

acid, dimethyl ester unit has been reported by LakshmiKantham, Cava & Carroll (1984). A niobium complex with a methylene-1,3-dithiole-4,5-bis-(trifluoromethyl) unit has been published by Amaudrut, Sala-Pala, Guerschais & Mercier (1990). Beer, Frew, Johnson & Paul (1970) have reported an isothiathiophene containing a methylene-1,3-dithiole-4-(4-bromophenyl) unit. The structure of (pivaloyl-methylene)-1,3-dithiole-4-pivaloyl has been reported by Galloy, Declercq & Van Meerssche (1978). A 4,5-unsubstituted methylene-1,3-dithiole derivative has been reported by Barnes, Paton & Nicholls (1982). The structures of the related compounds mentioned here are less accurate than that reported in this paper.

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## Structure of Methyl 2-(2,5-Dihydro-2-oxo-3-phenyl-5-furyl)lactate

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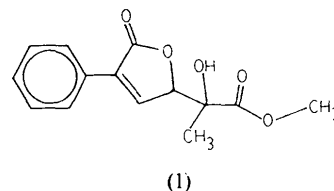
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**Abstract.**  $C_{14}H_{14}O_5$ ,  $M_r = 262.26$ , monoclinic,  $P2_1/n$ ,  $a = 11.319$  (3),  $b = 6.5115$  (15),  $c = 18.105$  (5) Å,  $\beta = 105.660$  (20)°,  $V = 1284.8$  (6) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.356$  g cm<sup>-3</sup>,  $\lambda(\text{Cu } K\alpha_1) = 1.54056$  Å,  $\mu = 8.26$  cm<sup>-1</sup>,  $F(000) = 553.89$ ,  $T = 295$  K,  $R = 0.036$ ,  $wR = 0.033$  for 1692 reflections with  $I > 2.5\sigma(I)$ . The structure determination established the identity of the product of a  $\text{Co}_2(\text{CO})_8$ -catalyzed carbonylation reaction with an epoxy alcohol.

**Experimental.** A colourless crystal of the title compound (1) was grown from THF solution. Cell parameters from 20 reflections in the  $2\theta$  range 85–90°. Data were collected on a Picker diffractometer using profile analysis (Grant & Gabe, 1978). Three standards measured every 100 reflections were used for scaling and had an overall drop in intensity of 6%. Averaging of symmetry-equivalent reflections gave a merging  $R$  value of 3.3%. Parameters related to crystal data and intensity collection are given in Table 1. The structure was solved by direct methods and refined by cycles of least-squares calculations. H atoms were located in a difference map and refined isotropically. Final atomic parameters are listed in Table 2. All non-H atoms were refined anisotropically. All computing was performed with the *NRCVAX* system of structure-solving programs (Gabe, Le Page, Charland, Lee & White, 1989).

Scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV).\*



An *ORTEP* plot of (1) is shown in Fig. 1. Bond lengths and angles, except those involving H atoms, are given in Table 3. A stereoplot showing packing of the unit cell has been included as Fig. 2. The structure consists of a planar lactone ring with maximum deviation from planarity 0.0098 (18) Å, rotated slightly from the plane of the phenyl ring, with a dihedral angle of 16.73 (6)°. The diastereomer adopts an *anti* configuration. The molecules are packed with the lactone rings approximately parallel to the  $b$  axis. The closest intermolecular approach is 2.14 (2) Å.

\* Lists of structure factors, anisotropic thermal parameters and least-squares-planes calculations have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54457 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR0352]

Table 1. Data collection and crystal parameters

Crystal size (mm)	0.3 × 0.3 × 0.1
Radiation, wavelength (Å)	Cu Kα <sub>1</sub> , 1.54056
Scan method	θ/2θ
2θ range (°)	2–120
<i>h</i> , <i>k</i> , <i>l</i> ranges	–12 to 12, 0 to 7, 0 to 20
Reflections measured	3003
No. of unique data	1915
No. with <i>I</i> > 2.5σ( <i>I</i> )	1692
No. of parameters	229
Secondary extinction*	2.87 (15)
<i>R</i> , <i>wR</i> for significant data†	0.036, 0.033
<i>R</i> , <i>wR</i> for all data	0.041, 0.033
Goodness of fit	4.85
Max. shift/e.s.d. in last cycle	0.082
Difference Fourier peaks (e Å <sup>-3</sup> )	0.15, –0.15

\* Secondary extinction as in Larson (1970). The refined parameter is the average mosaic block size in μm.

