# Diels-Alder reactions utilizing (ferra-.beta.-diketonato)BF2 complexes containing alkynyl substituents as activated dienophiles

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$$c = [\mathrm{Sn}]_{\mathrm{total}}[\mathrm{Cl}^-]_{\mathrm{total}}$$

The chemical shift was then calculated for each point

$$\delta_{\mathrm{calcd}} = ([\mathrm{A}]\delta_{\mathrm{A}} + [\mathrm{C}]\delta_{\mathrm{C}})/([\mathrm{A}] + [\mathrm{C}])$$

and the results were compared to the experimental values. The fit parameters, $K$, $\delta_{\mathrm{A}}$, and $\delta_{\mathrm{C}}$, were varied iteratively using the gradient search procedure to find the best fit. Since $\delta_{\mathrm{A}}$ and $\delta_{\mathrm{C}}$ were known from the limiting values of the observed chemical shift, the best value of K could be found rapidly.

For two coupled equilibria (Scheme II), the situation was more complex. The chemical shift is given by

$$\delta_{\mathrm{calcd}} = ([\mathrm{A}]\delta_{\mathrm{A}} + [\mathrm{B}]\delta_{\mathrm{B}} + [\mathrm{C}]\delta_{\mathrm{C}})/([\mathrm{A}] + [\mathrm{B}] + [\mathrm{C}])$$

Using the equilibrium expressions and the substitutions

$$[\mathrm{A}] = [\mathrm{Sn}]_{\mathrm{total}} - [\mathrm{B}] - [\mathrm{C}]$$

$$[\mathrm{Cl}^-] = [\mathrm{Cl}^-]_{\mathrm{total}} - [\mathrm{B}] - 2[\mathrm{C}]$$

one obtains the following equations for [B] and [C]:

$$[\mathrm{B}] = (-b - (b^2 - 4c)^{1/2})/2$$

$$-b = [\mathrm{Sn}]_{\mathrm{total}} + [\mathrm{Cl}^-]_{\mathrm{total}} - 3[\mathrm{C}] + 1/K_1$$

$$c = -[\mathrm{C}](2[\mathrm{Sn}]_{\mathrm{total}} + [\mathrm{Cl}^-]_{\mathrm{total}} - 2[\mathrm{C}]) + [\mathrm{Sn}]_{\mathrm{total}}[\mathrm{Cl}^-]_{\mathrm{total}}$$

$$[\mathrm{C}] = [\mathrm{B}]([\mathrm{Cl}^-]_{\mathrm{total}} - [\mathrm{B}])/(2[\mathrm{B}] + 1/K_2)$$

Rather than solving this pair of simultaneous equations explicitly for [B] and [C], we evaluated them iteratively by the "fixed-point" method.[7] An initial estimate for [C] was used in calculating [B], this new value of [B] was used to recalculate [C], and so on until convergence gave an unchanging value for the calculated chemical shift. In this way, $\delta_{\mathrm{calcd}}$ was obtained for the full range of chloride/tin ratios for which the chemical shift had been measured experimentally.

In practice, the fixed-point iteration did not converge reliably for large $K$'s (on the order of 100 or more) when the ratio of added halide ion to tin sites was greater than 1:1 (i.e., a 2:1 molar ratio of added $Cl^-$ to ditin compound), so the few data points at higher ratios had to be neglected in the computer fit. This may reflect a shortcoming of the method. We believe that when species C becomes the predominant species, a lack of convergence can result from a sign change in the derivative of the expression for [C].

The best values for $K_1$ and $K_2$ were determined by comparison of the observed and calculated chemical shift values, using the gradient search procedure. The results proved to be quite sensitive to the initial choice of $\delta_{\mathrm{B}}$, the chemical shift value assigned to the 1:1 complex of ditin host and chloride. Allowing the program to vary this parameter did not provide a unique best value. Comparison of the results for the compounds **1**, **2**, and **4** suggested that −48 ppm was a good estimate for the chemical shift of species B, and this value was used to obtain optimized equilibrium constants for the purposes of comparison among the ligands. Additional improvement in the fit to the experimental data could be achieved by allowing the computer program to vary the other two chemical shift parameters, $\delta_{\mathrm{A}}$ and $\delta_{\mathrm{C}}$, after the equilibrium constants had been calculated. Only small changes in these two parameters were necessary to obtain the best possible fit, a reassuring result since their values were expected to lie close to the observed chemical shifts for the free ligands ($\delta_{\mathrm{A}}$) and the ligands in the presence of excess added chloride ($\delta_{\mathrm{C}}$).

