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Structure of 1,1′:2,2′-Bis(oxybismethylene)ferrocene

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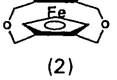
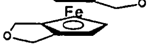
Abstract. $C_{14}H_{14}FeO_2$, $M_r = 270.1$, monoclinic, $P2_1/n$, $a = 12.884\,(6)$, $b = 12.012\,(6)$, $c = 14.442\,(7)$ Å, $\beta = 101.45\,(3)^\circ$, $V = 2191\,(2)$ Å^3, $Z = 8$, $D_x = 1.638$ g cm^{-3}, $\lambda(\text{Mo }K\alpha) = 0.71073$ Å, $\mu = 13.58$ cm^{-1}, $F(000) = 1120$, $T = 296$ K, $R_F = 4.75\%$ for 2654 reflections with $F_o > 5\sigma(F_o)$ and 308 parameters. The complex is a doubly bridged ferrocene which is the product of the acid-catalyzed double dehydration of 1,1′,2,2′-tetrakis(hydroxymethyl)ferrocene. The molecule crystallizes with the two oxybismethylene bridges in an *endo-exo* conformation. Comparisons made with related bridged ferrocenes suggest that the preference for *endo-exo* geometry derives from favorable dipole–dipole interaction between the ether oxygens.

Introduction. In the course of our efforts to construct cooperative ferrocene-dicoronands (Petter & Milberg, 1989; Petter, Milberg & Rao, 1990), we discovered that 1,1′,2,2′-tetrakis(hydroxymethyl)ferrocene [(1)] undergoes a remarkable acid-catalyzed tandem dehydration in aqueous solvents (Petter *et al.*, 1990). Our initial assignment of the product as (2) rested on mechanistic considerations and our interpretation of the electron impact mass spectrum, which exhibits a molecular ion ($m/z = 270$) which is also the base peak. In our experience this behavior is typical of heteroannularly bridged ferrocenes, whereas ferrocenes which lack such tethers readily fragment by separation of one of the cyclopentadienyl (Cp) rings. Though (3) might be expected to be less stable than (2) owing to angle strain, analogous structures have been reported (Slocum, Rockett & Hauser, 1965). NMR cannot resolve this lingering ambiguity because the two candidate structures have equivalent symmetry [assuming ready rotation about the central axis of the ferrocene in (3)]. Definitive assignment of (2) as the product of the tandem dehydration of (1) emerged from single-crystal X-ray analysis of the product.

(1)



(2)



(3)

Table 1. *Atomic coordinates* (× 10^4) *and equivalent isotropic displacement coefficients* (Å^2 × 10^3) *for* (2)

	x	y	z	U_{eq}*
Fe	6024 (1)	7064 (1)	9108 (1)	28 (1)
Fe′	654 (1)	7966 (1)	7766 (1)	28 (1)
C(1)	4794 (4)	8120 (5)	8922 (4)	32 (2)
C(2)	4635 (4)	7261 (5)	8224 (4)	32 (2)
C(3)	4631 (4)	6234 (5)	8721 (5)	39 (2)
C(4)	4801 (5)	6444 (5)	9701 (5)	41 (2)
C(5)	4903 (5)	7620 (5)	9830 (4)	33 (2)
C(6)	7080 (4)	8170 (5)	8821 (4)	31 (2)
C(7)	6902 (4)	7293 (5)	8142 (4)	31 (2)
C(8)	7215 (5)	6295 (5)	8622 (5)	39 (2)
C(9)	7561 (5)	6516 (6)	9596 (5)	45 (3)
C(10)	7391 (5)	7483 (6)	9722 (4)	39 (2)
C(11)	4942 (5)	9347 (5)	8752 (5)	40 (2)
C(12)	6788 (5)	9375 (5)	8643 (5)	40 (2)
C(13)	4571 (5)	7394 (6)	7189 (4)	43 (2)
C(14)	6417 (5)	7414 (6)	7108 (4)	36 (2)
O(1)	5986 (3)	9767 (3)	9128 (3)	44 (2)
O(2)	5424 (3)	7991 (4)	6928 (3)	42 (1)
C(1′)	−331 (4)	7049 (5)	8362 (4)	30 (2)
C(2′)	−225 (4)	8149 (5)	8751 (4)	33 (2)
C(3′)	−585 (4)	8900 (6)	8001 (4)	38 (2)
C(4′)	−907 (4)	8281 (6)	7154 (4)	38 (2)
C(5′)	−738 (4)	7151 (5)	7380 (4)	34 (2)
C(6′)	1947 (4)	7068 (5)	8243 (4)	34 (2)
C(7′)	2045 (4)	8166 (5)	8641 (4)	35 (2)
C(8′)	1979 (4)	8926 (6)	7887 (4)	39 (2)
C(9′)	1827 (5)	8323 (6)	7028 (5)	40 (2)
C(10′)	1824 (4)	7188 (5)	7245 (4)	37 (2)
C(11′)	256 (5)	8469 (6)	9752 (4)	43 (2)
C(12′)	2107 (5)	8452 (6)	9676 (5)	47 (2)
C(13′)	41 (5)	5991 (5)	8881 (5)	43 (2)
C(14′)	1873 (5)	5987 (5)	8764 (5)	42 (2)
O(1′)	1222 (3)	9093 (4)	9844 (3)	51 (2)
O(2′)	1116 (3)	5983 (4)	9368 (3)	51 (2)