†  $w = 1/[\sigma(F_o)]^2$  with  $\sigma(F_o)$  based on counting statistics.

Table 2. Atomic parameters and *B*<sub>eq</sub> values with *e.s.d.*'s in parentheses

*B*<sub>eq</sub> for non-H atoms is the mean of the principal values for the anisotropic mean displacement tensor, *B*, transformed to orthogonal axes. For H atoms, *B*<sub>eq</sub> = *B*<sub>iso</sub>

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> <sub>eq</sub> (Å <sup>2</sup> )
O(1)	0.61867 (10)	0.68205 (16)	0.08890 (6)	4.43 (6)
O(2)	0.65665 (10)	0.47800 (15)	–0.00059 (6)	3.85 (5)
O(3)	0.88614 (11)	0.25240 (20)	0.04117 (6)	4.68 (6)
O(4)	0.88759 (12)	0.49622 (19)	–0.07669 (6)	5.06 (6)
O(5)	0.72120 (11)	0.34802 (18)	–0.15304 (6)	5.09 (6)
C(1)	0.61838 (16)	0.4484 (3)	0.23267 (11)	4.45 (9)
C(2)	0.60201 (18)	0.4194 (4)	0.30521 (11)	5.23 (11)
C(3)	0.57251 (18)	0.2278 (4)	0.32702 (12)	5.40 (11)
C(4)	0.55842 (19)	0.0666 (4)	0.27644 (13)	5.53 (11)
C(5)	0.57402 (17)	0.0944 (3)	0.20473 (12)	4.61 (9)
C(6)	0.60504 (13)	0.28577 (24)	0.18138 (9)	3.60 (8)
C(7)	0.62828 (14)	0.31063 (22)	0.10576 (9)	3.36 (8)
C(8)	0.63340 (14)	0.51065 (25)	0.06841 (9)	3.53 (8)
C(9)	0.66774 (15)	0.26098 (22)	–0.01183 (9)	3.56 (8)
C(10)	0.64822 (15)	0.1681 (3)	0.05813 (9)	3.73 (8)
C(11)	0.79221 (15)	0.21876 (24)	–0.02727 (9)	3.72 (8)
C(12)	0.79740 (23)	–0.0028 (3)	–0.05375 (14)	5.29 (11)
C(13)	0.80765 (16)	0.3699 (3)	–0.08733 (9)	3.92 (8)
C(14)	0.7207 (3)	0.5005 (5)	–0.21149 (13)	6.40 (14)
H(O3)	0.9415 (22)	0.349 (4)	0.0298 (12)	10.1 (8)
H(1)	0.6402 (15)	0.584 (3)	0.2203 (8)	5.0 (4)
H(2)	0.6117 (17)	0.544 (3)	0.3417 (11)	7.3 (5)
H(3)	0.5586 (17)	0.211 (3)	0.3789 (10)	7.0 (5)
H(4)	0.5424 (18)	–0.058 (3)	0.2912 (10)	6.7 (6)
H(5)	0.5607 (16)	–0.020 (3)	0.1685 (9)	6.0 (5)
H(9)	0.6016 (13)	0.2212 (22)	–0.0584 (8)	4.0 (3)
H(10)	0.6529 (14)	0.0217 (25)	0.0647 (8)	4.6 (4)
H(12A)	0.7311 (16)	–0.030 (3)	–0.1005 (9)	5.4 (4)
H(12B)	0.8735 (20)	–0.034 (3)	–0.0633 (11)	7.5 (6)
H(12C)	0.7911 (17)	–0.101 (3)	–0.0102 (10)	6.6 (5)
H(14A)	0.7996 (24)	0.506 (4)	–0.2217 (12)	9.5 (8)
H(14B)	0.6553 (21)	0.452 (3)	–0.2523 (12)	8.4 (6)
H(14C)	0.691 (3)	0.652 (5)	–0.1958 (16)	16.1 (12)

between O(4) and H(O3) at (2 – *x*, 1 – *y*, – *z*). There is no evidence for intermolecular hydrogen bonding.

