = 4$; $F(000) = 720$; $V = 1625$ Å^3; $\mu = 8{\cdot}84$ cm^{-1} (Cu $K\alpha$); D_{exp}(flotation) = 1·42 g.cm^{-3}; $D_{\text{calc}} = 1{\cdot}40$ g.cm^{-3}; Cu $K\alpha = 1{\cdot}54178$ Å; graphite monochromator.

Intensity data were collected on a Philips PAILRED diffractometer for layers $0kl$ to $6kl$. A scan rate of 2·5° per minute over a range of 3·4 to 4·0° was used. The larger scan range was used for lower angles and upper levels. A background count of ten seconds was taken on either side of the ω-scan range. The intensities of several reference reflections were monitored during data collection. At the termination of data collection, the crystal had become pale yellow; however, variations in the reference reflection intensities were less than 5%. No corrections were made for these changes. A total of 2511 independent reflections were measured and 2109 were considered to have an observed intensity ($\sigma_F < \frac{1}{2}F_o$). The data were corrected for Lorentz and polarization factors. No absorption corrections were made. The data were adjusted to an absolute scale by a Wilson plot. Standard deviations in observed structure amplitudes, $\sigma(F_o)$ were assigned according to the equation

$$\sigma(F_o) = \tfrac{1}{2}\sqrt{\frac{S + t^2B + [0{\cdot}03(S - tB)]^2}{Lp(S - tB)}}$$

where S is the integrated scan count, B is the sum of background counts, t is the ratio of time spent on scan to time spent on background and 0·03 is an empirical error parameter. In addition to the structure factor magnitudes, $|F_o|$, the normalized structure factor

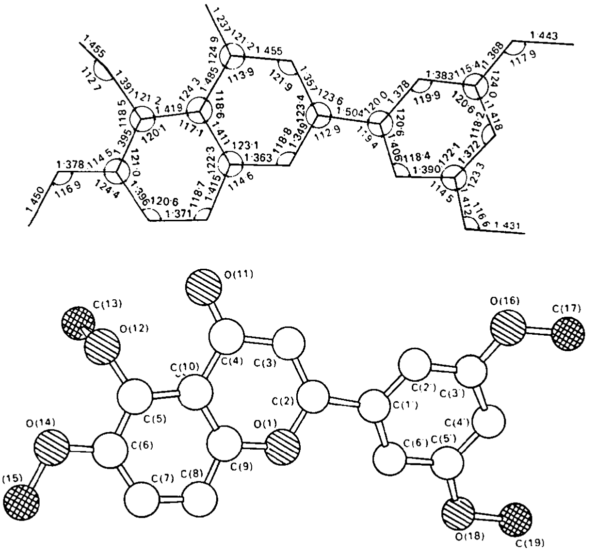

Fig. 1. Molecular structure of 3′,5,5′,6-tetramethoxyflavone

Table 2. *Atomic* (× 10^4) *and thermal parameters for* 3′,5,5′,6-*tetramethoxyflavone*

Thermal parameters are of the form $\exp\,[-0{\cdot}25(B_{11}h^2a^{*2} + B_{22}k^2b^{*2} + B_{33}l^2c^{*2} + 2B_{12}hka^*b^* + 2B_{13}hla^*c^* + 2B_{23}klb^*c^*)]$.