The $\chi^2$ statistic provides an estimate of reliability of our mathematical model. Assuming a standard deviation of ±2 ppm in the chemical shift measurements resulting mainly from errors in the measured concentrations, we obtained reduced $\chi^2$ values ($\chi^2$ divided by the number of degrees of freedom, in our case usually 15–21 data points and five parameters) below 0.5. This permits good confidence (greater than 90% probability) in the validity of our mathematical model.

**Registry No.** **1**, 105162-88-7; **2**, 105182-65-8; **3**, 105162-89-8; **4**, 105162-90-1; **5**, 683-18-1; tetraethylammonium chloride, 16887-00-6; 5,5,16,16-tetraphenyl-5,16-distannaeicosane, 105162-91-2; 1,12-dibromo-1,1,12,12-tetraphenyl-1,12-distannadodecane, 87518-49-8; 1,1,10,10-tetraphenyl-1,10-distannacyclooctadecane, 87531-98-4; 1,1,12,12-tetraphenyl-1,12-distannacyclododecosane, 87518-37-4; 1,1,14,14-tetraphenyl-1,14-distannacyclohexacosane, 87518-38-5.

(7) See, for example: Conte, S. D.; de Boor, C. *Elementary Numerical Analysis; an Algorithmic Approach*, 2nd ed., McGraw-Hill: New York, 1972, p 84.

# Diels–Alder Reactions Utilizing (Ferra-β-diketonato)$BF_2$ Complexes Containing Alkynyl Substituents as Activated Dienophiles

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Two (ferra-β-diketonato)$BF_2$ complexes containing propynyl substituents on the ferra-chelate ring have been prepared. The propynyl substituents react with cyclopentadiene at room temperature to give the corresponding norbornadienyl Diels–Alder cycloaddition products. A complex containing both propynyl and methacryl substituents on the ferra-chelate ring exhibits chemoselectivity and stereoselectivity in Diels–Alder adduct formation.

## Introduction

We have reported recently that (ferra-β-diketonato)$BF_2$ complexes which have alkenyl substituents attached to the ferra-chelate ring react as activated dienophiles in Diels–Alder cycloaddition reactions.[1] For example, the addition of dimethylbutadiene to such a complex containing a methacryl substituent occurs at a rate 50 times greater

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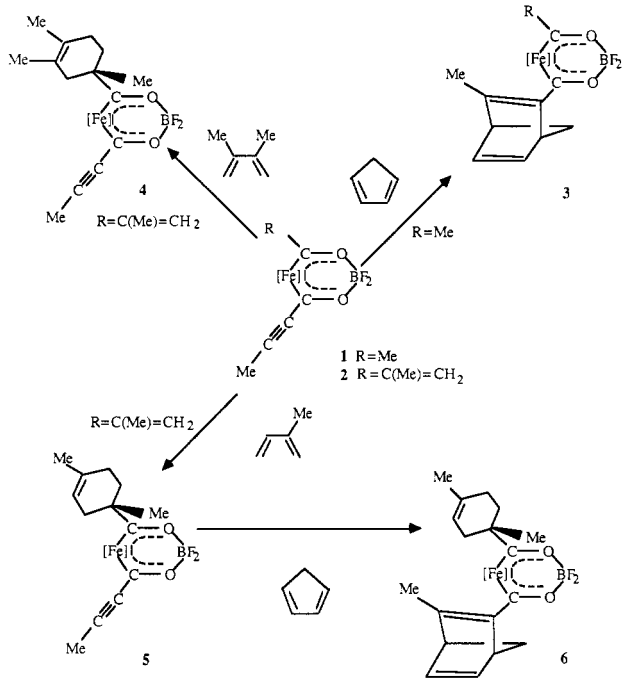
**Scheme I**




