

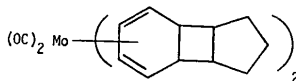
## Conformations of Fused Cycloalkanes in Organometallic Complexes. II.\* The Structure of Bis(tricyclo[6.3.0.0<sup>2,7</sup>]undeca-3,5-diene)dicarbonyl molybdenum, (C<sub>11</sub>H<sub>14</sub>)<sub>2</sub>Mo(CO)<sub>2</sub>†

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Bis(tricyclo[6.3.0.0<sup>2,7</sup>]undeca-3,5-diene)dicarbonylmolybdenum, (C<sub>11</sub>H<sub>14</sub>)<sub>2</sub>Mo(CO)<sub>2</sub>, crystallizes in space group *P*2<sub>1</sub>/*c* with *a* = 11.608 (2), *b* = 12.533 (3), *c* = 13.759 (2), *β* = 101.01 (1)°, *D<sub>x</sub>* = 1.50 g cm<sup>-3</sup> and *Z* = 4. Counter data were collected with the use of Mo *Kα* radiation. The structure, which was refined to *R*<sub>1</sub> = 0.030 and *R*<sub>2</sub> = 0.040, very closely parallels that of (C<sub>11</sub>H<sub>14</sub>)Fe(CO)<sub>3</sub>. A boat-shaped cyclohexadiene ring



is fused to a planar four-membered ring, which, in turn, is fused to an envelope-shaped cyclopentane ring. The consistency of the derived parameters for the three crystallographically independent ligands (two in this compound and one in the iron compound) suggests that in these complexes, and probably in related organometallic complexes, the conformation of the fused cycloalkanes is dependent entirely on intramolecular forces and that intermolecular forces have no consequential effect on the conformation.

### Introduction

For simple substituted cyclobutanes not involving fused rings, the dihedral angle in the four-membered ring is *ca.* 27 ± 3°, unless crystal packing imposes planarity on the ring by placing it on a center of symmetry. In a series of organometallic complexes containing fused cyclobutane rings we found a large variety of dihedral angles ranging from 1° to 16° [see Cotton & Frenz (1974) for a summary]. These molecules cannot possess an imposed center of symmetry and from a check of intermolecular distances it does not appear that non-bonded contacts could affect the conformation of the organic ring system. However, the energy for ring puckering is small; hence subtle changes in crystal packing might produce substantial changes in ring conformation. In order to proceed with a discussion of the factors responsible for the conformation of cyclobutane when fused to other cycloalkanes, it was desirable to have more concrete proof that crystal packing forces were not responsible for the ring conformations.

We have previously reported (Cotton, Day, Frenz, Hardcastle & Troup, 1973) the structure of tricyclo[6.3.0.0<sup>2,7</sup>]undeca-3,5-dienetricarbonyliron, (C<sub>11</sub>H<sub>14</sub>)Fe(CO)<sub>3</sub>. In this compound the six-membered ring has a boat conformation, the fused four-membered ring is planar, and the five-membered ring has an envelope conformation. The same ring system occurs doubly in (C<sub>11</sub>H<sub>14</sub>)<sub>2</sub>Mo(CO)<sub>2</sub>. Since this molecule cannot have the same crystalline environment as (C<sub>11</sub>H<sub>14</sub>)Fe(CO)<sub>3</sub>,

it provides a suitable test of the effects (or lack of effects) of crystal packing on the ring conformations.

There is an additional reason for our interest in the crystal structure of (C<sub>11</sub>H<sub>14</sub>)<sub>2</sub>Mo(CO)<sub>2</sub>. The envelope-shaped five-membered ring in (C<sub>11</sub>H<sub>14</sub>)Fe(CO)<sub>3</sub> has the 'flap' in a *syn* relationship with respect to the four-membered ring. *A priori*, there is no obvious reason for the *syn* relationship being preferred over the *anti* relationship. Hence we were curious about which conformation would be found in (C<sub>11</sub>H<sub>14</sub>)<sub>2</sub>Mo(CO)<sub>2</sub>.

### Experimental

The title compound was prepared by Dr G. Deganello as previously described (Cotton & Deganello, 1973) and crystallized from hexane by Mr J. M. Troup. A yellow crystal having approximate dimensions of 0.13 × 0.26 × 0.62 mm was mounted with its long dimension roughly parallel to the *φ* axis of the goniometer. The coilimator (1.5 mm) and beam were of sufficient size to accommodate the large crystal.

