

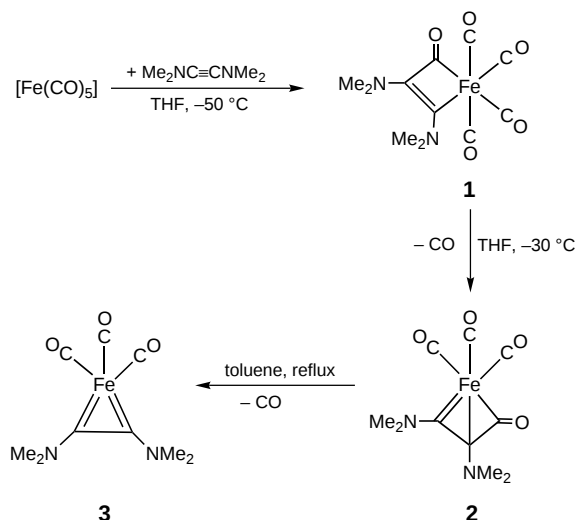
A Reaction Pathway of $[\text{Fe}(\text{CO})_5]$ with Alkynes via Ferrabicyclobutenones**

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*Dedicated to Professor Gottfried Huttner
on the occasion of his 65th birthday*

Several studies have been carried out on the reactions of $[\text{Fe}(\text{CO})_5]$ with alkynes since the pioneering work of W. Hübel.^[1] These reactions are complex and in general not selective affording a plethora of organometallic complexes and carbocycles.^[1, 2] Common products are the tricarbonyl(η^4 -cyclopentadienone)iron complexes,^[3] which have been widely used in organic synthesis.^[4] The reactions of $[\text{Fe}(\text{CO})_5]$ with alkynes follow a dissociative pathway and need photochemical or high thermal activation because of the large Fe–CO bond dissociation energy.^[5] Along this reaction pathway $[\text{Fe}(\text{CO})_4(\eta^2\text{-alkyne})]$ complexes have been isolated as thermally sensitive solids.^[6] The elementary steps for their conversion into the products remain however unclear. Following our studies on diaminoacetylene complexes^[7] we present here the first example for an associative reaction of $[\text{Fe}(\text{CO})_5]$ with $\text{Me}_2\text{N}-\text{C}\equiv\text{C}-\text{NMe}_2$ to afford a ferrabicyclobutenone and its selective C–C coupling and cleavage reactions to give a multitude of novel organoiron compounds.

Thus, treatment of $[\text{Fe}(\text{CO})_5]$ with $\text{Me}_2\text{N}-\text{C}\equiv\text{C}-\text{NMe}_2$ in THF at -50°C affords a very thermolabile intermediate, which from in situ low-temperature IR and NMR spectroscopy results is suggested to be the ferracyclobutenone **1** (Scheme 1, see Table 1). Complex **1** decarbonylates above about -30°C to give selectively the ferrabicyclobutenone **2**,



Scheme 1.

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which was isolated as an orange, thermally stable solid in 79 % yield (Scheme 1). Complex **2** loses CO either in refluxing toluene or upon melting at 124°C to afford selectively the red alkyne complex **3** in 95 % yield (Scheme 1, Table 1). Compounds **2** and **3**, are the first fully characterized members of two classes of long sought after complexes (Table 1).^[8, 9] The molecular structure of **2** reveals a distorted square-pyramidal complex, in which the C4 and C8 atoms of the ferracycle and two carbonyl ligands (C1–O1, C3–O3) occupy the basal coordination sites (Figure 1).^[10] The four-membered ferracycle is puckered (folding angle $\text{Fe}, \text{C4}, \text{C5}/\text{Fe}, \text{C5}, \text{C8} = 133.0^\circ$).

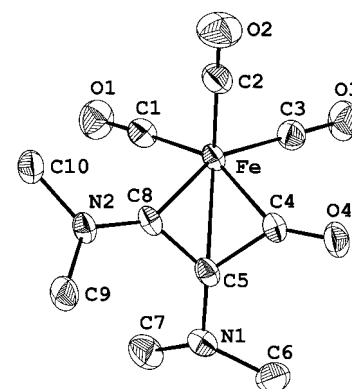


Figure 1. DIAMOND plot of the molecular structure of **2**, hydrogen atoms have been omitted (thermal ellipsoids at the 50 % probability level). Selected bond lengths [Å] and bond angles $^\circ$: Fe–C4 1.933(3), Fe–C5 2.215(2), Fe–C8 1.896(3), C4–C5 1.469(3), C5–C8 1.400(4), C4–O4 1.210(3); C8–Fe–C4 71.91(11), Fe–C4–C5 79.99(15), C4–C5–C8 103.2(2), C5–C8–Fe 82.97(16).

