

The octamolybdate anion is built up of eight distorted Mo—O octahedra linked together by bridging oxygen atoms. There are two types of Mo—Mo distances: (a) those between octahedra sharing edges (3.222–3.544 Å) and (b) octahedra sharing corners (4.593–4.619 Å). The Mo—O distances are classified in three types: short (1.693–1.749 Å), medium (1.895–2.014 Å) and long (2.156–2.484 Å), as commonly occurs in this kind of polyanion.

The distortions of MoO₆ octahedra have been evaluated by different equations (1–3). The results are shown in Table 3.

$$\Delta_1 = (1/6) \sum_i [(R_i - R)/R]^2, \quad (1)$$

$$\Delta_e = (1/12) \sum_i [(D_i - D)/D]^2, \quad (2)$$

$$\Delta_\alpha = (1/3) \sum_i [(\alpha_i - 180)/180]^2, \quad (3)$$

where R_i = individual Mo—O distances, R = mean MoO distance, D_i = individual O—O distances, D = mean O—O distance and α_i = individual O—Mo—O_{trans} angles.

Plotting Δ_1 versus Δ_α we obtain the linear regression formula $\Delta_1 = 0.0070 + 0.671\Delta_\alpha$ with a correlation coefficient of $r = 0.982$. Mo(2), Mo(3) and Mo(4) octahedra present a similar degree of distortion. The Mo(1) octahedron shows a lower degree of distortion as Mo(1) occupies a central position in the polyanion.

Two crystallographically independent 2-methylpyridinium cations have standard dimensions (Table 2). Two N—H...O [N...O = 2.778 (5), 2.857 (5) Å] and three C—H...O [C...O = 3.164 (5), 3.240 (6), 3.271 (6) Å] hydrogen contacts (Taylor & Kennard, 1982; McCarron & Harlow, 1983) have been detected in the structure (Fig. 2). No doubt the C—H...O contacts cannot be considered as hydrogen bonds, but they indicate some degree of polarization.

Table 3. Octahedral distortion within the MoO₆ units

Distortion	Mo(1)	Mo(2)	Mo(3)	Mo(4)
$\Delta_1 \times 10^4$	126	167	203	171
$\Delta_e \times 10^4$	10	18	24	19
$\Delta_\alpha \times 10^4$	154	188	206	181

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Structure of a (Ferra- β -diketonato)BF₂ Complex Containing a Methacryl Substituent*†

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Abstract. $(\eta\text{-C}_5\text{H}_5)(\text{OC})\text{Fe}(\text{MeCO})\{\text{[H}_2\text{C}=\text{C}(\text{Me})\text{-CQ}\}\text{BF}_2$; $M_r = 309.89$, monoclinic, $P2_1/c$, $a = 7.8219$ (9), $b = 11.794$ (2), $c = 15.438$ (2) Å, $\beta =$

111.58 (1)°, $V = 1324.40$ Å³, $Z = 4$, $D_x = 1.554$ g cm⁻³, Mo $K\beta_1$, $\lambda = 0.63225$ Å (for cell constants), Mo $K\alpha$ for data collection, $\mu = 11.6$ cm⁻¹, $F(000) = 632$, $T = 294$ K, final $R = 0.042$ for 3863 observed reflections. The ferra- β -diketonato ring adopts a boat-shaped conformation having the carbonyl ligand in an axial position. The Fe—C(acyl) distances of

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† IUPAC name: difluoro[5-methyl-3-(carbonylcyclopentadienyl-ferra)-5-hexene-2,4-dionato-*O,O'*]boron.

1.862 (6) and 1.867 (2) Å indicate considerable multiple-bond character. The methacryl substituent is oriented relative to the ferra-chelate ring such that π -electron delocalization is not extensive.

