

$$w = 1/[\sigma^2(F_o^2) + (0.059P)^2 + 0.5656P]$$

where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$

Scattering factors from  
*International Tables for  
 Crystallography* (Vol. C)

Siemens (1994). *XP. Molecular Graphics Program*. Version 5.03. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.  
 Stetter, H., Mayer, J., Schwarz, M. & Wulff, K. (1960). *Chem. Ber.* **93**, 226–230.  
 Stetter, H., Schwarz, M. & Hirschhorn, A. (1959). *Chem. Ber.* **92**, 1629–1635.

Table 1. Selected geometric parameters (Å, °)

N—C11	1.345 (2)	O—C11	1.235 (2)
N—C1	1.474 (2)	C11—C12	1.501 (2)
C11—N—C1	125.13 (12)	C2—C1—C10	108.61 (12)
N—C1—C2	110.62 (11)	C8—C1—C10	108.44 (12)
N—C1—C8	111.12 (12)	O—C11—N	123.53 (14)
C2—C1—C8	110.07 (12)	O—C11—C12	120.02 (14)
N—C1—C10	107.91 (11)	N—C11—C12	116.44 (13)
C11—N—C1—C2	61.6 (2)	C1—N—C11—O	2.8 (2)
C11—N—C1—C8	−60.9 (2)	C1—N—C11—C12	−176.49 (13)
C11—N—C1—C10	−179.69 (13)		

Table 2. Hydrogen-bonding geometry (Å, °)

D—H...A	D—H	H...A	D...A	D—H...A
N—H0...O <sup>i</sup>	0.91 (2)	2.04 (2)	2.928 (2)	168 (1)
C12—H12A...O <sup>i</sup>	0.98	2.42	3.287 (2)	148
C2—H2B...O	0.99	2.51	3.107 (2)	118
C8—H8A...O	0.99	2.46	3.064 (2)	119

Symmetry code: (i)  $x, 1 - y, z - \frac{1}{2}$ .

H-atom treatment: NH coordinates free, U fixed; rigid methyl group (allowed to rotate but not tip); others riding. Despite the  $\beta$  angle being close to 90°, no higher Laue symmetry was appropriate.

Data collection: P3 (Nicolet XRD Corporation, 1987). Cell refinement: P3. Data reduction: XDISK (Nicolet XRD Corporation, 1987). Program(s) used to solve structure: SHELXS86 (Sheldrick, 1990). Program(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Molecular graphics: XP (Siemens, 1994). Software used to prepare material for publication: SHELXL93.

We thank the Fonds der Chemischen Industrie for financial support and Mr A. Weinkauff for technical assistance.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG1344). Services for accessing these data are described at the back of the journal.

## References

- Amini, Bishop, R., Craig, D. C., Rae, A. D. & Scudder, M. L. (1989). *J. Chem. Soc. Perkin Trans. 1*, pp. 733–737.  
 Bakke, J. M. & Storm, C. B. (1989). *Acta Chem. Scand.* **43**, 399–400.  
 Cammenga, H. K., Epple, M., Blaschette, A. & Nèveke, M. (1989). *Thermochim. Acta*, **151**, 171–178.  
 Desiraju, G. R. (1996). *Acc. Chem. Res.* **29**, 441–449.  
 Kevill, D. N. & Weill, F. L. (1970). *J. Org. Chem.* **35**, 2526–2529.  
 Klimochkin, Yu. N., Bagrii, E. I., Dolgoplova, T. N. & Moiseev, I. K. (1988). *Izv. Akad. Nauk SSSR Ser. Khim.* **37**, 878–880; *Bull. Acad. Sci. USSR Div. Chem. Sci.* **37**, 757–759.  
 Nicolet XRD Corporation (1987). *Nicolet P3 Data Collection Operators Manual*. Nicolet XRD Corporation, 10061 Bubb Road, Cupertino, California 95014, USA.  
 Prozorovskii, A. E., Tafeenko, V. A., Rybakov, V. B., Shokova, E. A. & Kovalev, V. V. (1987). *Zh. Strukt. Khim.* **28**, No. 2, 98–106; *J. Struct. Chem.* **28**, 243–251.  
 Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.  
 Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