(*a*) Heavy atoms

	X	Y	Z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
O(1)	7242 (6)	4267 (4)	957 (2)	2·4 (2)	2·4 (2)	4·6 (3)	−0·5 (3)	1·5 (4)	0·8 (4)
C(2)	8579 (10)	3436 (6)	1170 (3)	2·7 (4)	3·3 (3)	2·9 (3)	0·3 (5)	1·2 (5)	−0·8 (5)
C(3)	10426 (9)	3610 (6)	1196 (4)	2·7 (4)	3·3 (3)	3·6 (3)	0·3 (5)	0·9 (5)	−0·1 (5)
C(4)	11127 (10)	4741 (6)	1021 (3)	2·3 (3)	3·2 (3)	3·2 (3)	−1·1 (5)	1·4 (5)	−1·7 (5)
C(5)	9947 (9)	6757 (6)	500 (4)	2·2 (3)	3·2 (3)	3·1 (3)	−0·4 (5)	1·2 (5)	−0·1 (5)
C(6)	8432 (10)	7503 (6)	225 (4)	3·1 (3)	2·8 (3)	3·3 (3)	−0·6 (5)	1·3 (5)	0·2 (5)
C(7)	6594 (9)	7182 (6)	225 (4)	3·1 (3)	2·8 (3)	3·6 (3)	1·1 (5)	1·4 (5)	0·8 (5)
C(8)	6227 (9)	6106 (6)	476 (4)	2·2 (3)	3·8 (3)	4·0 (3)	−1·4 (5)	1·4 (5)	−0·6 (6)
C(9)	7747 (8)	5328 (6)	737 (4)	1·9 (3)	3·0 (3)	3·8 (3)	−0·6 (4)	1·4 (5)	−1·0 (5)
C(10)	9626 (9)	5624 (6)	751 (3)	2·5 (3)	3·1 (3)	2·4 (3)	0·7 (4)	0·8 (5)	−0·5 (5)
O(11)	12832 (7)	4917 (4)	1100 (3)	2·3 (3)	3·8 (2)	6·0 (3)	−0·5 (4)	1·8 (4)	1·1 (5)
O(12)	11750 (6)	7125 (4)	492 (3)	2·8 (2)	3·3 (2)	4·2 (3)	−1·2 (4)	2·8 (4)	0·2 (4)
C(13)	12761 (10)	7655 (7)	1134 (5)	2·9 (4)	4·6 (4)	6·0 (5)	−2·0 (6)	2·0 (6)	−2·4 (7)
O(14)	8931 (7)	8580 (4)	4 (3)	3·6 (2)	3·4 (2)	5·8 (3)	−0·7 (4)	3·3 (4)	3·0 (4)
C(15)	7466 (11)	9471 (7)	−170 (5)	4·3 (4)	2·9 (3)	6·8 (5)	1·3 (6)	0·4 (7)	1·1 (7)
O(16)	9203 (8)	−720 (5)	1711 (3)	5·2 (3)	2·7 (2)	7·8 (4)	0·9 (4)	7·3 (5)	0·7 (5)
C(17)	8562 (11)	−1802 (6)	1958 (4)	5·3 (4)	2·2 (3	5·0 (4)	0·1 (6)	3·7 (7)	0·6 (6)
O(18)	3983 (7)	1549 (5)	2191 (3)	4·1 (3)	3·5 (2)	6·7 (3)	0·3 (4)	6·3 (5)	2·8 (5)
C(19)	3419 (10)	567 (7)	2540 (4)	3·2 (4)	4·4 (4)	5·4 (4)	−2·0 (6)	3·7 (6)	2·2 (6)
C(1′)	7833 (9)	2333 (6)	1426 (4)	2·8 (3)	3·1 (2)	3·5 (3)	−1·0 (5)	1·8 (5)	−1·2 (5)
C(2′)	8786 (9)	1279 (6)	1444 (4)	2·9 (3)	2·9 (2)	4·1 (3)	−0·4 (5)	2·6 (5)	−0·7 (5)
C(3′)	8164 (10)	283 (6)	1718 (4)	3·2 (4)	2·9 (3)	4·6 (4)	0·7 (5)	2·2 (6)	0·5 (6)
C(4′)	6556 (10)	337 (6)	1986 (4)	3·3 (4)	2·7 (3)	4·2 (3)	−0·9 (5)	2·2 (6)	0·5 (6)
C(5′)	5624 (9)	1396 (6)	1956 (4)	2·3 (3)	3·2 (2)	4·2 (3)	0·8 (5)	3·2 (5)	0·1 (6)
C(6′)	6192 (9)	2399 (6)	1664 (4)	2·8 (3)	3·1 (2)	4·2 (3)	0·3 (5)	3·6 (5)	−0·1 (5)

Table 2 (*cont.*)

(*b*) Hydrogen atoms

	X	*Y*	*Z*	*B*
H(3)	11330	2941	1339	2·0
H(7)	5556	7731	0043	2·0
H(8)	4974	5886	0475	2·0
H(2')	9890	1235	1264	2·0
H(4')	6137	−339	2181	2·0
H(6')	5490	3111	1626	2·0

magnitudes, $|E_h|$, were computed. The scattering factors of Cromer & Waber (1965) were used for the carbon and oxygen atoms while those of Stewart, Davidson & Simpson (1965) were used for the hydrogen atoms.