Preliminary examination and data collection were performed with Mo *Kα* radiation and a Syntex *P*I diffractometer equipped with a graphite-crystal incident-beam monochromator. Cell constants and an orientation matrix for data collection were obtained from least-squares refinement of the setting angles of 15 computer-centered reflections in the range 14° < 2*θ* < 26° [*λ*(Mo *Kα*) = 0.71068 Å]. Cell parameters and other crystal data are summarized in Table 1. The crystal mosaicity was determined from *ω* scans of several intense reflections; the width at half-height was 0.20°.

The data were collected at a temperature of 15° by

\* Part I: Cotton, Day, Frenz, Hardcastle & Troup (1973).

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Table 1. *Crystal data*

Bis(tricyclo[6.3.0.0 <sup>2,7</sup> ]undeca-3,5-diene)dicarbonylmolybdenum		
C <sub>24</sub> H <sub>28</sub> MoO <sub>2</sub>	F.W. 444.43	<i>F</i> (000) = 230
Monoclinic, space group <i>P</i> 2 <sub>1</sub> / <i>c</i>		
Mo <i>K</i> α radiation		
λ = 0.71068 Å		
<i>a</i> = 11.608 (2) Å	<i>V</i> = 1965.0 (6) Å <sup>3</sup>	
<i>b</i> = 12.533 (3)	<i>Z</i> = 4	
<i>c</i> = 13.759 (2)	<i>D</i> <sub>x</sub> = 1.50 g cm <sup>-3</sup>	
β = 101.01 (1)°	μ = 6.78 cm <sup>-1</sup>	

the  $\theta$ - $2\theta$  scan technique with a variable scan rate ranging from 2–24° min.<sup>-1</sup> The scan range extended from  $2\theta(\text{Mo } K\alpha_1) - 0.9^\circ$  to  $2\theta(\text{Mo } K\alpha_2) + 0.9^\circ$ . Of the 4734 independent reflections collected in the range  $0^\circ < 2\theta \leq 55^\circ$  only the 3595 reflections with  $F_o^2 > 3\sigma(F_o^2)$  were included in subsequent least-squares refinements. Two other reflections (012 and 102) had intensities exceeding the detector's ability to measure them accurately; hence this pair of reflections was not included in refinements.

During data collection four reference reflections measured after every 100 reflections showed no significant change in intensity. No absorption correction was applied; from trial calculations on a variety of

reflections, transmission factors varied only from 0.87 to 0.92. An extinction correction was applied; the final value for the coefficient is  $5.5(4) \times 10^{-7}$ . This low value indicates that the correction was probably unnecessary, as was also shown by an examination of the low-angle reflections. Additional details on data collection have been given previously (Cotton, Frenz, Deganello & Shaver, 1973).

### Structure determination and refinement

The position of the Mo atom was readily located from a three-dimensional Patterson map. One cycle of least-squares refinement of the Mo coordinates plus a scale factor gave  $R_1 = \sum ||F_o| - |F_c|| / \sum |F_o| = 0.409$ . This was followed by a difference Fourier synthesis which revealed the positions of all other non-hydrogen atoms. Three cycles of isotropic full-matrix least-squares refinement reduced  $R_1$  to 0.061, using only the 1790 reflections in the 0–40°  $2\theta$  shell. This was followed by one cycle of anisotropic refinement. The positions of the 28 H atoms were calculated assuming a C–H distance of 0.95 Å. The H atom positions and isotropic temperature parameters of 5.0 Å<sup>2</sup> were included in the structure factors but were not refined in two final cycles of anisotropic refinement (245 variables) using

Table 2. *Positional and anisotropic thermal ( $\times 10^4$ ) parameters and their estimated standard deviations*

The atomic numbering scheme used here follows the chemical numbering scheme and is depicted in Table 4. Numbers in parentheses following the parameters in this and other tables are estimated standard deviations in the least significant digits. The form of the thermal ellipsoid is  $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)]$ .