The Fe–C $_{\alpha}$ bond (Fe–C8 1.896(3) Å) is considerably shorter than the Fe–C $_{\beta}$ distance (Fe–C5 2.215(2) Å) and compares well with those of metallacyclic iron–carbene complexes.^[11] This situation suggests in connection with the coplanar arrangement of the atoms Fe, C8, C5, and N2, the planarity of the C $_{\alpha}$ -bonded amino group (sum of the bond angles at N2 359.6°), the short C8–N2 bond (1.306(4) Å) and the short C5–C8 bond (1.400(4) Å) an extensive π -electron delocalization over the atoms Fe, C8, N2, and C5. The C $_{\beta}$ -bonded amino group is not planar as shown by the sum of the bond angles at N1 of 343.1° , and the C5–N1 bond (1.384(3) Å) is intermediate in length between a C–N single (1.46 Å) and C–N double bond (1.27 Å).^[12] The spectroscopic data of **2** also confirm to the solid-state structure (Table 1). Thus, the IR spectrum of **2** in THF displays two characteristic bands at $\tilde{\nu} = 1733$ and 1632 cm^{-1} , which are assigned to the $\nu(\text{C}=\text{O})$ and $\nu(\text{C}_{\beta}\cdots\text{C}_{\alpha}\cdots\text{N})$ vibrations, respectively. The $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **2** (CDCl_3 , 25°C) shows a single resonance for the carbonyl ligands at $\delta = 211.4$ suggesting rapid intramolecular CO scrambling and three signals at $\delta = 60.6$, 216.4, and 223.1, which by heteronuclear multiple bond correlation (HMBC) were assigned to the β -, α -, and acyl-carbon atom of the ferrabicyclobutenone, respectively. Furthermore, the variable-temperature ^1H NMR spectra in CDCl_3 (206–298 K) show that complex **2** is fluxional because of hindered rotation of the C $_{\beta}$ -bonded amino group. The activation barrier $\Delta G^\ddagger$ for the site exchange of the methyl groups was calculated to be

9.95 kcal mol⁻¹ ($T_c = 215$ K, $\Delta\nu = 152$ Hz). In comparison, rotation of the C_a-bonded amino group is frozen even at ambient temperature giving rise to two methyl proton resonance signals at $\delta = 3.33$ and 3.59 ($\Delta G^\ddagger > 14.4$ kcal mol⁻¹).

The coordination geometry of complex **3** can be described as distorted square pyramidal with two CO ligands (C3–O1, C4–O2) and the alkyne carbon atoms occupying the basal coordination sites (Figure 2).^[10] Distortion results from the

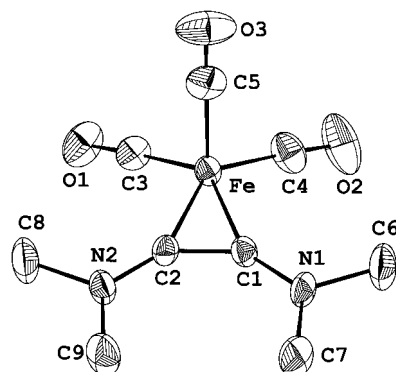
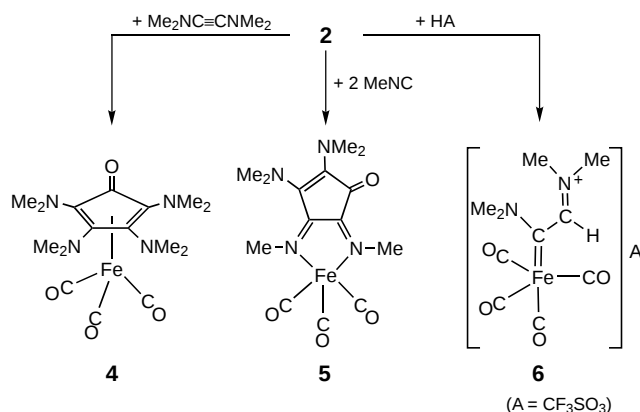


