

temperature factor ( $U = 0.088 \text{ \AA}^2$ ).  $w = 1/(\sigma^2 + 0.00012F^2)$ ,  $R = 0.042$ ,  $wR = 0.038$ ,  $S = 2.15$  for 602 observed reflections. Final maximum shift to e.s.d. 0.07. Maximum and minimum heights in final difference Fourier synthesis 0.32 and  $-0.34 \text{ e \AA}^{-3}$ . Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV).

The atomic coordinates and temperature factors are given in Table 1. Fig. 1 is a stereoscopic view of the molecule, showing the numbering of the atoms (*PLUTO*; Motherwell & Clegg, 1978). Bond distances and angles are given in Table 2.\*

**Related literature.** The title molecule has the same skeleton as thiouracil. Thiouracil derivatives with known structures are: 2-thiouracil (Tiekink, 1989),

\* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55229 (6 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA0110]

4-amino-2-thioxo-6-pyrimidinone monohydrate (Raper, Oughtred & Nowell, 1985), 4-benzyl-2-thiouracil (Delage, H'Naifi & Goursolle, 1986), 6-methyl-2-thiouracil (Delage, H'Naifi, Goursolle & Carpy, 1986) and 6-ethylthio-1,3-diphenyl-2-thiouracil (Schaumann, Bauch, Sieveking & Adiwidjaja, 1982).

#### References

- DELAGE, C., H'NAIFI, A. & GOURSOLLE, M. (1986). *C. R. Acad. Sci.* **302**, 1645–1649.  
 DELAGE, C., H'NAIFI, A., GOURSOLLE, M. & CARPY, A. (1986). *C. R. Acad. Sci.* **302**, 219–224.  
 EVARISTE, F. (1991). PhD thesis, Univ. of Louvain, Belgium.  
 MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.  
 RAPER, E. S., OUGHTRED, R. E. & NOWELL, I. W. (1985). *Acta Cryst.* **C41**, 758–760.  
 SCHAUMANN, E., BAUCH, H. G., SIEVEKING, S. & ADIWIDJAJA, G. (1982). *Chem. Ber.* **115**, 3340–3356.  
 SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.  
 SHELDRIK, G. M. (1985). In *Crystallographic Computing 3*, edited by G. M. SHELDRIK, C. KRÜGER & R. GODDARD, pp. 175–189. Oxford Univ. Press.  
 TIEKINK, E. R. T. (1989). *Z. Kristallogr.* **189**, 79–85.

*Acta Cryst.* (1992). **C48**, 2033–2035

## 4',6,7-Trimethoxyisoflavone

BY M. SHOJA

*Chemistry Department, Fordham University, Bronx, NY 10458, USA*

(Received 18 November 1991; accepted 24 February 1992)

**Abstract.**  $C_{18}H_{16}O_5$ ,  $M_r = 312.33$ , orthorhombic,  $P2_12_12_1$ ,  $a = 7.296$  (1),  $b = 13.099$  (2),  $c = 15.754$  (2)  $\text{\AA}$ ,  $V = 1505.6$  (4)  $\text{\AA}^3$ ,  $Z = 4$ ,  $D_x = 1.38 \text{ g cm}^{-3}$ , Cu  $K\alpha$  radiation,  $\lambda = 1.5418 \text{ \AA}$ ,  $\mu = 8.5 \text{ cm}^{-1}$ ,  $F(000) = 656$ ,  $T = 293 \text{ K}$ , final  $R = 0.055$  for 1132 observed reflections. 4',6,7-Trimethoxyisoflavone is not planar. The dihedral angle between the phenyl ring and the mean plane of the  $\gamma$ -pyrone moiety is  $-42.8$  (1) $^\circ$ . The methoxy groups at C(7) and C(4') lie close to the planes of their respective rings. The C(6) methoxy group is oriented out of the plane of the  $\gamma$ -benzopyrone ring with a torsion angle C(12)—O(4)—C(6)—C(5) =  $-19.8$  (5) $^\circ$ .