	<i>x</i>	<i>y</i>	<i>z</i>	β <sub>11</sub>	β <sub>22</sub>	β <sub>33</sub>	β <sub>12</sub>	β <sub>13</sub>	β <sub>23</sub>	
Mo	-0.15568 (2)	0.17025 (2)	0.11113 (1)	46.8 (2)	33.8 (2)	27.2 (1)	0.4 (1)	6.2 (1)	1.8 (1)	
O(1)	-0.3927 (2)	0.2127 (2)	0.5274 (2)	77 (2)	96 (2)	109 (2)	-36 (2)	12 (2)	-35 (2)	
O(2)	-0.1158 (2)	0.4146 (2)	0.4054 (2)	107 (2)	77 (2)	48 (1)	17 (2)	28 (1)	21 (1)	
C(12)	-0.3079 (3)	0.2420 (2)	0.0580 (2)	66 (2)	53 (2)	51 (2)	3 (2)	15 (2)	12 (1)	
C(13)	-0.1295 (2)	0.1175 (2)	-0.0196 (2)	54 (2)	44 (2)	44 (2)	-9 (2)	9 (1)	-3 (1)	
Ligand 1										
C(1)	0.1350 (2)	0.3761 (2)	0.0298 (2)	63 (2)	37 (2)	40 (2)	-6 (2)	14 (1)	-2 (1)	
C(2)	0.0312 (2)	0.2957 (2)	0.0068 (2)	58 (2)	39 (2)	28 (1)	-5 (1)	7 (1)	-4 (1)	
C(3)	-0.0782 (2)	0.3210 (2)	0.0465 (2)	55 (2)	36 (2)	38 (1)	0 (1)	7 (1)	3 (1)	
C(4)	-0.0720 (3)	0.3301 (2)	0.1505 (2)	70 (2)	40 (2)	44 (2)	-11 (2)	20 (2)	-10 (1)	
C(5)	-0.0003 (2)	0.2524 (2)	0.2072 (2)	69 (2)	60 (2)	26 (1)	-21 (2)	5 (1)	-2 (1)	
C(6)	0.0529 (2)	0.1763 (2)	0.1560 (2)	55 (2)	45 (2)	39 (1)	-8 (2)	2 (1)	11 (1)	
C(7)	0.1053 (2)	0.2090 (2)	0.0687 (2)	50 (2)	38 (2)	38 (1)	-2 (1)	6 (1)	1 (1)	
C(8)	0.2113 (2)	0.2882 (2)	0.0927 (2)	62 (2)	49 (2)	42 (2)	-12 (2)	1 (1)	4 (1)	
C(9)	0.3118 (3)	0.2655 (3)	0.0389 (3)	51 (2)	57 (2)	81 (2)	-7 (2)	14 (2)	4 (2)	
C(10)	0.2643 (3)	0.2977 (3)	-0.0678 (3)	78 (3)	58 (2)	68 (2)	-15 (2)	36 (2)	-11 (2)	
C(11)	0.1962 (3)	0.3984 (2)	-0.0569 (2)	77 (2)	51 (2)	51 (2)	-17 (2)	21 (2)	5 (1)	
Ligand 2										
C(1)	-0.4272 (2)	-0.0522 (2)	0.2006 (2)	67 (2)	40 (2)	49 (2)	-1 (2)	16 (2)	3 (1)	
C(2)	-0.3891 (2)	0.0403 (2)	0.1385 (2)	51 (2)	41 (2)	36 (1)	-2 (1)	4 (1)	1 (1)	
C(3)	-0.2778 (2)	0.0215 (2)	0.0989 (2)	64 (2)	40 (2)	39 (1)	-4 (2)	16 (1)	0 (1)	
C(4)	-0.1708 (2)	0.0032 (2)	0.1652 (2)	63 (2)	37 (2)	57 (2)	7 (2)	20 (2)	12 (1)	
C(5)	-0.1511 (2)	0.0708 (2)	0.2495 (2)	57 (2)	57 (2)	35 (1)	-9 (2)	2 (1)	16 (1)	
C(6)	-0.2383 (3)	0.1450 (2)	0.2566 (2)	71 (2)	45 (2)	36 (1)	-16 (2)	14 (1)	-4 (1)	
C(7)	-0.3652 (2)	0.1120 (2)	0.2306 (2)	58 (2)	39 (2)	42 (2)	5 (1)	15 (1)	-1 (1)	
C(8)	-0.4001 (2)	0.0185 (2)	0.2949 (2)	52 (2)	60 (2)	43 (2)	-1 (2)	10 (1)	7 (1)	
C(9)	-0.5150 (3)	0.0317 (3)	0.3314 (2)	71 (3)	78 (3)	60 (2)	-4 (2)	27 (2)	1 (2)	
C(10)	-0.6090 (3)	0.0138 (3)	0.2401 (3)	55 (2)	98 (3)	82 (3)	0 (2)	19 (2)	17 (2)	
C(11)	-0.5602 (3)	-0.0747 (3)	0.1852 (3)	77 (3)	74 (3)	65 (2)	-28 (2)	14 (2)	5 (2)	