Introduction. (Ferra- β -diketonato)BF₂ complexes containing alkenyl substituents react as activated dienophiles in Diels–Alder cycloaddition reactions (Lenhart, Lukehart & Sacksteder, 1986). The complex of this type having a methacryl substituent on the ferra-chelate ring forms Diels–Alder adducts with isoprene, 2,3-dimethyl-1,3-butadiene, *trans*-2-methyl-1,3-pentadiene and cyclopentadiene. Kinetic studies reveal that this complex reacts with 2,3-dimethyl-1,3-butadiene at a rate 50 times greater than the rate of reaction of this diene with methyl methacrylate under similar reaction conditions. Furthermore, owing to the highly asymmetric Fe center within the methacryl complex, cycloaddition with isoprene, 2,3-dimethyl-1,3-butadiene and *trans*-2-methyl-1,3-pentadiene occurs with very high stereoselectivity (>90% yield of one enantiomeric pair constituting two of the four possible diastereomers). A structural study was undertaken to confirm unambiguously the identity of the ferra-dienophile and to examine structural effects that might relate to the activation of the methacryl substituent toward Diels–Alder cycloadditions and to the observed high stereoselectivity of these reactions.

Experimental. Above complex prepared as described elsewhere (Lenhart, Lukehart & Sacksteder, 1986) and crystallized from ether/hexane solution at 253 K as yellow–orange crystals; value of D_m not measured; preliminary precession photographs showed $2/m$ Laue symmetry and systematic absences along $0k0$ for k odd and on $h0l$ for l odd which identified the space group as $P2_1/c$. Cell parameters determined by least-squares fit of 2θ , ω , and χ diffractometer settings for 15 reflections in the range $41^\circ \leq 2\theta \leq 45^\circ$ as measured at both $\pm 2\theta$ with Mo $K\beta_1$ radiation ($\lambda = 0.63225$ Å).

Intensity data collected from a crystal with dimensions $0.9 \times 0.2 \times 0.5$ mm, mounted with longest dimension parallel to the ϕ axis of four-circle automated Picker diffractometer; control software used for the Picker FACS-I system has been reported previously (Lenhart, 1975). 9537 reflections were measured out to $\sin\theta/\lambda$ of 0.7042 Å⁻¹ ($\pm h \pm k \pm l$ for $0^\circ \leq 2\theta \leq 20^\circ$ and $\pm hkl$, $\pm h-k-l$, for $20^\circ \leq 2\theta \leq 60^\circ$) using Mo $K\alpha$ radiation and $\theta-2\theta$ step scans; step size $0.05^\circ 2\theta$ with a scan width of 1.15° plus a dispersion term; each step counted for 1 s and the background counted for 10 s at each end of each scan; 0.002 in (0.005 cm) Nb filter used below $20^\circ 2\theta$ and 0.001 in (0.0025 cm) Nb filter above 20° . Four standard reflections monitored throughout data collection; the intensities decreased by 6%. Absorption corrections calculated with *ORABS* (Wehe, Busing & Levy, 1962) using a Gaussian grid of

$8 \times 8 \times 8$; maximum, minimum, and average transmission factors 0.791, 0.552, and 0.689, respectively; symmetry-related reflections averaged to give 3863 independent structure factors. All reflections (including 188 with zero intensity) used in the refinement.

Fe position determined by means of a sharpened Patterson function, and the phases provided by the Fe atom used for difference syntheses which revealed the positions of the remaining atoms. After preliminary least-squares refinement, all H atoms except those on the C(4) methyl group were located from a difference synthesis. The remaining three H atoms were treated as six half-hydrogens at 60° intervals to approximate the rotationally disordered methyl group.

Final refinement carried out with all heavy atoms having anisotropic temperature factors and H atoms having isotropic temperature factors. C(4) hydrogens were not included in the refinement. The quantity minimized was $\sum w(|F_o| - |F_c|)^2$ where $w = 1/\sigma(F_o)^2$. The variance, σ^2 , was based on counting statistics alone and included the usual instability term (4.0% in this case). Atomic scattering factors were those tabulated by Cromer & Mann (1968) except for hydrogen where those of Stewart, Davidson & Simpson (1965) were used. Anomalous-dispersion factors used for all atoms except H were those given by Cromer & Liberman (1970). Using all reflections, final $R = 0.042$, $wR = 0.041$. Maximum shift-to-e.s.d. ratio for final refinement cycle = 0.0419; e.s.d. of an observation of unit weight = 1.9. Maximum and minimum electron densities on the final difference map 0.25 and -0.50 e Å⁻³, near the F and Fe atoms respectively. Atom numbering is shown in Fig. 1 and a complete listing of atomic parameters is given in Table 1. Values shown were used before rounding to calculate the final structure factors. A list of selected interatomic distances and angles is provided in Table 2.*

Structure factor, electron density and bond distance and angle calculations were performed with the *XRAY67* programs as implemented and updated on the Vanderbilt DEC-10 computer (Stewart, 1967).