* Equivalent isotropic U defined as one third of the trace of the orthogonolized U_{ij} tensor.

Experimental. Yellow–orange crystals (0.20 × 0.25 × 0.35 mm) were obtained by recrystallization from 4:1 hexane–ethyl acetate. Nicolet *R3m/E* diffractometer, Wyckoff scans, lattice parameters from least-squares fit of 25 reflections, $18 \le 2\theta \le 25^\circ$, empirical absorption correction (*XEMP*) ($\mu = 13.58$ cm^{-1}, $T_{\max} = 0.734$, $T_{\min} = 0.692$, $\mu r = 0.18$), $2\theta_{\max} = 50^\circ$ ($h = \pm 16$, $k = +15$, $l = +18$), standard reflections (701, 360, 428), $\le 1\%$ decay, 5514 reflections collected, 5041 independent, $R_{\text{int}} = 3.46\%$, 2654 reflections with $F_o > 5\sigma(F_o)$, direct methods solution located two Fe atoms, remaining non-H atoms located from subsequent difference Fourier syntheses, refinement on F for 308 parameters, all non-H atoms aniso-

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Table 2. *Bond lengths* (Å) *and angles* (°) *for* (2)

Fe—C(1)	2.006 (5)	Fe—C(2)	1.995 (5)
Fe—C(3)	2.032 (6)	Fe—C(4)	2.074 (7)
Fe—C(5)	2.053 (6)	Fe—C(6)	2.003 (6)
Fe—C(7)	1.982 (6)	Fe—C(8)	2.032 (7)
Fe—C(9)	2.073 (6)	Fe—C(10)	2.060 (6)
Fe′—C(1′)	1.999 (6)	Fe′—C(2′)	1.998 (6)
Fe′—C(3′)	2.033 (6)	Fe′—C(4′)	2.064 (5)
Fe′—C(5′)	2.022 (5)	Fe′—C(6′)	1.990 (6)
Fe′—C(7′)	1.992 (5)	Fe′—C(8′)	2.039 (6)
Fe′—C(9′)	2.061 (7)	Fe′—C(10′)	2.040 (6)
C(1)—C(2)	1.428 (8)	C(1)—C(5)	1.424 (8)
C(1)—C(11)	1.513 (8)	C(2)—C(3)	1.427 (9)
C(2)—C(13)	1.490 (8)	C(3)—C(4)	1.411 (9)
C(4)—C(5)	1.427 (9)	C(6)—C(7)	1.425 (8)
C(6)—C(10)	1.429 (8)	C(6)—C(12)	1.505 (8)
C(7)—C(8)	1.403 (9)	C(7)—C(14)	1.507 (7)
C(8)—C(9)	1.414 (9)	C(9)—C(10)	1.419 (10)
C(11)—O(1)	1.439 (7)	C(12)—O(1)	1.437 (8)
C(13)—O(2)	1.425 (8)	C(14)—O(2)	1.432 (7)
C(1′)—C(2′)	1.431 (8)	C(1′)—C(5′)	1.416 (8)
C(1′)—C(13′)	1.505 (9)	C(2′)—C(3′)	1.415 (8)
C(2′)—C(11′)	1.506 (8)	C(3′)—C(4′)	1.422 (9)
C(4′)—C(5′)	1.403 (9)	C(6′)—C(7′)	1.435 (9)
C(6′)—C(10′)	1.425 (9)	C(6′)—C(14′)	1.513 (9)
C(7′)—C(8′)	1.411 (9)	C(7′)—C(12′)	1.521 (9)
C(8′)—C(9′)	1.415 (9)	C(9′)—C(10′)	1.399 (9)
C(11′)—O(1′)	1.437 (8)	C(12′)—O(1′)	1.436 (8)
C(13′)—O(2′)	1.424 (7)	C(14′)—O(2′)	1.432 (9)
C(1)—Fe—C(2)	41.8 (2)	C(1)—Fe—C(3)	69.0 (2)