all 3595 reflections. The final agreement values are  $R_1=0.030$  and  $R_2=[\sum w(|F_o|-|F_c|)^2/\sum wF_o^2]^{1/2}=0.040$ . In the last cycle the largest parameter shift was 0.8 times its standard deviation. The error in an observation of unit weight was 1.39 and plots of  $\sum w(|F_o|-|F_c|)^2$  versus  $|F_o|$  as a function of the parameter  $p$  (Cotton, Frenz, Deganello & Shaver, 1973) showed that the original value of 0.04 was the most appropriate one for the calculation of  $\sigma(F_o^2)$ . Comparisons of  $\sum w(|F_o|-|F_c|)^2$  as a function of various classes of indices,  $\lambda^{-1} \sin \theta$ , and reflection number did not show any unusual trends. The two highest peaks ( $0.6 \text{ e } \text{\AA}^{-3}$ ) in the final difference Fourier map were symmetrically located  $0.8 \text{ \AA}$  from the Mo atom. The third highest peak had a height of  $0.4 \text{ e } \text{\AA}^{-3}$  compared to  $0.8 \text{ e } \text{\AA}^{-3}$  for H atoms in a previous map.

The function minimized in least-squares refinements was  $\sum w(|F_o|-|F_c|)^2$ , where  $|F_o|$  and  $|F_c|$  are the observed and calculated structure factor amplitudes and the weight  $w$  is defined by  $4F_o^2/\sigma^2(F_o^2)$ . Scattering factors were taken from Cromer & Waber (1974); anomalous dispersion factors for the Mo atom were those of Cromer & Liberman (1970).

Positional and anisotropic thermal parameters for the non-hydrogen atoms are tabulated in Table 2.\*

### Results

As anticipated, the structure of  $(C_{11}H_{14})_2Mo(CO)_2$  very closely parallels that of the previously studied compound,  $(C_{11}H_{14})Fe(CO)_3$ . As illustrated in Fig. 1, each  $C_{11}H_{14}$  ring system is bound to the Mo atom through the diene portion of the boat-shaped cyclohexadiene ring. Fused to the six-membered ring is an essentially planar cyclobutane ring which, in turn, is fused to an envelope-shaped cyclopentane ring.

\* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30392 (22 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

The present structure can be described in terms of the  $(C_{11}H_{14})Fe(CO)_3$  structure as a simple replacement of one of the carbonyl groups, directed away from the ring system, by a second  $C_{11}H_{14}$  ring system. The virtual mirror plane of the second ring system is rotated *ca.*  $90^\circ$  from that of the first. Thus, the C(12)–O(1) carbonyl group extends over the 2C(2)–2C(7) bond while the C(13)–O(2) carbonyl group extends over the 1C(2)–1C(7) bond. The molecule possesses approximate  $C_2$  symmetry with the twofold axis passing through the Mo atom and midway between the carbonyl groups.

The molecules are well separated in the unit cell (Fig. 2). There are no intermolecular contacts less than  $3.5 \text{ \AA}$  between non-hydrogen atoms.

Table 3 lists bond distances and bond angles in the Mo coordination sphere. All other important bond distances, bond angles and dihedral angles are tabulated in Table 4 alongside the analogous parameters for  $(C_{11}H_{14})Fe(CO)_3$ .