[Fe]=(η-$C_5H_5$)(OC)Fe, where the $C_5H_5$ ligand projects above the pseudo-plane of the chelate ring in an axial site of the boat conformation.[1]

than the rate of cycloaddition of dimethylbutadiene to methyl methacrylate. Furthermore, although these organometallic dienophiles react with a regioselectivity consistent with that known for similar carbon dienophiles, the highly asymmetric Fe center affords high stereoselectivity when diastereomeric adducts are formed.

We now report an extension of this chemistry to include (ferra-β-diketonato)$BF_2$ complexes which contain alkynyl substituents attached to the ferra-chelate ring. These complexes activate the alkynyl group for subsequent Diels–Alder cycloaddition with cyclopentadiene at room temperature. Similar reactivity is known only for alkynoyl chlorides[2] or chromium carbene complexes having alkynyl substituents.[3] In addition, we report that preparation of a (ferra-β-diketonato)$BF_2$ complex containing two different dienophilic substituents on the ferra-chelate ring. Chemoselective Diels–Alder cycloaddition to these dienophiles affords unsymmetrically substituted ferra-chelate rings having organic substituents of considerable complexity. These or related complexes are potentially important molecules for synthetic applications to organic synthesis because of our demonstrated ability to effect interligand C–C bond formation across metalla-β-diketonato chelate rings by two independent methods.[4] Such successful interligand coupling reactions would combine these two ferra-chelate ring fragments into the same organic molecule.

## Results and Discussion

The principal results of this study are shown in Scheme I. The new (ferra-β-diketonato)$BF_2$ complexes **1** and **2** were prepared by using standard methods.[4] Each complex contains a propynyl substituent on the ferra-chelate ring and either a methyl, **1**, or methacryl, **2**, group as the other ferra-chelate ring substituent. In the infrared spectra of **1** and **2** in $CH_2Cl_2$ solution, both complexes exhibit an alkynyl C–C triple bond stretching band at 2195 $cm^{-1}$ and terminal C–O stretching bands at approximately 2000 and 1960 $cm^{-1}$. The relative intensities of these CO bands indicate that **1** exists predominantly in the boat conformation having an axial, terminal CO ligand while complex **2** exists as a nearly equal abundance of both boat isomers with only slight preference for the isomer having the axial, terminal CO ligand.[5]

Complex **1** reacts with cyclopentadiene at room temperature to give the norbornadienyl Diels–Alder adduct **3** in 34% yield as the sole isolated cycloaddition product. The presence of the bicyclo [2.2.1] substituent is indicated by a comparison of the $^1H$ NMR data of **3** with those of similar organic model compounds.[2,6,7] Methyl tetrolate (the closest carbon analogue to **1**) reacts with cyclopentadiene at 160 °C to give a Diels–Alder adduct in 26% yield.[8] The CO stretching band observed for **3** at 1960 $cm^{-1}$ indicates that the ferra-chelate ring exits only as the boat conformation having the terminal CO ligand in the equatorial position. This isomer preference is expected because of the presence of the bulky bicyclo [2.2.1] substituent attached to the ferra-chelate ring.[1,5] Complex **1** does not form Diels–Alder adducts with isoprene, 2,3-dimethyl-1,3-butadiene (DMB), or furan, presumably reflecting the weak dienophilicity of the propynyl group.

Compound **2** adds DMB to the methacryl substituent to give **4**. Major and minor resonances at δ 4.73 and 4.69, respectively, with relative intensities of 95:5 are observed in the $^1H$-NMR spectrum of **4** for the $C_5H_5$ ligand. Because regioisomerism is not possible for this DMB addition, we assign these resonances to the two diastereomers that could form during this reaction. The diastereomer shown is presumed to be the major one based on the results of our more comprehensive study of Diels–Alder additions to methacryl substituents of ferra-chelate rings.[1]

Isoprene adds to **2** to give complex **5**. Infrared data confirms the presence of a free propynyl substituent and only the boat isomer having an equatorial carbonyl ligand. The comparison of the $^1H$ NMR data for **5** with those of related complexes[1] indicates that the regiochemistry of the Diels–Alder cycloaddition occurs to give only the "para" isomer. Evidence of diastereomer formation was not detected by NMR.