*Acta Cryst.* (1997). **C53**, 1436–1438

## 3-Cyano-6-(4-methoxyphenyl)-5-methyl-4-methylthio-2H-pyran-2-one

ABUL AZIM,<sup>a</sup> VIRINDER S. PARMAR<sup>a</sup> AND WILLIAM ERRINGTON<sup>b</sup>

<sup>a</sup>Department of Chemistry, University of Delhi, Delhi 110 007, India, and <sup>b</sup>Department of Chemistry, University of Warwick, Coventry CV4 7AL, England. E-mail: w.errington@warwick.ac.uk

(Received 23 January 1997; accepted 15 May 1997)

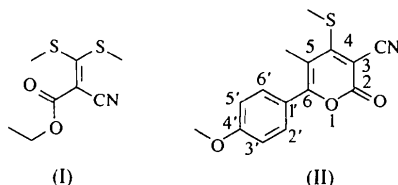
## Abstract

In the title compound, C<sub>15</sub>H<sub>13</sub>NO<sub>3</sub>S, the two six-membered rings are inclined at an angle of 38.9(1)°. The conformations of the ring substituents are discussed and compared with the situation when the 5-methyl substituent is replaced with an H atom.

## Comment

Various 4-hydroxy-2H-pyran-2-ones and their ether derivatives have been found to be effective antibacterial and antifungal agents (Israili & Smissman, 1976; Kretzschmar, Meyer, Teschendorf & Zoellner, 1969). The addition of enolate anions derived from aryl-alkyl ketones to doubly active ketene dithioacetals has been shown to give 4-methylthio-2-pyrones ( $\delta$ -lactones) in low to moderate yields (Hatada *et al.*, 1975). These lactones have been found to be vulnerable to nucleophilic attack because of the presence of several electrophilic centres, and provide a single-step synthesis for a variety of heterocycles such as pyrazoles, isoxazoles, 2-aminopyridines and 2-iminopyridines; these reactions illustrate the tremendous synthetic potential of such compounds (Ram, Verma, Hussaini & Shoeb, 1991; Ram, Hussaini, Singh & Shoeb, 1993; Hussaini *et al.*, 1994; Ram *et al.*, 1994). Lactones with a methyl group in the C-5 position have not been synthesized previously and so we have synthesized several new 3-cyano-5-methyl-4-methylthio-6-aryl-2H-pyran-2-ones with a view to screening them for antibacterial and antifungal activity.

In this investigation, 4-methoxypropiofenone has been condensed with ethyl 2-cyano-3,3-bis(methylthio)acrylate, (I), to yield the novel title lactone, (II). This new compound was characterized by spectral data and its structure confirmed by X-ray studies.



The molecular structure of (II) is illustrated in Fig. 1. The bond lengths and angles are unexceptional (Allen *et al.*, 1987); the  $C_{sp^2}-S$  distance [1.738 (2) Å] is shorter than the  $C_{sp^3}-S$  distance [1.798 (3) Å] and is indicative of some double bonding in the  $sp^2$  case. The best planes through the two six-membered rings in the molecule are inclined at an angle of 38.9 (1)°. Alternatively, this deviation from planarity can be quantified by noting that the C4—C5—C8—O2 torsion angle is 137.6 (2)°, and this can be compared with an average value of 171.6 (6)° for the system (Kumar, Kumar, Parmar & Errington, 1996) without the methyl substituent (*i.e.* C15 in Fig. 1). Clearly one effect of the methyl substituent is to twist the molecule away from planarity in order to lessen the steric interactions. A related effect is that in the title compound the methyl group attached to the S atom points towards the cyanide group [*i.e.* the C14—S1—C10—C11 torsion angle is  $-14.7$  (2)°], but points in the other direction when the methyl ring substituent is missing [corresponding torsion angle is  $-179.3$  (5)°]. The methoxy group is almost coplanar with the phenyl