Structure determination and refinement

A program package, developed by Stemple (1970) for the IBM 1800 computer, was used in the structure analysis. The package contains a modified version of the program by Long (1965) which utilizes the Sayre's (1952) sign relations. Three appropriate origin-determining reflections were given positive signs and three additional reflections were chosen in the starting set. A total of 125 of the largest *E*'s were used in the calculation and all program modes were tried, Long (1965). No reasonable molecular structure or molecular fragment could be found in the *E* maps. The set of *E*'s was increased to 150 and the calculation was repeated. The coordinates of 25 atoms were obtained from the *E* map with the highest reliability index. A structure factor calculation for 425 reflections yielded an *R* value of 0·32 where $R=\sum||F_o|-|F_c||/\sum|F_o|$. Three cycles of isotropic least-squares refinement reduced *R* to 0·20 and indicated one methyl carbon was incorrectly placed. The correct coordinates of the methyl carbon were taken from an electron density map calculated with the signs generated during the last least-squares calculation. The function minimized in the refinement is $\sum w(kF_o-F_c)^2$ where $w=[1/\sigma(F_o)]^2$. The X-ray analysis showed the phenyl and one methoxyl group had been incorrectly assigned in the structure postulated. The compound is 3',5,5',6-tetramethoxyflavone rather than an isoflavone.

Two cycles of isotropic least-squares refinement were followed by four cycles of anisotropic block-diagonal refinement and the *R* value decreased to 0·11 for 847 reflections. An error analysis program indicated that secondary extinction was important. The data were corrected by applying the equation $F^2_{\text{corr}}=F^2_{\text{obs}}(1+2gI_{\text{calc}})$ where $2g$ was found to be $3{\cdot}85\times10^{-5}$. Four additional cycles of anisotropic refinement with 957 reflections reduced *R* to 0·088. The coordinates of the six ring hydrogen atoms were calculated. The hydrogen atom contributions were calculated in subsequent least-squares refinements, but the parameters were not refined. New hydrogen atomic parameters

Table 3. *Interatomic distances and bond angles for* 3',5,5',6-*tetramethoxyflavone*

O(1)-C(2)	1·349 (7) Å	C(9)—C(10)	1·411 (7) Å
O(1)-C(9)	1·363 (8)	O(12)-C(13)	1·455 (10)
C(2)-C(3)	1·357 (7)	O(14)-C(15)	1·450 (8)
C(2)-C(1')	1·504 (9)	O(16)-C(17)	1·443 (9)
C(3)-C(4)	1·455 (10)	O(18)-C(19)	1·431 (9)
C(4)-C(10)	1·485 (9)	C(1')-C(2')	1·378 (9)
C(4)-O(11)	1·237 (6)	C(1')-C(6')	1·406 (8)
C(5)-C(6)	1·395 (9)	C(2')-C(3')	1·383 (9)
C(5)-C(10)	1·419 (9)	C(3')-C(4')	1·418 (8)
C(5)-O(12)	1·391 (6)	C(3')-O(16)	1·368 (8)
C(6)-C(7)	1·396 (7)	C(4')-C(5')	1·372 (9)
C(6)-O(14)	1·378 (8)	C(5')-O(18)	1·412 (7)
C(7)-C(8)	1·371 (10)	C(5')-C(6')	1·390 (9)
C(8)-C(9)	1·415 (8)		
C(2)—O(1)-C(9)	118·8 (4)°	C(4)-C(10)-C(5)	124·3 (5)
O(1)—C(2)-C(3)	123·4 (6)	C(4)-C(10)-C(9)	118·6 (6)
O(1)—C(2)-C(1')	112·9 (4)	C(5)-C(10)-C(9)	117·1 (5)
C(3)—C(2)-C(1')	123·6 (6)	C(5)-O(12)-C(13)	112·7 (5)
C(2)—C(3)-C(4)	121·9 (6)	C(6)-O(14)-C(15)	116·9 (5)
C(3)—C(4)-C(10)	113·9 (5)	C(3')-O(16)-C(17)	117·9 (5)
C(3)—C(4)-O(11)	121·2 (6)	C(5')-O(18)-C(19)	116·6 (5)
C(10)-C(4)-O(11)	124·9 (6)	C(2)-C(1')-C(2')	120·0 (5)
C(6)—C(5)-C(10)	120·1 (5)	C(2)-C(1')-C(6')	119·4 (6)
C(6)—C(5)-O(12)	118·6 (6)	C(2')-C(1')-C(6')	120·6 (6)
C(10)-C(5)-O(12)	121·2 (5)	C(1')-C(2')-C(3')	119·9 (5)
C(5)—C(6)-C(7)	121·0 (6)	C(2')-C(3')-C(4')	120·6 (6)
C(5)—C(6)-O(14)	114·5 (5)	C(2')-C(3')-O(16)	115·4 (5)
C(7)—C(6)-O(14)	124·4 (5)	C(4')-C(3')-O(16)	124·0 (6)
C(6)—C(7)-C(8)	120·6 (6)	C(3')-C(4')-C(5')	118·2 (6)
C(7)—C(8)-C(9)	118·7 (5)	C(4')-C(5')-O(18)	123·3 (6)
O(1)—C(9)-C(8)	114·6 (4)	C(4')-C(5')-C(6')	122·1 (5)
O(1)—C(9)-C(10)	123·1 (5)	C(6')-C5(')-O(18)	114·5 (5)
C(8)—C(9)-C(10)	122·3 (6)	C(1')-C6(')-C(5')	118·4 (6)

