

C3	0.1283 (4)	0.0934 (2)	-0.0941 (5)	0.051 (1)
C4	-0.2889 (7)	0.1775 (4)	0.0221 (11)	0.089 (1)
C5	0.3413 (5)	0.1576 (4)	0.2350 (8)	0.077 (1)
C6	0.2480 (7)	0.0376 (4)	-0.3189 (8)	0.080 (1)

Table 4. Selected geometric parameters (Å, °) for (III)

O1—C1	1.218 (4)	N2—C2	1.370 (4)
O2—C2	1.213 (4)	N2—C1	1.392 (5)
O3—C3	1.323 (4)	N2—C4	1.464 (5)
O3—C6	1.453 (5)	N3—C3	1.364 (5)
N1—C3	1.284 (4)	N3—C2	1.382 (4)
N1—C1	1.365 (5)	N3—C5	1.452 (5)
C3—O3—C6	116.5 (3)	O1—C1—N2	119.5 (3)
C3—N1—C1	118.2 (3)	N1—C1—N2	118.0 (3)
C2—N2—C1	123.4 (3)	O2—C2—N2	123.3 (3)
C2—N2—C4	117.0 (4)	O2—C2—N3	121.1 (3)
C1—N2—C4	119.4 (4)	N2—C2—N3	115.6 (3)
C3—N3—C2	118.6 (3)	N1—C3—O3	121.7 (3)
C3—N3—C5	122.8 (3)	N1—C3—N3	126.1 (3)
C2—N3—C5	118.4 (3)	O3—C3—N3	112.2 (3)
O1—C1—N1	122.5 (4)		

For both compounds, program(s) used to solve structures: *SHELXS86* (Sheldrick, 1985); program(s) used to refine structures: *SHELXL93* (Sheldrick, 1993).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1180). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## 5,5-Dimethyl-1,3-dioxacyclohexane-2-spiro(2', $\gamma$ -dimethyl-3'-oxo-2'-cyclohexaneacrylic Acid)

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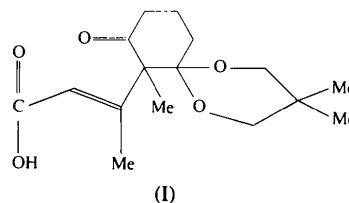
(Received 19 December 1994; accepted 24 April 1995)

## Abstract

The cyclohexanone ring in the title compound, C<sub>16</sub>H<sub>24</sub>O<sub>5</sub>, adopts a chair conformation with the methyl and acrylic acid groups in axial and equatorial positions, respectively. The dihedral angle between the planes of the two rings is 73(1)°. The bond angles around the spiro C atom are all nearly tetrahedral [104.8(2)–113.0(2)°].

## Comment

With the aim of elucidating the influence of the spiro C atom on ring-skeleton conformation, we have examined the conformation of several cyclohexanone-2-methylacrylic acid derivatives; the results of the X-ray analysis of one of these derivatives, (I), is reported here.



The two mean ring planes form a dihedral angle of 73°; the least-squares planes calculation indicates that the two rings are not individually planar. Following the Cremer & Pople (1975) notation, the ring-puckering parameters for the cyclohexanone ring are  $Q_2$

= 0.155 (3) Å and  $\theta_2 = +78.1 (2)^\circ$ . The most unusual feature of the angles is the pattern around C(10), the spiro C atom. The six angles around C(10) are 110.5 (2), 106.6 (2), 111.4 (2), 104.8 (2), 110.3 (2) and 113.0 (2)°. Of the three larger angles, two are endocyclic: O(4)—C(10)—O(5) and O(5)—C(10)—C(9) with values of 110.5 (2) and 110.3 (2)°, respectively. The magnitudes of these angles are obviously greater than the normal  $sp^3$  value as a result of the strain of bonding to the O atoms within the compressed ring system. The other angle of the three largest is C(9)—C(10)—C(5) with a value of 113.0 (2)°; this is the result of the strain of the methyl and acrylic acid groups bonded at the C(5) atom. The two ring O atoms are in an axial positions (with respect to the opposite ring). The smallest angle around the spiro C atom is O(4)—C(10)—C(5) with a value of 106.6 (2)°; this is due to the attraction between the ring O atoms and the H atoms of the methyl group on the opposite ring. Atoms C(15) and C(16) are equatorial and axial, respectively.