Figure 2. DIAMOND plot of the molecular structure of **3**, hydrogen atoms have been omitted (thermal ellipsoids at the 50% probability level). Selected bond lengths [Å] and bond angles [°]: Fe–C1 1.845(3), Fe–C2 1.844(3), Fe–C3 1.773(4), Fe–C4 1.785(4), Fe–C5 1.787(4), C1–C2 1.375(4), C1–N1 1.318(4), C2–N2 1.333(4); C1–Fe–C2 43.76(14), C3–Fe–C4 98.98(18), C3–Fe–C5 99.68(16), C4–Fe–C5 98.96(18), C_m–Fe–C5 114.87 (C_m denotes the midpoint of the alkyne C–C bond).

small bite angle of the alkyne ligand (43.8(1)°) and the bending of the apical carbonyl ligand (C5–O3) away from the alkyne moiety (C_m–Fe–C5 114.87°, Figure 2). The short Fe–C_{alkyne} bonds (1.844(3) and 1.845(3) Å), the long (C–C)_{alkyne} bond (1.375(4) Å), the short C_{alkyne}–N bonds (1.318(4) and 1.333(4) Å), and the planarity of the coordinated bis(dimethylamino)acetylene unit provide structural evidence for the presence of a four-electron-donor alkyne ligand with extensive π -electron delocalization.^[7] This assignment is supported by the spectroscopic data, such as the IR absorption band of the $\nu(\text{N}\equiv\text{C}\equiv\text{C}\equiv\text{N})$ vibration at 1698 cm⁻¹, the downfield-shifted resonance signal for the alkyne carbon atom at $\delta = 193.8$ ppm, and the hindered rotation of the amino groups, which gives rise to two methyl resonance signals in the ¹H and ¹³C{¹H} NMR spectra at –79 °C (Table 1).^[7] Furthermore the ¹³C{¹H} NMR spectrum of **3** (–79 °C) shows only one carbonyl-carbon resonance at $\delta = 220.5$ ppm indicating a rapid site exchange of the CO ligands on the NMR time scale (Table 1).

Complex **2** is a useful reactive starting material undergoing a variety of selective reactions with nucleophiles.^[13] Thus, treatment of **2** with Me₂N–C≡C–NMe₂ in THF at –50 °C affords selectively the yellow cyclopentadienone complex **4** (Scheme 2, Table 1),^[3d] which provides for the first time experimental evidence for the key role that ferrabicyclobutenones can play in iron-centered [2+2+1] cycloaddition reactions.^[1, 3, 4] Another example of the high reactivity of **2** is the fast reaction with methyl isocyanide (MeNC) in THF at –10 °C to yield, after double isocyanide insertion, the very air-sensitive, black 1,4-diaza-1,3-diene complex **5**, in which



Scheme 2.

Me₂N–C≡C–NMe₂, one carbon monoxide, and two isocyanide molecules have been C–C coupled to give a rare 4,5-diimino-2-cyclopentene-1-one ligand (Scheme 2, Table 1).^[14] Complex **5** adopts a coordination geometry between trigonal bipyramidal and square pyramidal, the carbonyl ligand C7–O3 being the pivot group (Figure 3).^[10] The 1,4-diaza-1,3-diene (DAD) ligand in **5** reveals similar bonding parameters to those of the few other structurally characterized [(DAD)Fe(CO)₃] complexes.^[15]

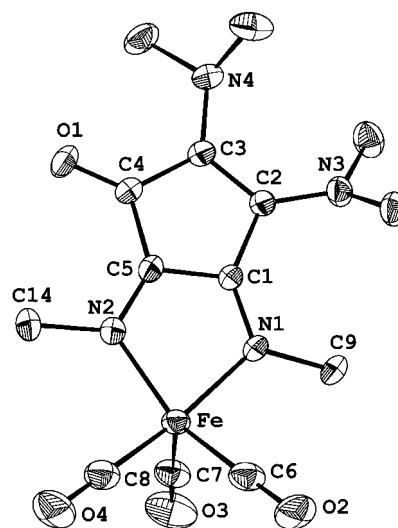


Figure 3. DIAMOND plot of the molecular structure of **5**, hydrogen atoms have been omitted (thermal ellipsoids at the 50% probability level). Selected bond lengths [Å] and bond angles [°]: Fe–N1 1.963(2), Fe–N2 1.928(2), C1–N1 1.316(3), C5–N2 1.318(3), C1–C2 1.487(3), C2–C3 1.391(3), C3–C4 1.487(3), C4–C5 1.487(3), C1–C5 1.412(3), N1–C9 1.470(3), N2–C14 1.473(3), C2–N3 1.367(3), C3–N4 1.391(3); N1–Fe–N2 81.64(9), N1–Fe–C8 165.96(10), N2–Fe–C6 147.94(11).