C(2)—Fe—C(3)	41.5 (2)	C(1)—Fe—C(4)	68.2 (2)
C(2)—Fe—C(4)	69.4 (2)	C(3)—Fe—C(4)	40.2 (3)
C(1)—Fe—C(5)	41.1 (2)	C(2)—Fe—C(5)	69.8 (2)
C(3)—Fe—C(5)	68.1 (2)	C(4)—Fe—C(5)	40.5 (2)
C(1)—Fe—C(6)	96.0 (2)	C(2)—Fe—C(6)	110.4 (2)
C(3)—Fe—C(6)	150.2 (2)	C(4)—Fe—C(6)	157.9 (2)
C(5)—Fe—C(6)	117.7 (2)	C(1)—Fe—C(7)	111.4 (2)
C(2)—Fe—C97)	95.6 (2)	C(3)—Fe—C(7)	118.1 (2)
C(4)—Fe—C(7)	157.9 (2)	C(5)—Fe—C(7)	150.4 (2)
C(6)—Fe—C(7)	41.9 (2)	C(1)—Fe—C(8)	150.7 (3)
C(2)—Fe—C(8)	118.4 (2)	C(3)—Fe—C(8)	111.6 (3)
C(4)—Fe—C(8)	131.8 (3)	C(5)—Fe—C(8)	168.2 (3)
C(6)—Fe—C(8)	68.8 (2)	C(7)—Fe—C(8)	40.9 (2)
C(1)—Fe—C(9)	157.3 (3)	C(2)—Fe—C(9)	158.4 (3)
C(3)—Fe—C(9)	132.0 (3)	C(4)—Fe—C(9)	119.9 (3)
C(5)—Fe—C(9)	130.6 (2)	C(6)—Fe—C(9)	68.8 (3)
C(7)—Fe—C(9)	69.1 (2)	C(8)—Fe—C(9)	40.3 (3)
C(1)—Fe—C(10)	117.5 (2)	C(2)—Fe—C(10)	149.5 (3)
C(3)—Fe—C(10)	168.5 (3)	C(4)—Fe—C(10)	131.2 (2)
C(5)—Fe—C(10)	110.0 (2)	C(6)—Fe—C(10)	41.1 (2)
C(7)—Fe—C(10)	69.5 (2)	C(8)—Fe—C(10)	67.8 (2)
C(9)—Fe—C(10)	40.2 (3)	C(1′)—Fe′—C(2′)	42.0 (2)
C(1′)—Fe′—C(3′)	69.3 (3)	C(2′)—Fe′—C(3′)	41.1 (2)
C(1′)—Fe′—C(4′)	68.8 (2)	C(2′)—Fe′—C(4′)	69.1 (2)
C(3′)—Fe′—C(4′)	40.6 (2)	C(1′)—Fe′—C(5′)	41.2 (2)
C(2′)—Fe′—C(5′)	69.7 (2)	C(3′)—Fe′—C(5′)	68.4 (2)
C(4′)—Fe′—C(5′)	40.1 (2)	C(1′)—Fe′—C(6′)	96.4 (2)
C(2′)—Fe′—C(6′)	111.6 (2)	C(3′)—Fe′—C(6′)	150.8 (2)
C(4′)—Fe′—C(6′)	157.7 (3)	C(5′)—Fe′—C(6′)	117.9 (2)
C(1′)—Fe′—C(7′)	111.2 (2)	C(2′)—Fe′—C(7′)	95.8 (2)
C(3′)—Fe′—C(7′)	117.7 (2)	C(4′)—Fe′—C(7′)	158.0 (3)
C(5′)—Fe′—C(7′)	150.5 (2)	C(6′)—Fe′—C(7′)	42.2 (3)
C(1′)—Fe′—C(8′)	150.1 (2)	C(2′)—Fe′—C(8′)	117.4 (2)
C(3′)—Fe′—C(8′)	110.2 (3)	C(4′)—Fe′—C(8′)	131.4 (3)
C(5′)—Fe′—C(8′)	168.3 (2)	C(6′)—Fe′—C(8′)	69.3 (3)
C(7′)—Fe′—C(8′)	40.9 (2)	C(1′)—Fe′—C(9′)	158.6 (3)
C(2′)—Fe′—C(9′)	157.4 (2)	C(3′)—Fe′—C(9′)	130.7 (3)
C(4′)—Fe′—C(9′)	119.5 (2)	C(5′)—Fe′—C(9′)	131.3 (2)
C(6′)—Fe′—C(9′)	69.0 (2)	C(7′)—Fe′—C(9′)	69.0 (2)
C(8′)—Fe′—C(9′)	40.4 (3)	C(1′)—Fe′—C(10′)	119.0 (3)
C(2′)—Fe′—C(10′)	151.4 (2)	C(3′)—Fe′—C(10′)	167.4 (2)
C(4′)—Fe′—C(10′)	130.8 (2)	C(5′)—Fe′—C(10′)	110.9 (2)
C(6′)—Fe′—C(10′)	41.4 (2)	C(7′)—Fe′—C(10′)	69.6 (2)
C(8′)—Fe′—C(10′)	67.8 (3)	C(9′)—Fe′—C(10′)	39.9 (3)
Fe—C(1)—C(2)	68.7 (3)	Fe—C(1)—C(5)	71.3 (3)
C(2)—C(1)—C(5)	108.6 (5)	Fe—C(1)—C(11)	121.0 (4)