**Related literature.** The reaction of β-epoxy alcohols with CO, catalyzed by Co [as Co<sub>2</sub>(CO)<sub>8</sub>] and the phase-transfer agent tris(polyoxaheptyl)amine, has been reported recently (Alper, Eisenstat & Satyanarayana, 1990). The title compound is the methylated derivative of the product when the starting alcohol is 3-(hydroxymethyl)-2-phenyloxirane. The product represents a novel route to an unsaturated lactone which may be viewed as a formal triple carbonylation reaction.

Table 3. Selected bond distances (Å) and bond angles (°) with *e.s.d.*'s in parentheses

O(1)—C(8)	1.2019 (20)	C(3)—C(4)	1.374 (3)
O(2)—C(8)	1.3611 (21)	C(4)—C(5)	1.369 (3)
O(2)—C(9)	1.4381 (18)	C(5)—C(6)	1.3912 (25)
O(3)—C(11)	1.4151 (19)	C(6)—C(7)	1.4711 (23)
O(4)—C(13)	1.1991 (22)	C(7)—C(8)	1.4758 (22)
O(5)—C(13)	1.3284 (19)	C(7)—C(10)	1.3273 (24)
O(5)—C(14)	1.450 (3)	C(9)—C(10)	1.4732 (24)
C(1)—C(2)	1.388 (3)	C(9)—C(11)	1.5332 (25)
C(1)—C(6)	1.3896 (24)	C(11)—C(12)	1.526 (3)
C(2)—C(3)	1.377 (3)	C(11)—C(13)	1.5113 (23)
C(8)—O(2)—C(9)	109.22 (12)	O(2)—C(8)—C(7)	108.90 (13)
C(13)—O(5)—C(14)	115.92 (17)	O(2)—C(9)—C(10)	104.06 (13)
C(2)—C(1)—C(6)	120.69 (19)	O(2)—C(9)—C(11)	108.76 (13)
C(1)—C(2)—C(3)	119.97 (20)	C(10)—C(9)—C(11)	116.20 (13)
C(2)—C(3)—C(4)	119.65 (21)	C(7)—C(10)—C(9)	111.26 (14)
C(3)—C(4)—C(5)	120.66 (22)	O(3)—C(11)—C(9)	108.83 (13)
C(4)—C(5)—C(6)	120.95 (20)	O(3)—C(11)—C(12)	109.74 (14)
C(1)—C(6)—C(5)	118.07 (17)	O(3)—C(11)—C(13)	108.89 (14)
C(1)—C(6)—C(7)	121.58 (15)	C(9)—C(11)—C(12)	109.99 (15)
C(5)—C(6)—C(7)	120.30 (15)	C(9)—C(11)—C(13)	107.75 (13)
C(6)—C(7)—C(8)	124.23 (14)	C(12)—C(11)—C(13)	111.58 (15)
C(6)—C(7)—C(10)	129.21 (14)	O(4)—C(13)—C(11)	123.98 (15)
C(8)—C(7)—C(10)	106.56 (15)	O(4)—C(13)—C(11)	124.05 (14)
O(1)—C(8)—O(2)	120.38 (14)	O(5)—C(13)—C(11)	111.97 (15)
O(1)—C(8)—C(7)	130.70 (16)		

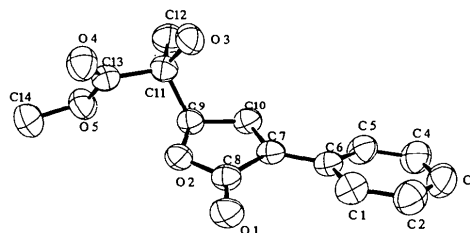


Fig. 1. ORTEPII (Johnson, 1976) plot of (1) showing the numbering scheme. Ellipsoids are drawn at 50% probability. H atoms have been omitted for clarity.

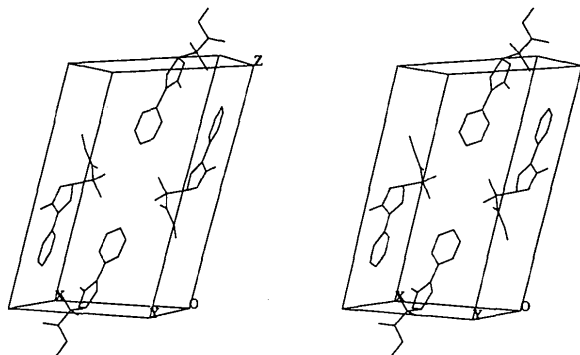


Fig. 2. Stereoplot of the unit cell showing the packing arrangement.

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