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## Structure of a Novel C<sub>22</sub>H<sub>24</sub> Cage Dimer

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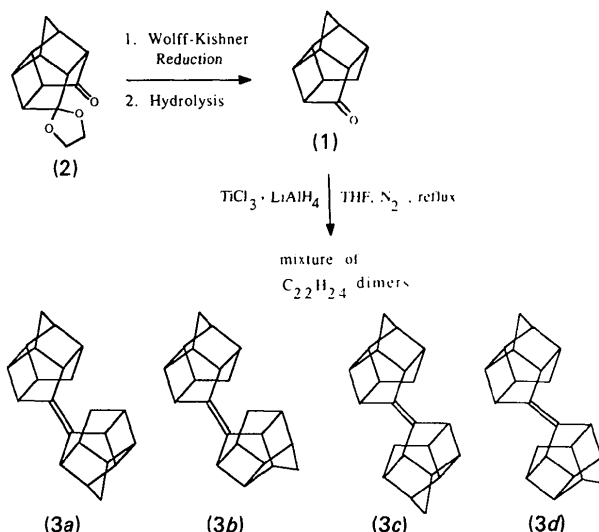
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**Abstract.** 8,11'-Bipentacyclo[5.4.0.0<sup>2,6</sup>.0<sup>3,10</sup>.0<sup>5,9</sup>]-undecanylidene, C<sub>22</sub>H<sub>24</sub>, *M<sub>r</sub>* = 288.43, triclinic, *P* $\bar{1}$ , *a* = 6.613 (2), *b* = 10.809 (3), *c* = 10.883 (2) Å,  $\alpha$  = 97.52 (2),  $\beta$  = 99.85 (1),  $\gamma$  = 99.32 (2)°, *V* = 746.2 (2) Å<sup>3</sup>, *Z* = 2, *D<sub>x</sub>* = 1.284 Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha)$  = 0.71073 Å,  $\mu$  = 0.07 mm<sup>-1</sup>, *F*(000) = 312, *T* = 295 K, final *R* = 0.048, *wR* = 0.048 for 1118 observed reflections. The molecule, which has an unusually high density for a hydrocarbon, consists of two cage moieties which are related by an approximate twofold axis along the C(11)–C(11') double bond. There are no intermolecular approaches less than van der Waals separations; the high density is probably due to compression of the cage C atoms ensuing from the small bond angles. For example, the internal ring angles at C(11) and C(11'), both *sp*<sup>2</sup> C atoms, are only 102.6 (2) and 102.2 (2)°, respectively.

**Introduction.** As part of a program that is involved in the synthesis and chemistry of energetic polycyclic 'cage' compounds (Marchand, 1987), the titanium-induced dicarbonyl-coupling reaction of pentacyclo[5.4.0.0<sup>2,6</sup>.0<sup>3,10</sup>.0<sup>5,9</sup>]undecan-8-one (1) has been studied. Compound (1) was synthesized *via* Wolff–Kishner reduction of the pentacyclo[5.4.0.0<sup>2,6</sup>.0<sup>3,10</sup>.0<sup>5,9</sup>]undecane-8,11-dione monoethylene ketal (2) (Eaton, Cassar, Hudson & Hwang, 1976) followed by hydrolysis of the resulting product. Reaction of (1) with 'McMurry's reagent' (*i.e.* TiCl<sub>3</sub>–LiAlH<sub>4</sub>) (McMurry, 1983) potentially could result in the formation of four dimers [*i.e.* (3a)–(3d)], each of which possesses a twofold symmetry element. The product that was

obtained by refluxing a mixture of (1) and TiCl<sub>3</sub>–LiAlH<sub>4</sub> in tetrahydrofuran under nitrogen for 72 h was purified *via* column chromatography (silica-gel stationary phase, hexane eluent). Careful fractional recrystallization of the purified product from hexane afforded a single isomer, m.p. 473–474 K. The results of a single-crystal X-ray structural analysis on the isomer thereby obtained revealed that this material possesses structure (3a) (see below).



**Experimental.** Clear colorless crystal, 0.18 × 0.35 × 0.42 mm, automated Nicolet R3m diffractometer with incident-beam graphite monochromator, 25 centered

Table 1. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ )Equivalent isotropic  $U$  defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor.