Table 2 (*cont.*)

C(2)—C(1)—C(11)	126.5 (5)	C(5)—C(1)—C(11)	124.6 (5)
Fe—C(2)—C(1)	69.5 (3)	Fe—C(2)—C(3)	70.7 (3)
C(1)—C(2)—C(3)	106.5 (5)	Fe—C(2)—C(13)	121.3 (4)
C(1)—C(2)—C(13)	127.0 (6)	C(3)—C(2)—C(13)	126.3 (6)
Fe—C(3)—C(2)	67.8 (3)	Fe—C(3)—C(4)	71.5 (3)
C(2)—C(3)—C(4)	109.5 (5)	Fe—C(4)—C(3)	68.3 (4)
Fe—C(4)—C(5)	69.0 (4)	C(3)—C(4)—C(5)	107.5 (6)
Fe—C(5)—C(1)	67.7 (3)	Fe—C(5)—C(4)	70.6 (4)
C(1)—C(5)—C(4)	107.9 (5)	Fe—C(6)—C(7)	68.3 (3)
Fe—C(6)—C(10)	71.6 (3)	C(7)—C(6)—C(10)	107.7 (5)
Fe—C(6)—C(12)	121.0 (4)	C(7)—C(6)—C(12)	126.4 (5)
C(10)—C(6)—C(12)	125.7 (5)	Fe—C(7)—C(6)	69.8 (3)
Fe—C(7)—C(8)	71.5 (4)	C(6)—C(7)—C(8)	107.5 (5)
Fe—C(7)—C(14)	121.9 (4)	C(6)—C(7)—C(14)	126.0 (5)
C(8)—C(7)—C(14)	126.4 (5)	Fe—C(8)—C(7)	67.6 (4)
Fe—C(8)—C(9)	71.4 (4)	C(7)—C(8)—C(9)	109.5 (6)
Fe—C(9)—C(8)	68.3 (3)	Fe—C(9)—C(10)	69.4 (3)
C(8)—C(9)—C(10)	107.3 (6)	Fe—C(10)—C(6)	67.3 (3)
Fe—C(10)—C(9)	70.4 (3)	C(6)—C(10)—C(9)	108.0 (5)
C(1)—C(11)—O(1)	114.8 (5)	C(6)—C(12)—O(1)	114.4 (5)
C(2)—C(13)—O(2)	115.3 (5)	C(7)—C(14)—O(2)	114.0 (5)
C(11)—O(1)—C(12)	114.2 (5)	C(13)—O(2)—C(14)	114.6 (5)
Fe′—C(1′)—C(2′)	69.0 (3)	Fe′—C(1′)—C(5′)	70.2 (3)
C(2′)—C(1′)—C(5′)	107.5 (5)	Fe′—C(1′)—C(13′)	120.9 (4)
C(2′)—C(1′)—C(13′)	126.1 (5)	C(5′)—C(1′)—C(13′)	126.1 (5)
Fe′—C(2′)—C(1′)	69.1 (3)	Fe′—C(2′)—C(3′)	70.8 (4)
C(1′)—C(2′)—C(3′)	107.3 (5)	Fe′—C(2′)—C(11′)	121.9 (4)
C(1′)—C(2′)—C(11′)	126.9 (5)	C(3′)—C(2′)—C(11′)	125.6 (5)
Fe′—C(3′)—C(2′)	68.1 (3)	Fe′—C(3′)—C(4′)	70.9 (4)
C(2′)—C(3′)—C(4′)	108.7 (6)	Fe′—C(4′)—C(3′)	68.5 (3)
Fe′—C(4′)—C(5′)	68.3 (3)	C(3′)—C(4′)—C(5′)	107.5 (5)
Fe′—C(5′)—C(1′)	68.5 (3)	Fe′—C(5′)—C(4′)	71.5 (3)
C(1′)—C(5′)—C(4′)	109.0 (5)	Fe′—C(6′)—C(7′)	69.0 (3)
Fe′—C(6′)—C(10′)	71.2 (3)	C(7′)—C(6′)—C(10′)	107.2 (5)
Fe′—C(6′—C(14′)	120.4 (4)	C(7′)—C(6′)—C(14′)	126.8 (5)
C(10′)—C(6′)—C(14′)	125.8 (6)	Fe′—C(7′)—C(6′)	68.8 (3)
Fe′—C(7′)—C(8′)	71.3 (3)	C(6′)—C(7′)—C(8′)	107.4 (5)
Fe′—C(7′)—C(12′)	121.0 (4)	C(6′)—C(7′)—C(12′)	125.8 (6)
C(8′)—C(7′)—C(12′)	126.6 (6)	Fe′—C(8′)—C(7′)	67.7 (3)
Fe′—C(8′)—C(9′)	70.6 (4)	C(7′)—C(8′)—C(9′)	108.8 (6)
Fe′—C(9′)—C(8′)	69.0 (4)	Fe′—C(9′)—C(10′)	69.3 (4)
C(8′)—C(9′)—C(10′)	108.0 (6)	Fe′—C(10′)—C(6′)	67.4 (3)
Fe′—C(10)′—C(9′)	70.8 (4)	C(6′)—C(10′)—C(9′)	108.7 (6)
C(2′)—C(11′)—O(1′)	113.9 (5)	C(7′)—C(12′)—O(1′)	113.5 (5)
C(1′)—C(13′)—O(2′)	115.7 (5)	C(6′)—C(14′)—O(2′)	114.1 (5)
C(11′)—O(1′)—C(12′)	114.0 (5)	C(13′)—O(2′)—C(14′)	114.4 (5)