Complex **5** reacts with cyclopentadiene to give the Diels–Alder bis-adduct **6** in 76% yield. This compound exists only as the boat isomer having an equatorial, terminal carbonyl ligand. The presence of the norbornadienyl ligand is evident from the $^1H$ NMR data and by comparison to the spectra data of complex **3**. Additional minor resonances for the $C_5H_5$ ligand at δ 4.73 and 4.62 probably represent diastereomers arising from differing stereochemistry at the Fe, norbornadienyl, or C($sp^3$)–Me cyclohexenyl asymmetric centers. The ratio of the area of the largest of these minor resonances to that of the major isomer is 12:88. We tentatively assign the preferred stereochemistry as that shown for **5** and **6** by direct analogy to the observed stereochemical preferences observed for isoprene addition to methacryl substituents in related

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(ferra-β-diketonato)$BF_2$ compounds.[1] These earlier stereochemical assignments have been confirmed by x-ray structural determinations.

## Experimental Section

All reactions and other manipulations were performed under dry, prepurified nitrogen. Diethyl ether, pentane, hexane, and tetrahydrofuran (THF) were dried over Na/K alloy with benzophenone having been added to the diethyl ether and THF. Methylene chloride was dried over $P_2O_5$. All solvents were distilled under nitrogen before use.

Infrared (IR) spectra were recorded on a Perkin-Elmer 727 spectrometer as methylene chloride solutions in 0.10-mm sodium chloride cavity cells with the solvent as a reference and a polystyrene film as a calibration standard. $^1H$ NMR spectra were obtained on either a JEOL MH-100 or a JEOL FX90-Q NMR spectrometer as $CDCl_3$ solutions with $Me_4Si$ as an internal reference. Microanalyses were performed by MicAnal Organic Microanalysis, Tucson, AZ.

The compounds ($\eta$-$C_5H_5$)$(OC)_2$ FeC(O)Me (**7**)[9] and ($\eta$-$C_5H_5$)$(OC)_2$Fe{[$H_2C$=C(Me)]C(O)} (**8**)[10] were prepared by literature methods.

**Preparation of ($\eta$-$C_5H_5$)(OC)Fe(MeCO){[(Me)C≡C]CO}$BF_2$ (1).** To a stirred solution of 1.02 g (4.64 mmol) of **7** in 100 mL of THF at room temperature was added 0.29 g of 93% propynyllithium which previously had been weighed out in a glovebag. Upon dissolution, a rapid color change to orange occurred and the solution was stirred for 1.5 h. The solvent was removed at reduced pressure using a warm water bath. $CH_2Cl_2$ (100 mL) was added to the residue to produce a clear orange solution which was then cooled to 0 °C. Gaseous $BF_3$ was bubbled through the solution at a moderate rate for 60 s. The 0 °C bath was removed, and the solution was allowed to warm to room temperature. The solvent was removed at reduced pressure. The residue was dissolved in 5 mL of $CH_2Cl_2$ and was chromatographed through a 13-cm column of Florisil in hexane. Methylene chloride was used to elute a bright orange band which was concentrated to approximately 20 mL. Pentane was added until the solution became slightly turbid. Crystallization at −20 °C afforded 0.49 g (35%) of orange needles: mp 126–129 °C; IR ($CH_2Cl_2$) $\nu$(C≡C) 2195 (m) $cm^{-1}$, II $\nu$(CO) 2000 (vs), 1960 (w) $cm^{-1}$; $^1H$ NMR ($CDCl_3$) $\delta$ 2.25 (s, 3, C≡C(Me)), 2.91 (s, 3, MeCO), 5.19 (s, 5, $C_5H_5$). Anal. Calcd for $C_{12}H_{11}O_3BF_2Fe$: C, 46.81; H, 3.61. Found: C, 46.59; H, 3.68.