	$x$	$y$	$z$	$U_{eq}$
C(1)	2240 (4)	10392 (3)	8683 (3)	39 (1)
C(2)	2574 (4)	9073 (3)	9004 (3)	39 (1)
C(3)	3822 (5)	8523 (3)	8064 (3)	40 (1)
C(4)	2542 (5)	7210 (3)	7527 (3)	48 (1)
C(5)	517 (5)	7671 (3)	7149 (3)	42 (1)
C(6)	279 (5)	8477 (3)	8369 (3)	43 (1)
C(7)	-85 (5)	9794 (3)	8034 (3)	42 (1)
C(8)	-374 (5)	9633 (3)	6606 (3)	45 (1)
C(9)	1077 (4)	8710 (3)	6357 (3)	36 (1)
C(10)	3401 (4)	9326 (2)	7006 (3)	32 (1)
C(11)	3542 (4)	10635 (2)	7714 (2)	31 (1)
C(1')	6010 (4)	11980 (3)	6618 (3)	39 (1)
C(2')	7894 (5)	13009 (3)	7360 (3)	48 (1)
C(3')	7392 (5)	13454 (3)	8642 (3)	52 (1)
C(4')	7764 (6)	14897 (3)	8758 (3)	61 (1)
C(5')	6314 (5)	14873 (3)	7500 (3)	49 (1)
C(6')	7159 (5)	14006 (3)	6558 (3)	50 (1)
C(7')	5259 (5)	12984 (3)	5810 (3)	45 (1)
C(8')	3368 (5)	13419 (3)	6199 (3)	51 (1)
C(9')	4247 (5)	14027 (3)	7559 (3)	46 (1)
C(10')	4994 (5)	13020 (3)	8375 (3)	41 (1)
C(11')	4657 (4)	11727 (3)	7566 (2)	34 (1)

reflections with  $25 < 2\theta < 36^\circ$  used for determining lattice parameters;  $2\theta_{max} = 46^\circ$ , range of  $hkl$ :  $0 \leq h \leq 7$ ,  $-11 \leq k \leq 11$ ,  $-11 \leq l \leq 11$ . Standards 300, 050, 004, monitored every 100 reflections with random variation of 2.5% over data collection,  $\theta$ - $2\theta$  mode, scan width ( $2.2 + 42\theta_{\alpha 1\alpha 2}$ ), scan rate a function of count rate ( $30^\circ \text{ min}^{-1}$  minimum,  $60^\circ \text{ min}^{-1}$  maximum), 2190 reflections measured, 1600 unique ( $R_{int} = 0.022$ ), 1118 observed [ $F_o > 3\sigma(F_o)$ ]. Data corrected for Lorentz and polarization effects, but not for absorption.

Structure solved by direct methods. Full-matrix least-squares refinement, H atoms from difference maps,  $\sum w(|F_o| - |F_c|)^2$  minimized where  $w = 1/[\sigma^2(|F_o|) + g(|F_o|)^2]$  and  $g(F_o)^2$  is included to account for random instrumental error ( $g$  estimated to be 0.000225). Secondary-extinction parameter  $P = 0.0038(7)$  in  $F_c^* = F_c/[1.0 + 0.002(P)F_o^2/\sin 2\theta]^{0.25}$ . 272 parameters refined, atomic coordinates for all atoms, anisotropic temperature factors for non-H atoms, constant isotropic temperature factors (equal to final  $U_{eq}$  value of covalently bonded atom) for H atoms. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Final  $R = 0.048$ ,  $wR = 0.048$ , max.  $\Delta/\sigma = 0.010$ ,  $S = 1.43$ . Final difference Fourier excursions 0.16 and 0.16  $e \text{ \AA}^{-3}$ .† All calculations were performed using the *SHELXTL* system (Sheldrick, 1980).

† Lists of structure factors, H-atom coordinates and anisotropic thermal parameters for non-H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44963 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

**Discussion.** Atom numbering for Tables 1 and 2 follows that shown in Fig. 1. The two cage moieties of the molecule are related by an approximate twofold axis along the C(11)–C(11') bond. The four-membered rings are planar (av. absolute C–C–C–C torsion is

Table 2. Bond lengths ( $\text{\AA}$ ) and bond angles ( $^\circ$ )