**Experimental.** The title compound (Fig. 1) was purchased from the Indofine Chemical Company. Crystals grown from methanol solution. Data collected on an Enraf-Nonius CAD-4 diffractometer, graphite monochromator. Crystal dimensions  $0.20 \times 0.20 \times$

$0.50 \text{ mm}$ . Cell parameters measured on the diffractometer using 25 reflections in the  $2\theta$  range  $20$ – $40^\circ$ . Range of indices  $0 \leq h \leq 8$ ,  $0 \leq k \leq 14$ ,  $0 \leq l \leq 17$  ( $\theta \leq 60^\circ$ ). Three standards (140, 011, 102) measured after every 200 reflections showed a variation of 0.2%. No absorption corrections. Lorentz and polarization corrections. 1331 unique reflections measured, 1132 observed reflections with  $|F_o| > 3\sigma(|F_o|)$ . Direct methods (*MULTAN11/82*; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) used for structure determination. H atoms located by difference Fourier synthesis. Anisotropic full-matrix least-squares refinement (on  $F$ ) for non-H atoms, isotropic for H atoms. In the last cycle, the H atoms were fixed at idealized positions (0.94–0.98  $\text{\AA}$ ) with Debye-Waller temperature parameters fixed at  $5.0 \text{ \AA}^2$ .  $\sum w(|F_o| - |F_c|)^2$  minimized,  $w = 4F^2/[\sigma(I)^2 + (pF^2)^2]$ ,  $p = 0.04$ .  $wR = 0.055$ , maximum  $\Delta/\sigma = 0.05$ . Maximum peak height in the final difference

Table 1. Final fractional coordinates and equivalent isotropic temperature factors for non-H atoms with *e.s.d.*'s in parentheses

$$B_{eq} = (4/3) \sum_i \sum_j \beta_{ij} a_i \cdot a_j$$

	x	y	z	$B_{eq}(\text{\AA}^2)$
O(1)	0.4816 (4)	0.3363 (2)	0.1415 (1)	3.95 (5)
O(2)	0.5400 (4)	0.0356 (2)	0.2030 (1)	4.39 (6)
O(3)	0.5308 (4)	0.2611 (2)	-0.1525 (1)	4.31 (5)
O(4)	0.5672 (4)	0.0692 (2)	-0.1239 (1)	4.26 (5)
O(5)	0.4290 (4)	0.0962 (2)	0.5940 (1)	4.58 (6)
C(1')	0.4698 (5)	0.1800 (2)	0.3399 (2)	3.20 (7)
C(2)	0.4698 (6)	0.3038 (2)	0.2228 (2)	3.79 (7)
C(2')	0.5545 (5)	0.2392 (2)	0.4016 (2)	3.74 (7)
C(3)	0.4845 (5)	0.2066 (2)	0.2483 (2)	3.26 (7)
C(3')	0.5431 (6)	0.2145 (3)	0.4878 (2)	3.88 (7)
C(4)	0.5173 (5)	0.1264 (2)	0.1845 (2)	3.26 (7)
C(4')	0.4466 (6)	0.1289 (3)	0.5129 (2)	3.47 (7)
C(5)	0.5437 (5)	0.0939 (2)	0.0282 (2)	3.26 (7)
C(5')	0.3569 (5)	0.0703 (3)	0.4515 (2)	3.49 (7)
C(6')	0.3699 (5)	0.0945 (2)	0.3665 (2)	3.31 (7)
C(6)	0.5461 (5)	0.1287 (2)	-0.0536 (2)	3.18 (7)
C(7)	0.5248 (5)	0.2344 (2)	-0.0699 (2)	3.40 (7)
C(8)	0.5044 (5)	0.3023 (2)	-0.0038 (2)	3.50 (7)
C(9)	0.5031 (5)	0.2651 (2)	0.0792 (2)	3.15 (6)
C(10)	0.5208 (5)	0.1624 (2)	0.0971 (2)	2.99 (7)
C(11)	0.5256 (6)	0.3678 (3)	-0.1724 (2)	4.62 (9)
C(12)	0.6452 (6)	-0.0284 (3)	-0.1127 (2)	4.26 (8)
C(13)	0.5262 (8)	0.1482 (4)	0.6590 (2)	6.5 (1)