### Discussion

We have previously discussed at length the conformations of fused cycloalkanes in organometallic com-

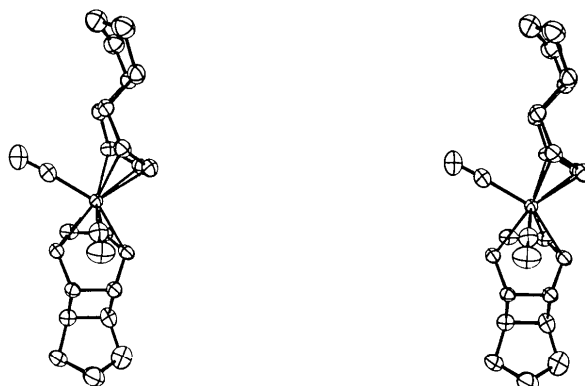


Fig. 1. Stereo view of the  $(C_{11}H_{14})_2Mo(CO)_2$  molecule, showing the 50% probability ellipsoids.

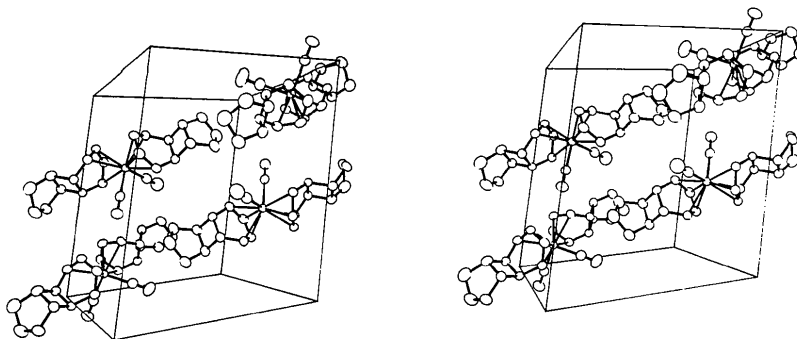


Fig. 2. Stereo packing diagram. The origin of the unit cell is in the lower right-hand corner at the back of the cell. The  $a$  axis extends from right to left, the  $b$  axis extends from back to front, and the  $c$  axis extends from bottom to top.

Table 3. Bond distances (Å) and bond angles (°) in the molybdenum coordination sphere

	Ligand 1	Ligand 2
Mo—C(3)	2.336 (3)	2.325 (3)
Mo—C(4)	2.244 (3)	2.236 (3)
Mo—C(5)	2.266 (3)	2.264 (3)
Mo—C(6)	2.380 (3)	2.396 (3)
C(12)—Mo—C(3)	82.8 (1)	81.4 (1)
C(12)—Mo—C(4)	89.9 (1)	115.0 (1)
C(12)—Mo—C(5)	124.0 (1)	115.7 (1)
C(12)—Mo—C(6)	150.1 (1)	84.1 (1)
C(13)—Mo—C(3)	77.8 (1)	82.2 (1)
C(13)—Mo—C(4)	112.3 (1)	91.4 (1)
C(13)—Mo—C(5)	115.8 (1)	126.3 (1)
C(13)—Mo—C(6)	85.5 (1)	149.6 (1)
C(3)—Mo—C(4)	36.1 (1)	36.0 (1)
C(3)—Mo—C(5)	62.3 (1)	62.4 (1)
C(3)—Mo—C(6)	68.1 (1)	67.7 (1)
C(4)—Mo—C(5)	36.5 (1)	36.7 (1)
C(4)—Mo—C(6)	62.5 (1)	62.0 (1)
C(5)—Mo—C(6)	34.8 (1)	34.6 (1)

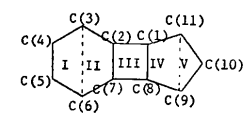
1C(3)—Mo—2C(3)	153.1 (1)	1C(5)—Mo—2C(3)	144.1 (1)
1C(3)—Mo—2C(4)	160.2 (1)	1C(5)—Mo—2C(4)	109.5 (1)
1C(3)—Mo—2C(5)	144.5 (1)	1C(5)—Mo—2C(5)	82.6 (1)
1C(3)—Mo—2C(6)	131.9 (1)	1C(5)—Mo—2C(6)	88.5 (1)
1C(4)—Mo—2C(3)	163.8 (1)	1C(6)—Mo—2C(3)	128.2 (1)
1C(4)—Mo—2C(4)	144.2 (1)	1C(6)—Mo—2C(4)	94.8 (1)
1C(4)—Mo—2C(5)	110.3 (1)	1C(6)—Mo—2C(5)	86.6 (1)
1C(4)—Mo—2C(6)	98.1 (1)	1C(6)—Mo—2C(6)	109.7 (1)
Mo—C(12)	1.991 (3)	C(12)—O(1)	1.142 (4)
Mo—C(13)	1.991 (3)	C(13)—O(2)	1.144 (3)
Mo—C(12)—O(1)	177.0 (3)	C(12)—Mo—C(13)	95.5 (1)
Mo—C(13)—O(2)	178.7 (2)		

plexes (Cotton & Frenz, 1974). A premise in these discussions was that the forces responsible for the conformations were intramolecular in nature and that intermolecular forces had an inconsequential effect on the conformation. The present structure confirms this premise.