were calculated after every two cycles of refinement, and the temperature factors were fixed at 2·0. After five cycles of refinement, the R value for 1249 reflections was 0·086. At this stage all bond lengths and bond angles appeared to be reasonable and the refinement was terminated. Because of limited computer time and funds only 1249 of the strongest reflections were included in the refinement. This corresponded to approximately 6 reflections per parameter refined. The squares of the 2511 observed and calculated structure factors are included in Table 1. Those reflections assigned a zero intensity have a calculated intensity below the observed minimum.

Standard deviations obtained from the inverses of the normal equations matrices from the block-diagonal refinement, were multiplied by 1·13. This factor was derived by comparing the deviations calculated for one cycle of full-matrix refinement with those obtained from one cycle of block diagonal refinement during an earlier stage of analysis. The atomic and thermal parameters along with their estimated standard deviations are given in Table 2. The shifts of all parameters during the last cycle of refinement were less than the estimated standard deviations of the parameters. A final difference Fourier synthesis contained no peak larger than 0·6 e.Å^{-3}.

Discussion

The structure of 3′,5,5′,6-tetramethoxyflavone is shown in Fig. 1. The numbering system used in the Tables and the bond lengths and bond angles are included in the Figure. Fig. 2 shows a projection of the unit-cell contents on the *bc* plane. The γ-benzopyrone portion of the molecule is almost perpendicular to the *ac* plane. Bond lengths, angles and some intermolecular distances are included in Table 3.

A least-squares plane fitted to the eleven atoms of the γ-benzopyrone portion of the molecule and the three atoms attached to it has a standard deviation of