Complex **2** also reacts with electrophiles as shown by the C–C bond cleavage reaction with CF₃SO₃H in CH₂Cl₂ at 20 °C to afford selectively the orange-yellow aminocarbene complex **6** (Scheme 2, Table 1). Compound **6** is the first reported carbene complex bearing an electron-withdrawing iminium group at the carbene-carbon atom. An almost orthogonal orientation of the carbene-ligand plane (Figure 4; Fe, C5, N1, C8) and the plane of the iminium group

Table 1. Selected analytical data of complexes **1–6**.^[a]

[Fe(CO) ₅]: IR: $\tilde{\nu}$ = 2019 (s), 1993 cm ⁻¹ (vs) ($\nu(\text{C}\equiv\text{O})$).	
1 : IR (THF, -50 °C): $\tilde{\nu}$ = 2086 (w), 2034 (s), 2014 cm ⁻¹ (vs) ($\nu(\text{C}\equiv\text{O})$); ¹ H NMR ([D ₈]THF, -50 °C, 300.1 MHz): δ = 2.50 (s, 6H, C _β -NMe ₂), 3.39 ppm (broad s, 6H, C _α -NMe ₂); ¹³ C{ ¹ H} NMR ([D ₈]THF, -50 °C, 75.5 MHz): δ = 44.6 (C _β -NMe ₂), 46.0 (broad s, C _α -NMe ₂), 126.6 (C _β), 170.3, 172.0 (C=O, C _α), 204.7 (1 × C≡O), 208.2 (1 × C≡O), 212.8 ppm (2 × C≡O).	
2 : IR: $\tilde{\nu}$ = 2035 (vs), 1963 (vs, sh), 1955 (vs) ($\nu(\text{C}\equiv\text{O})$), 1733 (m) ($\nu(\text{C}=\text{O})$), 1632 cm ⁻¹ (m) ($\nu(\text{C}\equiv\text{C}\equiv\text{N})$); ¹ H NMR (CDCl ₃): δ = 2.08 (s, 6H, C _β -NMe ₂), 3.33 (s, 3H, C _α -NMe ₄), 3.59 ppm (s, 3H, C _α -NMe ₆); ¹³ C{ ¹ H} NMR (CDCl ₃): δ = 40.4 (C _β -NMe ₂), 43.8 (C _α -NMe ₄), 46.6 (C _α -NMe ₆), 60.6 (C _β), 211.4 (3 × C≡O), 216.4 (C _α), 223.1 ppm (C=O).	
3 : yield 95 %, red solid, m.p. 71 °C. IR: $\tilde{\nu}$ = 2011 (s), 1927 (vs) ($\nu(\text{C}\equiv\text{O})$), 1698 cm ⁻¹ (m) ($\nu(\text{N}\equiv\text{C}\equiv\text{C}\equiv\text{N})$); ¹ H NMR ([D ₈]toluene, -79 °C): δ = 1.93 (s, 6H, 2 × NMe ₄), 2.95 ppm (s, 6H, 2 × NMe ₆); ¹³ C{ ¹ H} NMR ([D ₈]toluene, -79 °C): δ = 45.1 (2 × NMe ₄), 47.1 (2 × NMe ₆), 193.8 (2 × C _{alkyne}), 220.5 ppm (3 × C≡O).	
4 : yield 89 %, yellow solid, m.p. 101 °C. IR: $\tilde{\nu}$ = 2030 (s), 1959 (vs) ($\nu(\text{C}\equiv\text{O})$), 1638 cm ⁻¹ (m) ($\nu(\text{C}=\text{O})$); ¹ H NMR (CDCl ₃): δ = 2.74 (s, 12H, 2 × NMe ₂), 2.79 ppm (s, 12H, 2 × NMe ₂); ¹³ C{ ¹ H} NMR (CDCl ₃): δ = 41.8 (2 × NMe ₂), 43.8 (2 × NMe ₂), 97.9 (2 × C-NMe ₂), 108.8 (2 × C-NMe ₂), 159.4 (C=O), 211.6 ppm (3 × C≡O).	
5 : yield 78 %, black solid, m.p. 93 °C (decomp.). IR: $\tilde{\nu}$ = 2015 (vs), 1941 (vs) ($\nu(\text{C}\equiv\text{O})$), 1670 (m), 1565 (m), 1533 (w), 1505 cm ⁻¹ (m) ($\nu(\text{C}=\text{O})$, $\nu(\text{C}=\text{C})$, $\nu(\text{C}=\text{N})$); ¹ H NMR (C ₆ D ₆): δ = 2.03 (s, 6H, NMe ₂), 2.51 (s, 6H, NMe ₂), 3.54 (s, 3H, NMe), 3.97 ppm (s, 3H, NMe); ¹³ C{ ¹ H} NMR (C ₆ D ₆): δ = 41.3 (2C, NMe ₂), 42.9 (2C, NMe ₂), 45.9 (1C, NMe), 47.3 (1C, NMe), 145.6 (1C, C _{ring}), 147.6 (1C, C _{ring}), 148.4 (1C, C _{ring}), 163.9 (1C, C _{ring}), 181.3 (1C, C _{ring}), 216.5 ppm (3 × C≡O).	
6 : yield 85 %, orange-yellow solid, m.p. 106 °C (decomp.). IR: $\tilde{\nu}$ = 2053 (s), 1984 (m), 1951 (vs), 1944sh ($\nu(\text{C}\equiv\text{O})$), 1672 (w), 1589 cm ⁻¹ (w) ($\nu(\text{C}=\text{N})$); ¹ H NMR (CD ₂ Cl ₂): δ = 3.37(s, broad, 6H, NMe ₂), 3.72 (s, 3H, NMe ₄), 3.89 (s, 3H, NMe ₆), 8.84 ppm (s, 1H, CH); ¹³ C{ ¹ H} NMR (CD ₂ Cl ₂): δ = 44.1 (very broad, NMe ₂), 48.9 (NMe ₄), 51.7 (NMe ₆), 150.0 (C(H)NMe ₂), 211.6 (4 × C≡O), 239.6 ppm (Fe=C).	