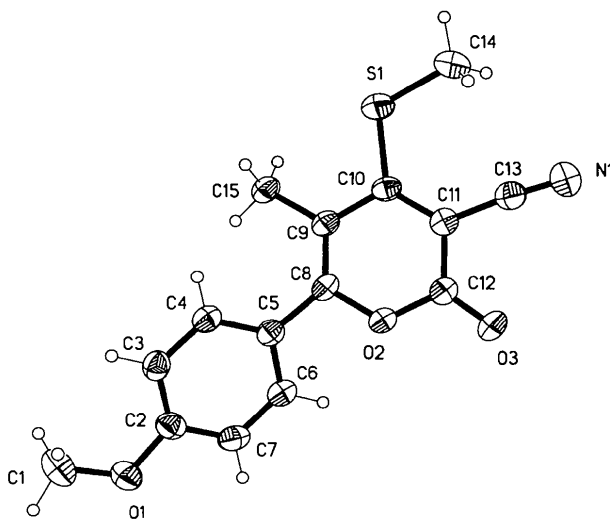


Fig. 1. View of the molecule showing the atomic numbering. Displacement ellipsoids are drawn at the 50% probability level.

group as shown by the C1—O1—C2—C3 torsion angle of  $-3.2$  (3)°.

The molecules pack together in the lattice in a head-to-tail fashion. The shortest intermolecular distances between the centroids of adjacent six-membered rings are 3.636 (3) Å.

## Experimental

A mixture of ethyl 2-cyano-3,3-bis(methylthio)acrylate [(I); 4.34 g, 0.02 mol], 4-methoxypropiofenone (6 ml, 0.02 mol), powdered KOH (2.24 g, 0.04 mol) and DMF (50 ml) was stirred at 305 K for 16 h. The mixture was poured onto ice water (300 ml) and stirred at room temperature for 4 h. The yellow solid that separated was filtered, washed with water and recrystallized from acetone to give yellow needles (3.15 g, 32% yield; m.p. 448 K); IR (nujol)  $\nu_{\max}$ : 2250 (CN), 1705 (C=O), 1620, 1380, 1310, 1250, 1165, 1010 and 840  $\text{cm}^{-1}$ ; UV (MeOH)  $\lambda_{\max}$ : 323 and 358 nm.  $^1\text{H NMR}$  (60 MHz, FT,  $\text{CDCl}_3$ )  $\delta$ : 2.42 (s, 3H, vinylic  $\text{CH}_3$ ), 3.21 (s, 3H,  $-\text{SCH}_3$ ), 4.02 (s, 3H,  $-\text{OCH}_3$ ), 7.01 (d,  $J = 8$  Hz, 2H), and 7.49 (d,  $J = 8$  Hz, 2H); EIMS,  $m/z$  (relative intensity): 287 ( $M^+$ , 10), 286 ( $M^+ - 1$ , 48), 258 (60), 243 (15), 184 (5), 135 (100), 107 (12), 92 (15) and 77 (25).

## Crystal data

$\text{C}_{15}\text{H}_{13}\text{NO}_3\text{S}$   
 $M_r = 287.32$   
 Monoclinic  
 $P2_1/c$   
 $a = 12.5744$  (4) Å  
 $b = 7.4608$  (2) Å  
 $c = 14.4604$  (4) Å  
 $\beta = 90.3040$  (10)°  
 $V = 1356.58$  (7) Å<sup>3</sup>  
 $Z = 4$   
 $D_x = 1.407$  Mg m<sup>-3</sup>  
 $D_m$  not measured