Table 2. Bond lengths ( $\text{\AA}$ ) and angles ( $^\circ$ ) with *e.s.d.*'s in parentheses

O(1)—C(2)	1.352 (4)	C(2')—C(3')	1.399 (5)
O(1)—C(9)	1.363 (4)	C(3)—C(4)	1.474 (4)
O(2)—C(4)	1.235 (4)	C(3')—C(4')	1.381 (5)
O(3)—C(7)	1.349 (4)	C(4)—C(10)	1.455 (4)
O(3)—C(11)	1.433 (4)	C(4')—C(5')	1.399 (5)
O(4)—C(6)	1.363 (4)	C(5)—C(6)	1.366 (4)
O(4)—C(12)	1.410 (4)	C(5)—C(10)	1.418 (4)
O(5)—C(4')	1.354 (4)	C(5')—C(6')	1.378 (5)
O(5)—C(13)	1.420 (5)	C(6)—C(7)	1.416 (4)
C(1')—C(2')	1.389 (5)	C(7)—C(8)	1.376 (4)
C(1')—C(3)	1.488 (4)	C(8)—C(9)	1.396 (4)
C(1')—C(6')	1.400 (5)	C(9)—C(10)	1.380 (4)
C(2)—C(3)	1.340 (4)		
C(2)—O(1)—C(9)	118.2 (2)	C(8)—C(9)—O(1)	115.9 (3)
C(3)—C(2)—O(1)	125.4 (3)	C(9)—C(10)—C(4)	120.5 (3)
C(4)—C(3)—C(2)	119.0 (3)	C(9)—C(10)—C(5)	118.1 (3)
C(4)—C(3)—C(1')	120.4 (3)	C(2')—C(1')—C(3)	121.0 (3)
C(3)—C(4)—O(2)	123.1 (3)	C(2')—C(1')—C(6')	118.0 (3)
C(10)—C(4)—O(2)	122.2 (3)	C(1')—C(2)—C(3)	121.6 (3)
C(3)—C(4)—C(10)	114.6 (3)	C(2')—C(3')—C(4')	119.7 (3)
C(10)—C(5)—C(6)	120.8 (3)	C(3')—C(4')—C(5')	119.1 (3)
C(5)—C(6)—C(7)	119.7 (3)	C(3')—C(4')—O(5)	125.1 (3)
C(5)—C(6)—O(4)	125.2 (3)	C(4')—C(5')—C(6')	120.9 (3)
C(6)—C(7)—C(8)	120.4 (3)	C(5')—C(6')—C(1')	120.7 (3)
C(6)—C(7)—O(3)	115.1 (3)	C(7)—O(3)—C(11)	117.6 (2)
C(7)—C(8)—C(9)	118.9 (3)	C(6)—O(4)—C(12)	117.5 (3)
C(8)—C(9)—C(10)	122.1 (3)	C(4')—O(5)—C(13)	118.8 (3)

Fourier map  $0.26 \text{ e \AA}^{-3}$ ,  $S = 1.913$  for 209 variables. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). Calculations performed using Enraf-Nonius SDP (B. A. Frenz & Associates, Inc., 1984). Atomic parameters are given in Table 1\* and bond distances and angles

\* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55231 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR0410]

in Table 2. The atomic numbering scheme is given in Fig. 1 and the packing diagram is shown in Fig. 2.

**Related literature.** The C(4)—C(3)—C(1')—C(6') torsion angle of  $-42.8^\circ$  is the lowest in any reported isoflavone system; licoricone monobromoacetate (Kaneda, Iitaka & Shibata, 1973) has the highest value ( $75^\circ$ ) as a result of the bulky bromoacetate group at C(6'). The C(1')—C(3) distance of  $1.488 \text{ \AA}$  (Table 2) is the same as in 7-hydroxy-4'-methoxyisoflavone (Shoja, 1992; torsion angle  $46^\circ$ ) and g enisteine (Breton, Precigoux, Courseille & Hos-