C(1)–C(2)	1.552 (4)	C(1)–C(7)	1.571 (4)
C(1)–C(11)	1.494 (4)	C(2)–C(3)	1.539 (4)
C(2)–C(6)	1.551 (4)	C(3)–C(4)	1.516 (4)
C(3)–C(10)	1.544 (4)	C(4)–C(5)	1.514 (4)
C(5)–C(6)	1.536 (4)	C(5)–C(9)	1.540 (4)
C(6)–C(7)	1.560 (4)	C(7)–C(8)	1.516 (4)
C(8)–C(9)	1.522 (4)	C(9)–C(10)	1.574 (4)
C(10)–C(11)	1.501 (3)	C(11)–C(11')	1.331 (3)
C(1')–C(2')	1.554 (4)	C(1')–C(7')	1.573 (4)
C(1')–C(11')	1.500 (4)	C(2')–C(3')	1.524 (4)
C(2')–C(6')	1.559 (4)	C(3')–C(4')	1.523 (4)
C(3')–C(10')	1.545 (4)	C(4')–C(5')	1.525 (5)
C(5')–C(6')	1.534 (4)	C(5')–C(9')	1.533 (4)
C(6')–C(7')	1.558 (4)	C(7')–C(8')	1.511 (4)
C(8')–C(9')	1.513 (4)	C(9')–C(10')	1.580 (4)
C(10')–C(11')	1.513 (4)		
C(7)–C(1)–C(2)	89.5 (2)	C(11)–C(1)–C(2)	104.6 (2)
C(11)–C(1)–C(7)	110.8 (2)	C(3)–C(2)–C(1)	107.3 (2)
C(6)–C(2)–C(1)	90.8 (2)	C(6)–C(2)–C(3)	102.8 (2)
C(4)–C(3)–C(2)	103.6 (2)	C(10)–C(3)–C(2)	100.4 (2)
C(5)–C(4)–C(3)	105.1 (2)	C(5)–C(4)–C(3)	94.9 (2)
C(9)–C(5)–C(4)	103.8 (2)	C(9)–C(5)–C(4)	104.3 (2)
C(6)–C(5)–C(4)	103.8 (2)	C(5)–C(6)–C(2)	102.8 (2)
C(7)–C(6)–C(2)	90.0 (2)	C(7)–C(6)–C(5)	107.1 (2)
C(6)–C(7)–C(1)	89.7 (2)	C(8)–C(7)–C(1)	111.2 (2)
C(8)–C(7)–C(6)	104.9 (2)	C(9)–C(8)–C(7)	101.1 (2)
C(8)–C(9)–C(5)	103.8 (2)	C(10)–C(9)–C(5)	102.9 (2)
C(10)–C(9)–C(8)	110.4 (2)	C(9)–C(10)–C(3)	101.7 (2)
C(11)–C(10)–C(3)	103.7 (2)	C(11)–C(10)–C(9)	111.2 (2)
C(10)–C(11)–C(1)	102.6 (2)	C(11')–C(11)–C(1)	128.7 (2)
C(11')–C(11)–C(10)	128.6 (2)	C(7')–C(11')–C(2')	89.6 (2)
C(11')–C(11')–C(2')	104.6 (2)	C(11')–C(11')–C(7')	111.1 (2)
C(3')–C(2')–C(1')	107.3 (2)	C(6')–C(2')–C(1')	90.6 (2)
C(6')–C(2')–C(3')	102.6 (3)	C(4')–C(3')–C(2')	104.1 (3)
C(10')–C(3')–C(2')	101.3 (2)	C(10')–C(3')–C(4')	105.4 (3)
C(5')–C(4')–C(3')	93.8 (2)	C(6')–C(5')–C(4')	103.3 (3)
C(9')–C(5')–C(4')	105.5 (3)	C(9')–C(5')–C(6')	101.3 (2)
C(5')–C(6')–C(2')	102.6 (3)	C(7')–C(6')–C(2')	89.9 (2)
C(7')–C(6')–C(5')	106.7 (2)	C(6')–C(7')–C(1')	89.9 (2)
C(8')–C(7')–C(1')	111.2 (3)	C(8')–C(7')–C(6')	104.9 (2)
C(9')–C(8')–C(7')	101.2 (3)	C(8')–C(9')–C(5')	103.9 (3)
C(10')–C(9')–C(5')	102.6 (2)	C(10')–C(9')–C(8')	111.0 (2)
C(9')–C(10')–C(3')	101.7 (2)	C(11')–C(10')–C(3')	103.1 (2)
C(11')–C(10')–C(9')	110.8 (2)	C(1')–C(11')–C(11)	129.3 (2)
C(10')–C(11')–C(11)	128.4 (2)	C(10')–C(11')–C(1)	102.2 (2)