**Preparation of ($\eta$-$C_5H_5$)(OC)Fe{[$H_2C$=C(Me)]CO}{[(Me)C≡C]CO}$BF_2$ (2).** To a stirred solution of 1.94 g (7.88 mmol) of **8** in 100 mL of THF at room temperature was added 0.34 g of 97% propynyllithium which previously had been weighed out in a glovebag. Upon dissolution, a rapiid color change to dark orange occurred and the solution was allowed to stir for 1.5 h. The solvent was removed at reduced pressure by using a warm water bath. $CH_2Cl_2$ (100 mL) was added to the red-orange solid residue. This produced a clear dark orange solution which was cooled to 0 °C. Gaseous $BF_3$ was bubbled through the solution at a moderate rate for 20 s. The 0 °C bath was removed, and the solution was allowed to warm to room temperature. The solvent was removed at reduced pressure. The residue was dissolved in 5 mL of $CH_2Cl_2$ and was chromatographed through a 13-cm column of Florisil in hexane using ether/$CH_2Cl_2$ (1:1) for band elution. The band was concentrated to approximately 30 mL, and pentane was added until the solution became slightly turbid. Crystallization at −20 °C afforded 1.32 g (50%) of orange micricrystalline material: mp 108–112 °C; IR ($CH_2Cl_2$) $\nu$(C≡C) 2195 (vs) $cm^{-1}$, $\nu$(CO) 2000 (vs), 1970 (s) $cm^{-1}$; $^1H$ NMR ($CDCl_3$) $\delta$ 1.86 (s, 3, C=C(Me), 2.22 (s, C≡C(Me)), 4.95 (s, 5, $C_5H_5$), 5.22, 5.41 (d, 2, $CH_2$=C, $J$ = 17.1 Hz). Anal. Calcd for $C_{14}H_{13}O_3BF_2Fe$: C, 50.35; H, 3.93. Found: C, 50.68, H. 3.87.

**Preparation of ($\eta$-$C_5H_5$)(OC)Fe(MeCO){(3-methyl-2-norbornyl-2,5-diene)CO}$BF_2$ (3).** Dicyclopentadiene was cracked thermally by a literature method.[11] In 8 mL of $CH_2Cl_2$ was dissolved 0.64 g of **1**. Cyclopentadiene (25 mL) was then added to the orange solution. This solution was stirred under an $N_2$ atmosphere for 4 days at room temperature. The solvent was evaporated at reduced pressure, and the residue was extracted into a minimum volume of ether. The solution was chromatographed on a 13-cm column of Florisil in hexane, and the brown-red product band was eluted by using ether/hexane (30:70). Dark brown microcrystalline material was obtained from the concentrated solution by crystallization at −20 °C: yield 0.27 g (34%); mp 144–145 °C; IR ($CH_2Cl_2$) $\nu$(CO) 1960 (vs) $cm^{-1}$; $^1H$ NMR ($CDCl_3$) 2.06 (s, 2, $CH_2$), 2.33 (s, 3, C=C(Me)), 2.84 (s, 3, MeCO), 3.49 (s, 1, H [bridgehead]), 4.22 (br, s, 1, H [bridgehead]), 4.72 (s, 5 , $C_5H_5$), 6.72 (br, s, 1, H), 6.89 (br, s, 1 H). Anal. Calcd for $C_{17}H_{17}O_3BF_2Fe$: C, 54.59; H, 4.59. Found: C, 54.24; H, 4.59.