An *ORTEP* view of the molecular structure is shown in Fig. 1.

Discussion. The ferra-chelate ring adopts a boat-shaped structure. In this boat the Fe and B atoms are displaced 0.45 and 0.42 Å, respectively, to the same side of the plane defined by C(1), O(1), C(2) and O(2) [maximum atomic deviation from coplanarity is 0.025 Å by atom

* Lists of final atomic positional and thermal parameters, additional bond distances and angles, selected least-squares planes and dihedral angle data, and final observed and calculated structure factors have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42845 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

O(2)]. The particular boat isomer observed has a carbonyl ligand in the axial position at the Fe atom with the cyclopentadienyl ligand occupying an equatorial position. This boat structure has been observed in one other (ferra- β -diketonato)BF₂ complex (Lenhart, Lukehart & Warfield, 1980). Both of these compounds exhibit a C—O stretching band at 2000 cm⁻¹ in the infrared spectrum that is apparently a diagnostic indication of this particular boat isomer. Intraring bond distances reveal a nearly symmetrical six-membered ring [Fe—C = 1.862 (6), 1.867 (2); C—O = 1.290 (2), 1.289 (2); O—B = 1.476 (3), 1.465 (3) Å]. The values of the Fe—C and C—O distances represent bonds having appreciable multiple-bond character (Lenhart, Lukehart & Warfield, 1980).

The plane of the methacryl substituent [defined by C(1), C(5), C(6), and C(7); maximum atomic deviation from coplanarity is 0.033 Å by atom C(5)] forms a dihedral angle of 70.5° with the [C(1), O(1), C(2), O(2)] plane. Conjugation of the π -electron system of the methacryl group and of the ferra-chelate ring is, therefore, not significant. The C(1)—C(5) distance of 1.500 (3) Å and the C(5)—C(6) distance of 1.317 (3) Å represent normal C(sp²)—C(sp²) single- and double-bond distances, respectively.

Although we now believe that this complex undergoes Diels—Alder cycloaddition reactions in solution as the alternate boat isomer with a coplanar *transoid* orientation of the C(1)—O(1) and C(5)—C(6) bonds about the C(1)—C(5) bond, the structure reported herein confirms the identity of the ferra-methacryl dienophile. Furthermore, the asymmetric environment about the Fe atom is presumably responsible for the stereoselectivity of these reactions. Even in this solid-state conformation, approach to either side of the planar methacryl substituent is clearly nondegenerate.

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Table 1. Fractional atomic coordinates and B or B equivalent thermal parameters (Å²) in the form: $B_{eq} = \frac{8}{3}\pi^2(U_{11}^2 + U_{22}^2 + U_{33}^2)$ where the U_{ii} are the r.m.s. principal axis deviations

	x	y	z	B or B _{eq}
Fe	0.60498 (3)	0.22166 (2)	0.14091 (1)	3.29
F(1)	0.14442 (17)	-0.02671 (10)	0.10178 (11)	7.32
F(2)	0.29790 (18)	0.07303 (10)	0.23199 (9)	6.76
O(1)	0.23145 (15)	0.15692 (10)	0.08776 (9)	4.55
O(2)	0.45120 (18)	0.00400 (10)	0.13994 (10)	5.24
O(3)	0.59684 (31)	0.22974 (15)	0.32615 (10)	8.26
B	0.27990 (29)	0.05112 (18)	0.14250 (18)	4.99
C(1)	0.34996 (22)	0.23273 (13)	0.08716 (11)	3.46
C(2)	0.58724 (22)	0.06373 (16)	0.13734 (11)	4.39
C(3)	0.59603 (28)	0.22468 (16)	0.25346 (12)	4.86
C(4)	0.73761 (31)	-0.01592 (19)	0.13509 (19)	7.22
C(5)	0.24745 (23)	0.33371 (15)	0.03404 (12)	4.25
C(6)	0.25435 (32)	0.42984 (18)	0.07859 (19)	6.09
C(7)	0.12834 (41)	0.31383 (27)	-0.06534 (18)	6.76
C(8)	0.64901 (30)	0.32090 (19)	0.03693 (14)	5.04
C(9)	0.68637 (29)	0.38707 (17)	0.11628 (14)	4.95
C(10)	0.83111 (27)	0.33636 (20)	0.18799 (16)	5.60
C(11)	0.88484 (27)	0.23914 (21)	0.15314 (17)	5.71
C(12)	0.77055 (30)	0.22889 (19)	0.05902 (16)	5.40
H(61)	0.2043 (30)	0.4945 (19)	0.0494 (15)	7.13 (58)
H(62)	0.3461 (25)	0.4432 (16)	0.1519 (13)	6.09 (50)
H(71)	0.0776 (34)	0.3835 (24)	-0.1016 (16)	10.04 (76)
H(72)	0.1989 (34)	0.2881 (19)	-0.0956 (16)	6.36 (65)
H(73)	0.0599 (36)	0.2407 (22)	-0.0789 (18)	9.32 (83)
H(8)	0.5678 (27)	0.3300 (18)	-0.0205 (13)	6.24 (53)
H(9)	0.6279 (29)	0.4525 (19)	0.1213 (14)	6.94 (58)
H(10)	0.8861 (26)	0.3659 (18)	0.2476 (14)	6.57 (54)
H(11)	0.9803 (29)	0.1907 (17)	0.1923 (14)	6.20 (51)
H(12)	0.7736 (28)	0.1747 (19)	0.0170 (13)	6.80 (55)