tropic, H atoms calculated and fixed in idealized positions [d(C—H) = 0.96 Å, $U = 1.2 \times U_{\rm iso}$ of attached C], $R_F = 4.75\%$, $wR_F = 5.97\%$. $S = 1.21$, $w^{-1} = \sigma^2(F_o) + gF_o^2$, $g = 0.001$, $(\Delta/\sigma)_{\rm max} = 0.003$, $(\Delta\rho)_{\rm max} = 0.51$ e Å^{-3}, $(\Delta\rho)_{\rm min} = -0.50$ e Å^{-3}, atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp., 99, 149). Computer programs: Sheldrick (1985). Atomic parameters are given in Table 1,* bond distances and angles in Table 2. The asymmetric unit consists of two chemically similar but crystallographically unique molecules of (2). Both molecules are ordered. The molecular structure and labeling scheme for each molecule is shown in Fig. 1. Each ferrocene molecule

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54145 (23 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR0317]

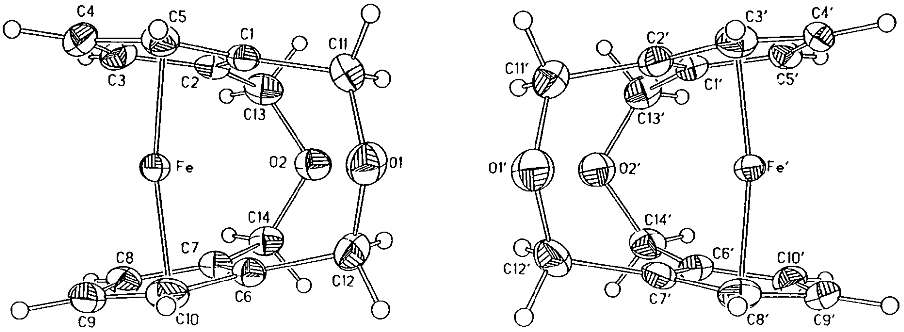


Fig. 1. Molecular structure and atom-numbering scheme in the two crystallographically unique molecules of (2). Non-H atoms are shown as 50% probability thermal ellipsoids; H atoms are drawn as spheres of arbitrary radius.