In the acrylic acid group {O(1)—C(1)[—O(2)]—C(2)}, the delocalization is well distributed over the three bonds. Consequently, the O(2)—C(1) distance of 1.224 (3) Å is nearer to those observed in aldehydes and ketones (Sutton, 1965) and in  $\alpha$ -oxalic acid (Derissen & Smit, 1974). The C(1)—C(2) bond length is in agreement with the values observed in the two analogous derivatives analysed by Koer, De Kok & Romers (1972), and by Haltiwanger, Walba & Wand (1980).

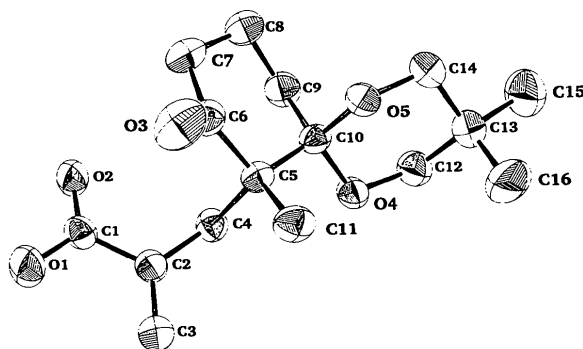


Fig. 1. View of the title compound with displacement ellipsoids plotted at the 50% probability level.

## Experimental

### Crystal data

$C_{16}H_{24}O_5$   
 $M_r = 296.36$   
 Monoclinic  
 $P2_1/a$   
 $a = 13.049 (1) \text{ \AA}$   
 $b = 10.411 (1) \text{ \AA}$   
 $c = 12.317 (3) \text{ \AA}$   
 $\beta = 105.5 (1)^\circ$

Mo  $K\alpha$  radiation  
 $\lambda = 0.71073 \text{ \AA}$   
 Cell parameters from 25 reflections  
 $\theta = 4-20^\circ$   
 $\mu = 0.09 \text{ mm}^{-1}$   
 $T = 293 \text{ K}$   
 Rectangular

$V = 1612.8 \text{ \AA}^3$   
 $Z = 4$   
 $D_x = 1.22 \text{ Mg m}^{-3}$   
 $D_m = 1.25 \text{ Mg m}^{-3}$   
 $D_m$  measured by flotation

$0.35 \times 0.28 \times 0.12 \text{ mm}$   
 Colourless

### Data collection

CAD-4 diffractometer  
 $\omega$  scans  
 Absorption correction: none  
 3154 measured reflections  
 2831 independent reflections  
 1641 observed reflections  
 $[I > 3\sigma(I)]$

$\theta_{\max} = 25^\circ$   
 $h = -13 \rightarrow 13$   
 $k = -11 \rightarrow 0$   
 $l = 0 \rightarrow 14$   
 3 standard reflections monitored every 100 reflections  
 intensity decay: <5%

### Refinement

Refinement on  $F$   
 $R = 0.050$   
 $wR = 0.062$   
 1641 reflections  
 287 parameters  
 H atoms refined isotropically  
 $w = 1/[\sigma^2(F) + 0.0004F^2]$

$(\Delta/\sigma)_{\max} = 0.56$   
 $\Delta\rho_{\max} = 0.16 (3) \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = 0.08 (3) \text{ e \AA}^{-3}$   
 Extinction correction: none  
 Atomic scattering factors from Cromer & Waber (1974)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

$$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i \cdot a_j$$

	x	y	z	$B_{eq}$
O1	0.3798 (2)	0.5571 (2)	0.0200 (2)	5.65 (5)
O2	0.5010 (1)	0.6487 (2)	-0.0483 (2)	4.33 (4)
O3	0.3039 (2)	0.7030 (2)	-0.2995 (2)	5.88 (6)
O4	0.3802 (1)	1.1319 (2)	-0.1896 (1)	3.38 (4)
O5	0.3352 (1)	1.0401 (2)	-0.3693 (1)	3.48 (4)
C1	0.4175 (2)	0.6553 (3)	-0.0211 (2)	3.28 (6)
C2	0.3576 (2)	0.7779 (2)	-0.0286 (2)	3.32 (6)
C3	0.2801 (3)	0.7858 (3)	0.0214 (3)	5.19 (8)
C4	0.3970 (2)	0.8907 (3)	-0.0838 (2)	3.25 (6)
C5	0.3457 (2)	0.9074 (2)	-0.2125 (2)	2.85 (6)
C6	0.3692 (2)	0.7874 (3)	-0.2740 (2)	3.61 (6)
C7	0.4780 (3)	0.7723 (3)	-0.2939 (3)	4.74 (7)
C8	0.5262 (2)	0.8942 (3)	-0.3259 (3)	4.81 (7)
C9	0.5131 (2)	1.0059 (3)	-0.2497 (3)	3.98 (7)
C10	0.3955 (2)	1.0248 (2)	-0.2557 (2)	2.91 (6)
C11	0.2261 (2)	0.9244 (3)	-0.2343 (2)	4.00 (7)
C12	0.4013 (2)	1.2545 (3)	-0.2339 (2)	4.19 (7)
C13	0.3386 (2)	1.2741 (3)	-0.3551 (2)	4.40 (7)
C14	0.3557 (2)	1.1565 (3)	-0.4216 (2)	4.16 (7)
C15	0.3798 (3)	1.3927 (3)	-0.4039 (3)	6.9 (1)
C16	0.2198 (3)	1.2886 (4)	-0.3624 (3)	7.2 (1)