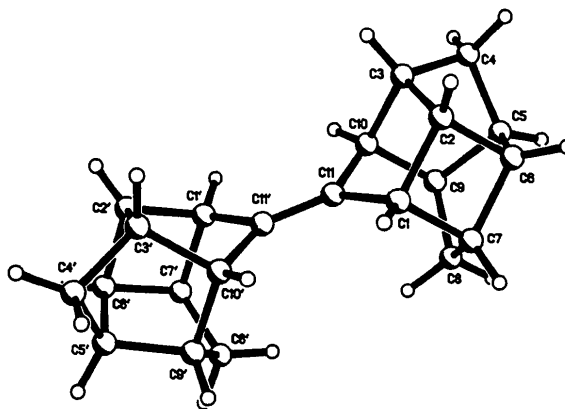


Fig. 1. Results of the X-ray study on (3a).

0.1°), all five-membered rings have an envelope conformation and the two six-membered rings are boats. The compound, which has an unusually high density for a hydrocarbon, has no intermolecular approaches less than van der Waals separations. The high density must be due to crowding within the molecule itself. There are two extremely small internal ring angles in the cage: 102.6 (2)° at the  $sp^2$  C(11) atom [102.2 (2)° at C(11')] and 94.9 (2)° at the  $sp^3$  C(4) atom [93.8 (2)° at C(4')], which lead to close intramolecular approaches of 2.34 Å for C(1)...C(10) [2.34 Å for C(1')...C(10')] and 2.33 Å for C(3)...C(5) [2.22 Å for C(3')...C(5')]. All the  $sp^3$ - $sp^3$  internal cage angles (excluding those involving the four-membered rings) are also smaller than normal [av. = 103.1 (2)° for 12 angles].

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## Structure of (*E*)-6-(3,4-Dimethoxyphenyl)-1-ethyl-4-mesitylimino-3-methyl-3,4-dihydro-2(1*H*)-pyrimidinone (FK664) Hemibenzene Solvate

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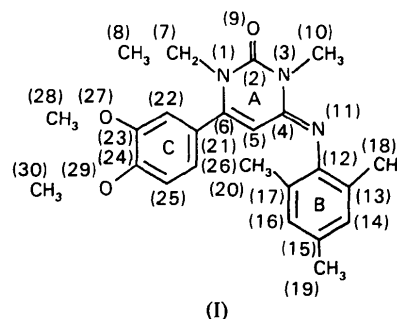
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**Abstract.**  $C_{24}H_{29}N_3O_3 \cdot \frac{1}{2}C_6H_6$ ,  $M_r = 446.57$ , monoclinic,  $P2_1/n$ ,  $a = 15.634$  (2),  $b = 6.857$  (1),  $c = 23.555$  (2) Å,  $\beta = 105.10$  (2)°,  $V = 2438.0$  (5) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.217$  Mg m<sup>-3</sup>, graphite-monochromated Cu  $K\alpha$  radiation,  $\lambda = 1.54178$  Å,  $\mu = 0.6024$  mm<sup>-1</sup>,  $F(000) = 956$ , room temperature, final  $R = 0.056$  for 3749 [ $F_o \geq 3\sigma(F_o)$ ] reflections. The 2-pyrimidone ring is planar and the endocyclic N(1) and N(3) atoms take the  $sp^2$  hybrid orbitals. The two phenyl planes of the N(11) and C(6) substituents twist 74.4 and 120.8° with respect to the 2-pyrimidone plane. Packing of the molecules is governed by normal van der Waals contacts.

**Introduction.** FK664 (I), a derivative of 2-pyrimidone, is an orally effective non-catechol and non-glycoside cardiostimulant agent. It has been developed by Fujisawa Pharmaceutical Co. Ltd as a potentially useful agent against heart failure. In spite of the effort to make clear the binding sites of *N*-methyl and *N*-ethyl groups at the 2-pyrimidone ring by various kinds of spectroscopic

measurements, they have remained ambiguous. In order to clarify the conformational features of FK664, we carried out X-ray crystallographic studies on a crystal of FK664 hemibenzene solvate.



**Experimental.** Colorless rod-like crystals were obtained from benzene solution of FK664 as a benzene solvate with dimensions 0.25 × 0.40 × 0.80 mm. Rigaku AFC-5 diffractometer, graphite-monochromated