Corresponding C—C bond lengths and C—C—C bond angles are statistically equivalent in the three crystal-

lographically independent  $C_{11}H_{14}$  ring systems (Table 4). This equivalence includes mirror symmetry across the ligand. A possible exception is the diene portion of the present structure. The Mo atom leans toward one side of the diene for each of its ligands. Thus the Mo—C(3) and Mo—C(4) bonds are slightly shorter than the respective Mo—C(5) and Mo—C(6) bonds. The C(3)—C(4) bond is longer than the C(5)—C(6) bond and the C(3)—Mo—C(4) bond angle is correspondingly larger than the C(5)—Mo—C(6) bond angle. It is likely that these perturbations are due to a steric interaction between the two bulky ring systems.

The similarity of the three determinations of the  $C_{11}H_{14}$  ligand extends to the dihedral angles between the various planes (Table 4). Interestingly, the values obtained for the  $(C_{11}H_{14})Fe(CO)_3$  structure are, in each

Table 4. Comparison of bond lengths (Å), bond angles (°) and dihedral angles in the  $C_{11}H_{14}$  ring system of  $(C_{11}H_{14})_2Mo(CO)_2$  and  $(C_{11}H_{14})Fe(CO)_3$ 


	$(C_{11}H_{14})_2Mo(CO)_2$	$(C_{11}H_{14})Fe(CO)_3$	Weighted average*
C(4)—C(5)	1.413 (4)	1.417 (4)	1.411 (4)
C(3)—C(4)	1.422 (4)	1.410 (4)	1.417 (3)
C(5)—C(6)	1.396 (4)	1.390 (4)	1.412 (3)
C(2)—C(3)	1.507 (4)	1.511 (4)	1.500 (3)
C(6)—C(7)	1.501 (4)	1.504 (4)	1.504 (3)
C(2)—C(7)	1.536 (4)	1.533 (4)	1.545 (3)
C(1)—C(2)	1.554 (4)	1.552 (4)	1.563 (3)
C(7)—C(8)	1.564 (4)	1.565 (4)	1.564 (3)
C(1)—C(8)	1.565 (4)	1.550 (4)	1.562 (3)
C(1)—C(11)	1.524 (4)	1.542 (4)	1.525 (3)
C(8)—C(9)	1.521 (4)	1.519 (4)	1.522 (3)
C(10)—C(11)	1.509 (4)	1.510 (5)	1.515 (4)
C(9)—C(10)	1.519 (5)	1.513 (5)	1.525 (4)

Table 4 (cont.)

Bond angle	$(C_{11}H_{14})_2Mo(CO)_2$		$(C_{11}H_{14})Fe(CO)_3$	Average
	Ligand 1	Ligand 2		
C(3)—C(4)—C(5)	114.3 (2)	114.6 (2)	115.2 (2)	115.7 (5)
C(4)—C(5)—C(6)	117.5 (2)	116.7 (2)	115.9 (2)	
C(2)—C(3)—C(4)	119.7 (2)	120.0 (2)	120.3 (2)	120.0 (2)
C(5)—C(6)—C(7)	120.1 (2)	119.5 (2)	120.5 (2)	
C(3)—C(2)—C(7)	111.6 (2)	110.9 (2)	111.3 (2)	111.2 (3)
C(2)—C(7)—C(7)	111.5 (2)	111.8 (2)	110.0 (2)	
C(1)—C(2)—C(3)	117.6 (2)	115.7 (2)	115.9 (2)	115.9 (4)
C(6)—C(7)—C(8)	115.2 (2)	114.7 (2)	116.1 (2)	
C(1)—C(2)—C(7)	90.9 (2)	90.4 (2)	90.3 (2)	90.4 (1)
C(2)—C(7)—C(8)	90.2 (2)	90.2 (2)	90.3 (2)	
C(2)—C(1)—C(8)	89.5 (2)	90.0 (2)	89.7 (2)	89.6 (1)
C(1)—C(8)—C(7)	89.4 (2)	89.4 (2)	89.6 (2)	
C(2)—C(1)—C(11)	114.7 (2)	116.3 (2)	115.2 (2)	115.4 (3)
C(7)—C(8)—C(9)	115.0 (2)	116.2 (2)	115.1 (2)	
C(8)—C(1)—C(11)	105.2 (2)	104.9 (2)	106.0 (2)	105.6 (3)
C(1)—C(8)—C(9)	105.5 (2)	106.7 (2)	105.4 (2)	
C(1)—C(11)—C(10)	104.8 (2)	105.0 (3)	104.4 (2)	104.7 (1)
C(8)—C(9)—C(10)	104.4 (2)	104.4 (3)	104.9 (2)	
C(9)—C(10)—C(11)	103.0 (3)	104.0 (3)	103.5 (2)	103.5 (2)