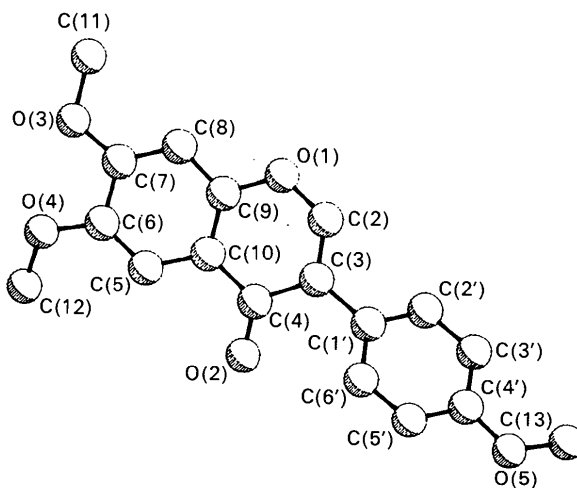


Fig. 1. Numbering of atoms and conformation of the molecule.

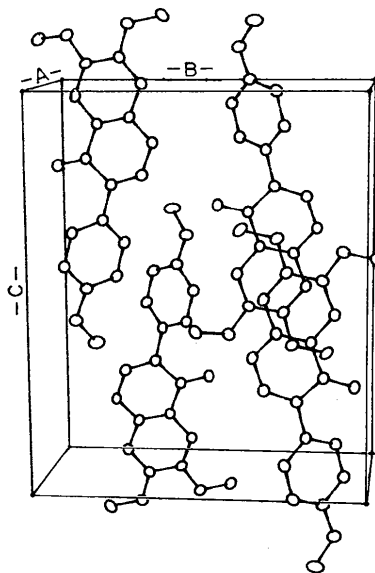


Fig. 2. Packing diagram of the molecule.

pital, 1975; dihedral angle 53°). A value of 1.51 Å was assigned to C(1')—C(3) in licoricone.

### References

B. A. FRENZ & ASSOCIATES, INC. (1984). *SDP Structure Determination Package*. College Station, Texas, USA, and Enraf-Nonius, Delft, The Netherlands.

BRETON, P., PRECIGOUX, G., COURSEILLE, C. & HOSPITAL, M. (1975). *Acta Cryst.* **B31**, 921–923.  
KANEDA, M., IITAKA, Y. & SHIBATA, S. (1973). *Acta Cryst.* **B29**, 2827–2832.  
MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1982). *MULTAN11/82. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.  
SHOJA, M. (1992). *Z. Kristallogr.* **199**, 161–166.

*Acta Cryst.* (1992). **C48**, 2035–2036

## Structure of a Cinnamic Acid Derivative

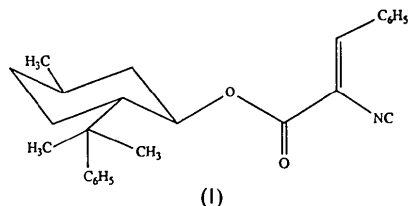
BY MICHAEL DYRBUSCH AND ERNST EGERT\*

*Institut für Organische Chemie der Universität Frankfurt, Niederurseler Hang, W-6000 Frankfurt am Main 50, Germany*

(Received 6 January 1992; accepted 18 February 1992)

**Abstract.** (1*R*,2*S*,5*R*)-5-Methyl-2-(1-methyl-1-phenylethyl)cyclohexyl (*Z*)-2-isocyano-3-phenylpropenoate, C<sub>26</sub>H<sub>29</sub>NO<sub>2</sub>, *M<sub>r</sub>* = 387.5, orthorhombic, *P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>, *a* = 9.777 (1), *b* = 10.211 (1), *c* = 22.585 (2) Å, *V* = 2254.7 (3) Å<sup>3</sup>, *Z* = 4, *D<sub>x</sub>* = 1.142 Mg m<sup>-3</sup>, λ(Mo *K*α) = 0.7107 Å, μ = 0.07 mm<sup>-1</sup>, *F*(000) = 832, *T* = 293 K, final *R* = 0.043 for 1982 unique observed reflections. The isocyano group is *syn* to the carbonyl O atom and *cis* to the phenyl group, which is rotated by 29° from the plane of the adjacent conjugated π system. One side of the C=C double bond is shielded by the second phenyl ring (distance approximately 3.5 Å).