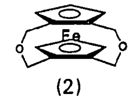
Table 3. *Selected molecular parameters for bridged ferrocenes*

	(2)*	(4)	(5)
Iron-to-ring distance (Å)	1.629 (6)	1.628 (6)	1.630 (1)
Ring-ring tilt angle (°)	15.7 and 16.6	15.0	13.1
Ring-ring twist angle (°)	0.3	0.3 (4)	1.1 (1)
Iron-to-oxygen distance (Å)	3.288 (5) (*endo*) 3.244 (5) (*exo*)	3.25 (2)	–
Iron-to-βC distance (Å)	–	3.30 (5)	3.312 (3)
αC-βO-αC angle (°)	114.5 (5) (*endo*) 114.1 (5) (*exo*)	113 (3)	–
αC-βC-αC angle (°)	–	111.8 (1)	113.1 (1)

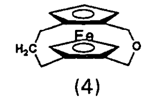
* These parameters are averaged over both independent molecules in the asymmetric unit.

has two heteroannular oxybismethylene bridges joining the 1,1′ and 2,2′ positions of the Cp rings. The opposing Cp rings are tilted in the direction of the tethers with dihedral angles between the rings being 15.7 and 16.6°, respectively, in the two crystallographically unique molecules. Thus, the substituted Cp carbons have an average Fe—C distance of 1.99 Å, the Cp carbon atoms adjacent to the two substituted carbons have an average distance of 2.04 Å, and the remaining Cp carbon atoms have an average distance of 2.07 Å. The overall average Fe—C_{Cp} distance of 2.027 (6) Å in (2) is slightly shorter than the average Fe—C distance of 2.045 (3) Å found in unsubstituted ferrocene (Seiler & Dunitz, 1979). There is no significant variation among C—C distances in either molecule of (2) [average C—C distance 1.420 (8) Å, comparable to that found in ferrocene].

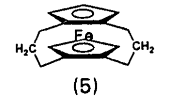
Discussion. Analogs of (2) in which one [(4)] or both [(5)] of the oxygens are replaced by carbon have also been studied by X-ray crystallography (Hillman & Austin, 1987). Structural parameters shown in Table 3 show very close agreement among the three; on average, the Fe—βO distance is slightly shorter that the Fe—βC distances, although a detailed comparison is difficult due to the large e.s.d.'s associated with compound (4). Compound (5) has an average Fe—βC distance of 3.312 (3) Å and (4) has an average Fe—βC distance of 3.30 (5) Å and an Fe—βO distance of 3.25 (2) Å. There are two types of Fe—O distances in (2), with the Fe—O_{endo} distance longer than the Fe—O_{exo} distance [3.288 (5) *versus* 3.244 (5) Å]. Possible conformations of the β-O or β-C atoms are depicted in the scheme below. Compound (5) crystallizes in the *exo-exo* conformation, which seems the most likely based on steric considerations. The β-carbon atoms in (4) are disordered and therefore may be participating in the *endo-endo* $\rightleftharpoons$ *endo-exo* $\rightleftharpoons$ *exo-exo* equilibrium depicted in the scheme below. In contrast to these analogs, the oxygen atoms of (2) are ordered in the *endo-exo* conformation. While the *endo-endo* configuration can be ruled out on the basis of steric repulsion, the preference on the part of (2) for *endo-exo* over *exo-exo* may be attributed to a favorable dipole–dipole interaction between the ethers in the *endo-exo* geometry. In fact, the *endo-exo* conformation occurs despite the presence of some significant steric repulsion in this conformation, as suggested by the longer Fe—O distances for the *endo* oxygens of (2).



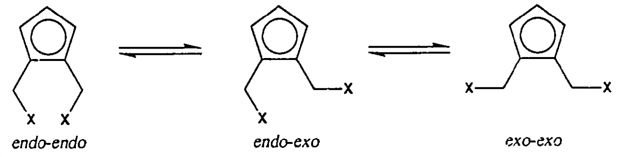
(2)



(4)



(5)



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