Table 4 (cont.)

Dihedral angle	$(C_{11}H_{14})_2Mo(Co)_2$		$(C_{11}H_{14})Fe(Co)_3$	Average
	Ligand 1	Ligand 2		
I-II	38.0	39.0	38.0	38.3 (3)
I-III	80.8	78.4	80.0	79.7 (7)
I-IV	34.9	38.9	36.1	36.6 (12)
I-V	5.2	1.2	2.3	2.9 (12)
II-III	118.8	117.3	118.0	118.0 (4)
II-IV	3.3	1.8	2.2	2.4 (4)
II-V	43.2	37.8	40.3	40.4 (16)
III-IV	115.7	117.2	116.0	116.3 (5)
III-V	75.7	79.5	77.7	77.6 (11)
IV-V	40.0	37.7	38.3	38.7 (7)

\* The weighted average is defined by  $\bar{d} = \sum(x_i/\sigma_i^2)/\sum(1/\sigma_i^2)$ . The standard deviation of the average is calculated using the following formula:  $\{\sum[(\bar{d} - x_i)/\sigma_i]^2/(N-1)\sum(1/\sigma_i^2)\}$ .

case, intermediate between the two respective values for the present structure. Of particular relevance with respect to the previous discussion of the conformation of four-membered rings is a comparison of the dihedral angles within the fused cyclobutane rings. In the  $(C_{11}H_{14})Fe(CO)_3$  structure this angle is  $1.3(3)^\circ$ , while the angle is  $0.2(3)^\circ$  for ligand 1 and  $2.1(3)^\circ$  for ligand 2 in  $(C_{11}H_{14})_2Mo(CO)_2$ . The average of these angles is  $\bar{d} = 1.2(5)^\circ$ . The internal e.s.d. is  $\sigma_{INT} = [1/\sum(1/\sigma_i^2)]^{1/2} = 0.17$  compared to the external e.s.d. of  $\sigma_{EXT} = [1/(N-1)\sum(\bar{d} - d_i)^2]^{1/2} = 0.55$ . This difference in the two e.s.d.'s suggests either that the three dihedral angles are not of the same population or that the individual e.s.d.'s are underestimated. Inter-experiment agreement studies (Hamilton & Abrahams, 1970) suggest that individual e.s.d.'s are routinely underestimated. If a factor of 2-3 is applied to the e.s.d.'s, then the three dihedral angles are statistically equivalent. This is satisfying from the chemical point of view since it is difficult to envision any reason why these angles should be different. In

any event, it can be concluded that, on the basis of this study, crystal packing forces have insubstantial effects on the conformations of the fused ring systems. In addition, statistically meaningful discussions of the dihedral angles in these related compounds requires that a factor of 2 or 3 be applied to the e.s.d.'s. If this procedure is followed it is clear that the arguments put forth in the previous paper still are justified.

One additional feature of the present structure worthy of note is the conformation of the fused cyclopentane ring. In the previous structure of  $(C_{11}H_{14})Fe(CO)_3$  as well as in both  $C_{11}H_{14}$  ligands in  $(C_{11}H_{14})_2Mo(CO)_2$ , the ring maintains an envelope conformation with the 'flap' lifted toward the four-membered ring. An examination of molecular models does not suggest any reason for this conformation being preferred over the anti relationship. We offer no explanation for this structural curiosity.

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