**Experimental.** Crystals of (I) obtained from methanol at 243 K were yellow rectangular prisms, 0.2 × 0.5 × 0.8 mm. A Stoe four-circle diffractometer was



used for data collection with monochromated Mo *K*α radiation. Lattice parameters were measured by centring 60 reflections with 20 < 2θ < 25°. Data were collected with the profile-fitting method (Clegg, 1981); 4274 measured intensities, 2θ<sub>max</sub> = 50° (−11 ≤ *h* ≤ 11, 0 ≤ *k* ≤ 12, 0 ≤ *l* ≤ 26). Three standard reflections measured every 100 reflections showed no

Table 1. Atomic coordinates (× 10<sup>4</sup>) and equivalent isotropic thermal parameters (Å<sup>2</sup> × 10<sup>3</sup>)

Equivalent isotropic *U* is defined as one third of the trace of the orthogonalized *U<sub>i</sub>* tensor.

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U<sub>eq</sub></i>
O(1)	3382 (2)	−1310 (2)	7135 (1)	51 (1)
C(2)	3155 (2)	−2319 (3)	7491 (1)	50 (1)
C(3)	3894 (3)	−2187 (3)	8062 (1)	54 (1)
C(4)	4781 (3)	−1242 (3)	8191 (1)	59 (1)
O(2)	2450 (2)	−3242 (2)	7376 (1)	73 (1)
N(31)	3640 (2)	−3230 (2)	8444 (1)	67 (1)
C(32)	3385 (4)	−4115 (4)	8741 (1)	98 (1)
C(41)	5634 (3)	−1055 (3)	8721 (1)	60 (1)
C(42)	6849 (3)	−380 (3)	8656 (1)	70 (1)
C(43)	7738 (4)	−206 (3)	9128 (1)	86 (1)
C(44)	7406 (4)	−690 (3)	9671 (1)	94 (1)
C(45)	6195 (4)	−1342 (4)	9751 (1)	101 (1)
C(46)	5298 (3)	−1535 (4)	9279 (1)	85 (1)
C(1')	2782 (2)	−1344 (2)	6538 (1)	47 (1)
C(2')	3913 (2)	−1782 (3)	6124 (1)	52 (1)
C(3')	3469 (3)	−1761 (3)	5477 (1)	58 (1)
C(3'')	4629 (3)	−2228 (4)	5076 (1)	85 (1)
C(4')	2985 (3)	−391 (3)	5329 (1)	65 (1)
C(5')	1836 (2)	58 (2)	5733 (1)	58 (1)
C(6')	2221 (2)	12 (2)	6395 (1)	44 (1)
C(7')	1013 (2)	453 (2)	6811 (1)	47 (1)
C(8')	343 (3)	1717 (3)	6571 (1)	61 (1)
C(9')	−112 (2)	−591 (3)	6837 (1)	63 (1)
C(71')	1548 (2)	789 (2)	7432 (1)	47 (1)
C(72')	2529 (3)	1758 (2)	7503 (1)	58 (1)
C(73')	3017 (3)	2127 (3)	8047 (1)	71 (1)
C(74')	2552 (3)	1524 (4)	8551 (1)	81 (1)
C(75')	1581 (3)	561 (3)	8497 (1)	78 (1)
C(76')	1079 (3)	201 (3)	7947 (1)	62 (1)

significant intensity variation. Intensities were corrected for Lorentz and polarization factors, but not for absorption. 2262 unique reflections were collected (*R*<sub>int</sub> = 0.021) of which 1982 with |*F*| > 3σ(*F*) were treated as observed. The structure was solved by direct methods. All H atoms were located by difference synthesis and refined with fixed individual displacement parameters [*U*(H) = 1.2*U*<sub>eq</sub>(C)] using a riding model with C—H = 0.96 Å. Anisotropic

\* To whom correspondence should be addressed.