Table 2. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

O1—C1	1.294 (3)	C5—C6	1.534 (4)
O2—C1	1.224 (3)	C5—C10	1.544 (4)
O3—C6	1.206 (3)	C5—C11	1.522 (4)
O4—C10	1.425 (3)	C6—C7	1.512 (5)
O4—C12	1.443 (3)	C7—C8	1.514 (5)
O5—C10	1.420 (3)	C8—C9	1.532 (4)
O5—C14	1.431 (4)	C9—C10	1.530 (4)
C1—C2	1.487 (4)	C12—C13	1.513 (4)
C2—C3	1.319 (5)	C13—C14	1.522 (4)
C2—C4	1.514 (4)	C13—C15	1.532 (5)
C4—C5	1.557 (3)	C13—C16	1.536 (5)

C10—O4—C12	113.9 (2)	C6—C7—C8	115.3 (2)
C10—O5—C14	114.7 (2)	C7—C8—C9	110.8 (3)
O1—C1—O2	121.4 (2)	C8—C9—C10	110.0 (2)
O1—C1—C2	117.0 (2)	O4—C10—O5	110.5 (2)
O2—C1—C2	121.5 (2)	O4—C10—C5	106.6 (2)
C1—C2—C3	118.7 (3)	O4—C10—C9	111.4 (2)
C1—C2—C4	117.2 (2)	O5—C10—C5	104.8 (2)
C3—C2—C4	123.8 (3)	O5—C10—C9	110.3 (2)
C2—C4—C5	116.0 (2)	C5—C10—C9	113.0 (2)
C4—C5—C6	108.9 (2)	O4—C12—C13	112.6 (2)
C4—C5—C10	109.5 (2)	C12—C13—C14	107.7 (2)
C4—C5—C11	109.6 (2)	C12—C13—C15	110.0 (2)
C6—C5—C10	108.5 (2)	C12—C13—C16	110.0 (3)
C6—C5—C11	109.8 (2)	C14—C13—C15	108.7 (3)
C10—C5—C11	110.6 (2)	C14—C13—C16	109.7 (3)
O3—C6—C5	120.1 (3)	C15—C13—C16	110.6 (3)
O3—C6—C7	120.7 (3)	O5—C14—C13	111.6 (2)
C5—C6—C7	119.0 (2)		

A total of 20 atoms were located from an *E* map. The remaining atoms were located in succeeding difference Fourier syntheses. H atoms were located and their positions and isotropic displacement parameters were refined. The structure was refined by full-matrix least squares.

Data collection and cell refinement were performed using *CAD-4 Software* (Enraf-Nonius, 1989). The structure was solved by direct methods (*SIR88*; Burla *et al.*, 1989). All calculations were performed on a VAX computer using *MoLEN* (Fair, 1990). Molecular graphics were obtained using *ORTEP* (Johnson, 1965).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CR1184). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Methyl 6-Benzoyl-3,5-diphenyl-1,4-thiomorpholine-2-carboxylate 1,1-Dioxide

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## Abstract

The crystal structure determination of C<sub>25</sub>H<sub>23</sub>NO<sub>5</sub>S reveals that the thiomorpholine ring adopts a slightly distorted chair conformation. All the substituents on heterorings are equatorially oriented. There are no short intermolecular distances and the structure is stabilized with van der Waals interactions.

## Comment

Thiomorpholine derivatives have attracted considerable interest because of their various chemotherapeutic properties, such as the antihistaminic activity of promethazine, the psychotherapeutic action of chlorpromazine and the insecticidal and anthelmintic properties of prothiazine (Katritzky & Boulton, 1968). Since the chemical and physiological characteristics of these compounds are directly defined by the conformation of their molecules and their relevant structural data are limited, X-ray crystallographic studies on a series of thiomorpholine derivatives have been undertaken. Here, we report the crystal and molecular structure of the title compound, (I).