**Preparation of ($\eta$-$C_5H_5$)(OC)Fe{[(Me)C≡C]CO}{[$CH_2CH_2$(Me)C=C(Me)$CH_2$(Me)C]CO}$BF_2$ (4).** To 0.25 g of **2** were added 9 mL of diethyl ether, 2 mL of $CH_2Cl_2$, and 5 mL of 2,3-dimethyl-1,3-butadiene. The dark orange solution was allowed to stir at room temperature for 30 h. The solvents were then removed at reduced pressure, and the residue was dissolved in 2 mL of $CH_2Cl_2$. The solution was chromatographed through a 13-cm column of Florisil in hexane and ether/hexane (50:50) was used to elute a red-brown band. The band was concentrated to approximately 25 mL and was cooled to -20 °C affording 0.10 g (32%) of brown crystals: mp 101–102 °C; IR ($CH_2Cl_2$) $\nu$(C≡C) 2195 $cm^{-1}$, $\nu$(CO) 1975 (vs) $cm^{-1}$; $^1H$ NMR ($CDCl_3$) $\delta$ 0.96 (s, 3, MeCCO), 1.27, 1.67, 1.71, 1.88, 2.38 (m, 6, ring $CH_2$ protons), 1.59 (br s, 6, MeC=CMe), 2.24 (s, 3, C≡CMe), 4.73 (s, 5, $C_5H_5$). Anal. Calcd for $C_{20}H_{23}O_3BF_2Fe$: C, 57.73; H, 5.58. Found: C, 57.77; H, 5.70.

**Preparation of ($\eta$-$C_5H_5$)(OC)Fe{[(Me)C≡C]CO}{[$CH_2CH_2$(Me)C=C(H)$CH_2$(Me)C]CO}$BF_2$ (5).** To 0.52 g of **2** were added 5 mL of $CH_2Cl_2$ and 5 mL of isoprene. The dark rust-colored solution was stirred at room temperature as a closed system for 48 h. The solvents were then removed at reduced pressure and the residue was extracted with a minimum volume ether. The solution was chromatographed on a 13-cm column of Florisil in hexane. A red band was eluted using hexane/ether (70:30), and the product was crystallized from ether/hexane solution at −20 °C as black crystals: yield 0.33 g (53%); mp 85–86 °C; IR ($CH_2Cl_2$) $\nu$(C≡C) 2195 (s) $cm^{-1}$; $^1H$ NMR ($CDCl_3$) $\delta$ 1.00 (s, 3, MeCCO), 1.55, 1.66, 1.88, 1.97, 2.57 (m, 6, ring $CH_2$ protons), 1.63 (br s, 3, MeC=CH), 2.24 (s, 3, C≡CMe), 4.73 (s, 5, $C_5H_5$), 5.29 (br s, 1, CH=CMe). Anal. Calcd for $C_{19}H_{21}O_3BF_2Fe$: C, 56.76; H, 5.28. Found: C, 56.89; H, 5.46.

**Preparation of ($\eta$-$C_5H_5$)(OC)Fe{(3-methyl-2-norbornyl-2,5-diene)CO}{[$CH_2CH_2$(Me)C=C(H)$CH_2$(Me)C]CO}$BF_2$ (6).** To 0.29 g of **5** was added 5 mL of $CH_2Cl_2$. Dicyclopentadiene was cracked,[11] and 25 mL of cyclopentadiene was added to the dark orange solution, which then was allowed to stir at room temperature for 4 days. The solvents were removed at reduced pressure, and the residue was chromatographed on a 13-cm Florisil column in hexane. A brown band was eluted with ether/hexane (20:80) and was concentrated until it was slightly turbid. Crystallization at −20 °C affored 0.26 g (76%) of brown crystalline material: mp 125.5–127 °C; IR ($CH_2Cl_2$) $\nu$(CO) 1947 (vs) $cm^{-1}$; $^1H$ NMR ($CDCl_3$) $\delta$ 1.05 (s, 3, MeCCO), 1.57, 1.79, 2.00, 2.53 (m, 6, $CH_2$ protons), 1.66 (br s, 3, MeC=CH), 2.04 (br s, 2, $CH_2$ of the norbornadienyl moiety), 2.28 (s, 3, CC=CMe), 3.48 (br s, 1 H [bridgehead]), 4.32 (br s, 1 H [bridgehead]), 4.66 (s, 5, $C_5H_5$), 5.34 (m, 1, CH=CMe), 6.75 (m, 1, H), 6.94 (m, 1, H). Anal. Calcd for $C_{24}H_{27}O_3BF_2Fe$: C; 61.57; H, 5.82. Found: C, 61.59; H, 5.82.

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