Mo  $K\alpha$  radiation  
 $\lambda = 0.71073$  Å  
 Cell parameters from 4149 reflections with  $I \geq 10\sigma(I)$   
 $\theta = 1.6$ – $26.0$ °  
 $\mu = 0.245$  mm<sup>-1</sup>  
 $T = 200$  (2) K  
 Slab  
 $0.28 \times 0.26 \times 0.12$  mm  
 Yellow

## Data collection

Siemens SMART CCD area-detector diffractometer  
 $\omega$  scans  
 Absorption correction: none  
 6873 measured reflections  
 2631 independent reflections

2197 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.046$   
 $\theta_{\max} = 26$ °  
 $h = -13 \rightarrow 16$   
 $k = -9 \rightarrow 7$   
 $l = -19 \rightarrow 19$

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.049$   
 $wR(F^2) = 0.110$   
 $S = 1.177$   
 2631 reflections  
 184 parameters  
 H atoms: see below  
 $w = 1/[\sigma^2(F_o^2) + (0.0275P)^2 + 0.8794P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.19$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.21$  e Å<sup>-3</sup>  
 Extinction correction: none  
 Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. Selected geometric parameters (Å, °)

S1—C10	1.738 (2)	O3—C12	1.209 (3)
S1—C14	1.798 (3)	N1—C13	1.145 (3)
O1—C2	1.369 (3)	C5—C8	1.468 (3)
O1—C1	1.429 (3)	C9—C15	1.508 (3)
O2—C12	1.369 (3)	C11—C13	1.435 (3)
O2—C8	1.374 (2)		
C10—S1—C14	108.08 (11)	C9—C10—S1	113.70 (15)
C2—O1—C1	117.8 (2)	C10—C11—C13	126.9 (2)
C12—O2—C8	123.27 (16)	C13—C11—C12	112.00 (19)
O1—C2—C3	124.6 (2)	O3—C12—O2	117.35 (19)
O1—C2—C7	115.4 (2)	O3—C12—C11	126.3 (2)
C9—C8—C5	129.82 (19)	O2—C12—C11	116.39 (18)
O2—C8—C5	108.77 (17)	N1—C13—C11	174.8 (3)
C11—C10—S1	126.93 (17)		
C1—O1—C2—C3	−3.2 (3)	C4—C5—C8—O2	137.6 (2)
C1—O1—C2—C7	176.6 (2)	C6—C5—C8—O2	−37.3 (3)
O1—C2—C3—C4	178.7 (2)	C14—S1—C10—C11	−14.7 (2)
O1—C2—C7—C6	−178.7 (2)	C14—S1—C10—C9	164.25 (17)
C12—O2—C8—C5	179.82 (18)	S1—C10—C11—C13	−7.6 (4)
C4—C5—C8—C9	−41.2 (3)	C10—C11—C13—N1	−146 (3)
C6—C5—C8—C9	143.8 (2)	C12—C11—C13—N1	31 (3)

The temperature of the crystal was controlled using the Oxford Cryosystems Cryostream Cooler (Cosier & Glazer, 1986). Data were collected over a hemisphere of reciprocal space, by a combination of three sets of exposures. Each set had a different  $\varphi$  angle for the crystal and each exposure of 10 s covered 0.3° in  $\omega$ . The crystal to detector distance was 5.01 cm. Coverage of the unique set was over 97% complete to at least 26° in  $\theta$ . Crystal decay was monitored by repeating the initial frames at the end of the data collection and analyzing the duplicate reflections. H atoms were added at calculated positions and refined using a riding model. Anisotropic displacement parameters were used for all non-H atoms; H atoms were given isotropic displacement parameters equal to 1.2 (or 1.5 for methyl H atoms) times the equivalent isotropic displacement parameter of the atom to which they are attached.

Data collection: SMART (Siemens, 1994b). Cell refinement: SAINT (Siemens, 1995). Data reduction: SAINT. Program(s) used to solve structure: SHELXTL/PC (Siemens, 1994a). Program(s) used to refine structure: SHELXL96 (Sheldrick, 1996). Molecular graphics: SHELXTL/PC. Software used to prepare material for publication: SHELXTL/PC.