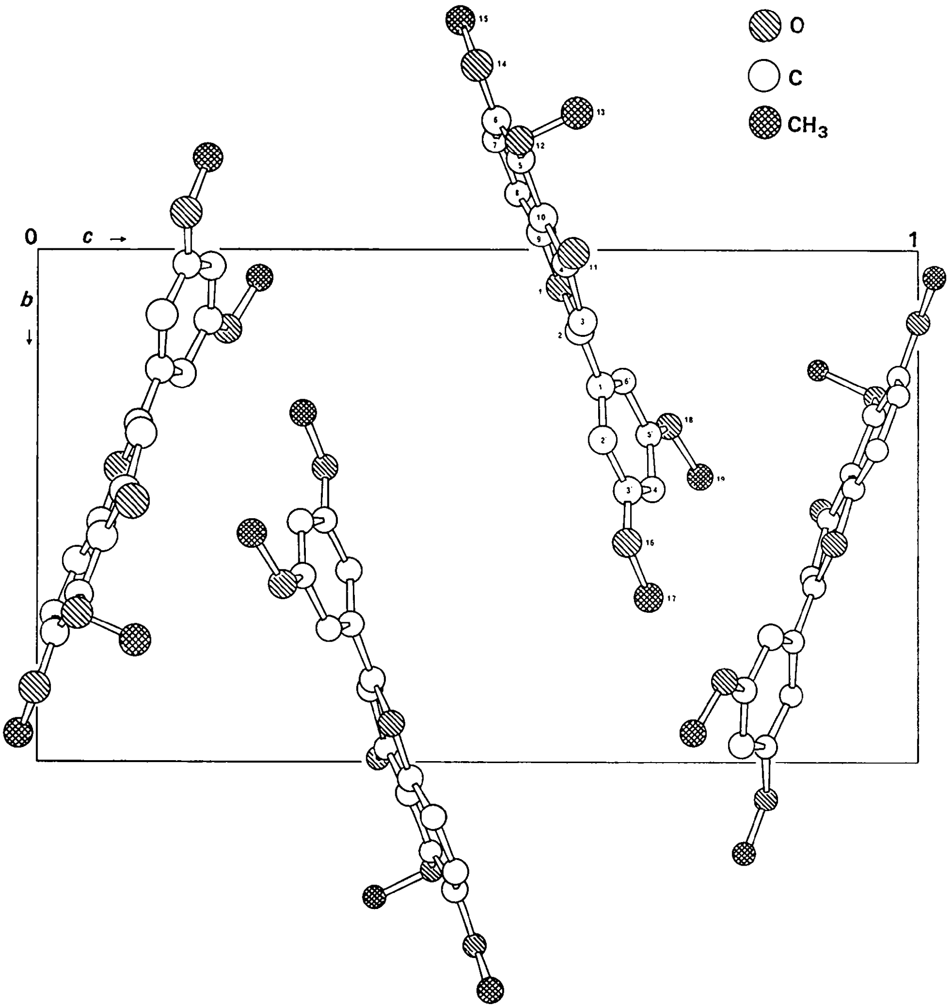

Fig. 2. Projection of the unit-cell contents onto the *bc* plane.

0·05 Å. The heterocyclic ring, fused to the benzene ring, is slightly puckered and atom C(3) is 0·1 Å out of the plane. A least-squares plane fitted to the six benzene carbon atoms and the four attached atoms has a standard deviation of 0·016 Å while the plane fitted only to the six ring carbon atoms has a deviation of 0·014 Å. All atoms are within three standard deviations of either plane. Methyl carbon C(15) lies 0·20 Å out of the least-squares plane while methyl carbon C(13) lies 1·34 Å from the plane. The plane defined by C(13)–O(12)–C(5) makes an angle of 85° with the benzene ring while the C(15)–O(14)–C(6) plane makes an angle of 9°. Steric interactions prevent methyl C(13) from lying in the plane of the benzene ring. The bond lengths and angles in the benzene ring show some variations due to fusion to a heterocyclic ring and unlike substituents at the other positions. The average bond length within the ring is 1·401 (16) Å and the average angle is 120 (2)°. A least-squares plane fitted to the heterocyclic ring has a standard deviation of 0·03 Å and the plane makes an angle of 4° with the plane of the benzene ring. The C(2)–C(3) double bond of 1·357 (7) Å, the two C–C single bonds of 1·455 (10) Å and 1·485 (9) Å and the two C–O bonds lengths of 1·349 (7) and 1·363 (8) Å can be rationalized by considering neighboring moieties.

A least-squares plane fitted to the phenyl ring and the three atoms attached to it has a standard deviation of 0·03 Å while a plane fitted to the six-membered ring has a deviation of 0·02 Å. In the latter plane all atoms lie within 3 standard deviations of the plane; however, in the former plane atom C(2) lies 0·045 Å out of the plane. The C(2)–C(1′) bond makes an angle of 4° with respect to the plane of the phenyl ring. The phenyl ring makes an angle of 28° with the plane of the γ-benzopyrone system. The two methoxy oxygen atoms lie in the plane of the phenyl ring while methyl carbon C(17) and C(19) lie 0·11 and 0·12 Å out of the plane. The plane defined by C(3′)–O(16)–C(17) makes an angle of 2° with the phenyl ring and plane C(5′)–O(18)–C(19) makes an angle of 7°. The C–C bond lengths within the phenyl ring average 1·391 ± 0·02 Å while the bond angles average 120 (1)°. The C–C single bond joining the two ring systems is 1·504 (9) Å.

The methoxyl groups achieve a coplanar orientation with the benzene rings unless inhibited by steric interactions. Three methoxyl groups make angles of 2, 7 and 8° with the rings while the fourth makes an angle of 84°. The C–O–C(M) angles for the first three coplanar methoxyl groups are 117·9, 116·6 and 116·9° while the other is 112·7°. This trend has been observed previously in several aromatic compounds (Yonemitsu, Nakai, Kanaoka, Karle & Witkop, 1969; Karle & Karle, 1968; Corbin, Earle & Karle, 1970; Gilardi & Karle, 1971). The average carbon–methoxyl oxygen distance is 1·38 (2) Å and the average methyl carbon–oxygen distance is 1·44 (1) Å.

The compound 3′,5,5′,6-tetramethoxyflavone also has been isolated from *Casimiroa edulis* Llave *et* Lex., (Dreyer, 1968). The structure was established by synthesis.

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