Table 2. Selected interatomic distances (Å) and angles (°)

Fe—C(1)	1.862 (2)	C(1)—C(5)	1.500 (2)
Fe—C(2)	1.867 (2)	C(5)—C(6)	1.317 (3)
Fe—C(3)	1.765 (2)	C(5)—C(7)	1.492 (3)
Fe—C(8)	2.115 (2)	C(8)—C(9)	1.390 (3)
Fe—C(9)	2.129 (2)	C(9)—C(10)	1.394 (3)
Fe—C(10)	2.131 (2)	C(10)—C(11)	1.395 (4)
Fe—C(11)	2.137 (2)	C(11)—C(12)	1.403 (3)
Fe—C(12)	2.118 (3)	C(12)—C(8)	1.400 (3)
C(1)—O(1)	1.290 (2)	B—O(1)	1.476 (3)
C(2)—O(2)	1.289 (2)	B—O(2)	1.464 (3)
C(3)—O(3)	1.121 (3)	B—F(1)	1.368 (2)
C(2)—C(4)	1.516 (3)	B—F(2)	1.361 (3)
Fe—C(1)—C(5)	124.68 (12)	B—O(2)—C(2)	124.57 (15)
Fe—C(1)—O(1)	127.08 (11)	C(8)—C(9)—C(10)	107.8 (2)
O(1)—C(1)—C(5)	108.24 (13)	C(9)—C(10)—C(11)	108.6 (2)
Fe—C(2)—O(2)	126.69 (14)	C(10)—C(11)—C(12)	107.6 (2)
Fe—C(2)—C(4)	124.69 (14)	C(8)—C(12)—C(11)	107.6 (2)
O(2)—C(2)—C(4)	108.6 (2)	C(9)—C(8)—C(12)	108.5 (2)
Fe—C(3)—O(3)	176.9 (2)	F(1)—B—O(1)	108.16 (15)
C(1)—Fe—C(2)	90.06 (7)	F(2)—B—O(1)	109.2 (2)
C(1)—Fe—C(3)	90.77 (9)	O(1)—B—O(2)	110.5 (2)
C(2)—Fe—C(3)	91.13 (8)	F(1)—B—F(2)	111.6 (2)
C(1)—C(5)—C(6)	119.1 (2)	F(1)—B—O(2)	107.1 (2)
C(1)—C(5)—C(7)	116.3 (2)	F(2)—B—O(2)	110.20 (15)
C(6)—C(5)—C(7)	124.2 (2)		
O(2)—C(2)—C(4)	108.6 (2)		
B—O(1)—C(1)	123.77 (13)		

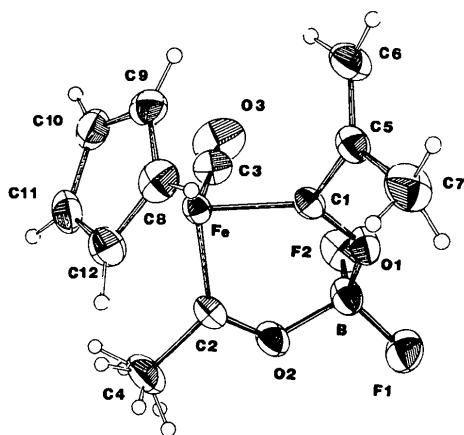


Fig. 1. An ORTEP view (Johnson, 1965) (ellipsoids at 30% probability) from below the chelate ring showing the atomic numbering scheme.