We wish to acknowledge the use of the EPSRC's Chemical Database Service at Daresbury Laboratory (Fletcher, McMeeking & Parkin, 1996) for access to the Cambridge Structural Database (Allen & Kennard, 1993).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1145). Services for accessing these data are described at the back of the journal.

## References

- Allen, F. H. & Kennard, O. (1993). *Chem. Des. Autom. News*, **8**, 31–37.  
 Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–S19.  
 Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.  
 Fletcher, D. A., McMeeking, R. F. & Parkin, D. (1996). *J. Chem. Inf. Comput. Sci.* **36**, 746–749.

- Hatada, T., Sone, M., Tominaga, Y., Natsuki, R., Matsuda, Y. & Kobayashi, G. (1975). *Yakugaku Zasshi*, **95**, 623–628; *Chem. Abstr.* **83**, 206076.  
 Hussaini, F. A., Pragma, Ram, V. J., Singh, S. K., Nath, M., Shoeb, A. & Bhaduri, A. P. (1994). *J. Chem. Res. (S)*, p. 86.  
 Israili, Z. H. & Smisman, E. E. (1976). *J. Org. Chem.* **41**, 4070–4074.  
 Kretzschmar, R., Meyer, H. J., Teschendorf, H. J. & Zoellner, B. (1969). *Arch. Int. Pharmacodyn. Ther.* **180**, 475–491.  
 Kumar, A., Kumar, N., Parmar, V. S. & Errington, W. (1996). *Acta Cryst.* **C52**, 127–129.  
 Ram, V. J., Hussaini, F. A., Singh, S. K. & Shoeb, A. (1993). *J. Chem. Res. (S)*, pp. 110–111.  
 Ram, V. J., Mumtazuddin, S., Pragma, Haque, N., Hussaini, F. A., Shoeb, A. & Bhaduri, A. P. (1994). *J. Chem. Res. (S)*, pp. 354–355.  
 Ram, V. J., Verma, M., Hussaini, F. A. & Shoeb, A. (1991). *J. Chem. Res. (S)*, pp. 98–99.  
 Sheldrick, G. M. (1996). *SHELXL96. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.  
 Siemens (1994a). *SHELXTL/PC*. Version 5.0. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.  
 Siemens (1994b). *SMART. Data Processing Software for the SMART System*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.  
 Siemens (1995). *SAINTE Software Reference Manual*. Version 4. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

*Acta Cryst.* (1997). **C53**, 1438–1440

## 1-(3,4-Dimethoxyphenyl)-3-(3-methylphenyl)prop-2-en-1-one

NAWAL K. SHARMA,<sup>a</sup> RAJESH KUMAR,<sup>a</sup> VIRINDER S. PARMAR<sup>a</sup> AND WILLIAM ERRINGTON<sup>b</sup>

<sup>a</sup>Department of Chemistry, University of Delhi, Delhi 110 007, India, and <sup>b</sup>Department of Chemistry, University of Warwick, Coventry CV4 7AL, England. E-mail: w.errington@warwick.ac.uk

(Received 21 January 1997; accepted 2 May 1997)

## Abstract

The synthesis and structure of the title compound, C<sub>18</sub>H<sub>18</sub>O<sub>3</sub>, are described. The molecule is almost flat and the *ortho*-methoxy groups point away from each other.

## Comment

1,3-Diarylprop-2-enones (chalcones) constitute an important group of natural products and some have been found to possess a wide range of biological activities, viz antibacterial, antifungal and anti-inflammatory (Ahluwalia, Kaila & Bala, 1986; Bhat, Bhamaria, Patel, Bellare & Deliwala, 1972; Mathew, Subba Rao & Rambhav, 1984; Oganessian, Yakavenko, Khartyan,