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Structure of *cis*-Tetrachloro(3-trifluoromethyl-2,5-dithiahexane)platinum(IV)

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Abstract. [PtCl₄(C₅H₉F₃S₂)], $M_r = 527.2$, monoclinic, $P2_1/n$, $a = 7.431$ (4), $b = 15.154$ (9), $c = 11.721$ (7) Å, $\beta = 99.29$ (1)°, $U = 1302.6$ (13) Å³, $Z = 4$, $D_x = 2.688$ g cm⁻³, D_m not measured, Mo $K\alpha$, $\lambda = 0.71069$ Å, $\mu = 120.2$ cm⁻¹, $F(000) = 976$, $T = 293$ K, $R = 0.020$ for 1648 unique reflections with $I \geq 3\sigma(I)$. The Pt atom displays distorted octahedral coordination with Pt-Cl = 2.314 (2)-2.338 (2) Å and Pt-S = 2.317 (2) and 2.320 (2) Å. The CF₃ substituent is pseudoequatorial with respect to the PtS₂C₂ chelate ring and the *S*-methyl substituents are mutually *anti*.

Introduction. The title complex was prepared by chlorine oxidation of the corresponding [PtCl₂L] complex, where $L =$ racemic MeSCH₂CH(CF₃)SMe. In its acetone-*d*⁶ solutions four isomers with relative abundances 52:40:4:4 can be observed by ¹⁹F NMR at ambient temperatures. The isomers can be identified by comparison with analogous platinum(II) complexes (Cross, Rycroft, Sharp & Torrens, 1980; Torrens, 1977; Hunter, 1982). They correspond to four enantiomeric pairs of diastereomers arising from the presence of a chiral C and two chiral S atoms. The PtS₂C₂ chelate ring constitutes a fourth chiral centre but interconversion in solution between its δ and λ forms is too fast for detection on the NMR timescale. This crystal-structure analysis reveals that the solid form of the title compound corresponds to the isomer identified as the second most abundant in solution.

Experimental. Crystals grown from acetone solution by solvent evaporation at 277 K; yellow needle; forms displayed and distances (cm) of faces from centroid

{011} 0.003 and {10 $\bar{1}$ } 0.009; Enraf-Nonius CAD-4F diffractometer, Mo $K\alpha$ X-rays, graphite monochromator; cell dimensions by least-squares fit to the setting angles of 23 automatically centred reflections with $10 < \theta < 13^\circ$; intensities of 3589 reflections with $2 \leq \theta \leq 25^\circ$ and $0 \leq h \leq 8$, $-18 \leq k \leq 7$, $-13 \leq l \leq 13$ measured from continuous $\theta/2\theta$ scans of 0.90° in θ ; two intensity standards measured every two hours; corrections for Lp and absorption (Gaussian quadrature, 96 grid points, transmission factors on F^2 0.45-0.72), no correction for decomposition or extinction required; R_{int} 0.022 for 1057 independent reflections measured at least twice; 2287 independent structure amplitudes; 1648 with $I \geq 3\sigma(I)$ used subsequently; structure solved by Patterson and difference syntheses; full-matrix least-squares refinement on F ; $w^{-1} = \sigma^2(F) + 2.25 \times 10^{-4} F^2$; final adjustment of 172 parameters (isotropic U for H atoms; anisotropic U_{ij} for the others) gave $wR = 0.019$, $S = 1.2$; maximum $\Delta/\sigma = 0.018$; final $|\Delta\rho|$ values < 0.63 e Å⁻³; neutral-atom scattering factors and complex anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974); *GX* crystallographic program package (Mallinson & Muir, 1985). Final atomic coordinates are presented in Table 1.*

*Lists of structure amplitudes, anisotropic displacement parameters, H-atom parameters and bond lengths and angles involving H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42884 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.