[a] Correct elemental analyses were obtained for complexes **2–6**. IR spectra were recorded with a Bruker IFS-55 spectrometer in THF at ambient temperature unless otherwise stated. ¹H and ¹³C{¹H} NMR spectra were recorded on a Bruker AM-300 spectrometer in solution at 25 °C unless otherwise stated.

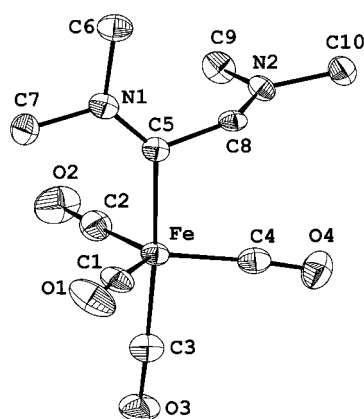


Figure 4. DIAMOND plot of the structure of the cation in **6**, hydrogen atoms have been omitted (thermal ellipsoids at the 50 % probability level). Selected bond lengths [Å] and bond angles [°]: Fe–C5 1.978(6), C5–N1 1.284(9), C5–C8 1.484(9), C8–N2 1.284(8); C5–C8–N2 123.8(6).

(C8,N2,C9,C10), is observed in the trigonal-bipyramidal complex cation (dihedral angle = 83.9°).^[10]

It is not only the unique reaction pathway of [Fe(CO)₅] with Me₂N–C≡C–NMe₂ to give the novel ferrabicyclobutenone **2**

and the experimental evidence that this type of complexes can be the key intermediate in the iron-mediated cyclization of alkynes to cyclopentadienones that merits consideration, but also the diverse reactivity of **2** which provides access to a multitude of new organoiron compounds. Compound **2** is a useful starting material for a multitude of electron-rich ferracycles, which react with electrophiles to give π -electron delocalized systems.

Experimental Section

2: A solution of [Fe(CO)₅] (2.07 g, 10.56 mmol) in THF (90 mL) was treated at -78 °C with Me₂N–C≡C–NMe₂ (0.79 g, 7.04 mmol). The mixture was allowed to warm to room temperature within 3 h and stirred for 1 h. Completion of the reaction was confirmed by IR spectroscopy. The resulting orange solution was evaporated to dryness and the dirty orange residue washed with cold pentane (0 °C; 3 × 10 mL) to remove traces of **4**. The solid was extracted with diethyl ether and the extract filtered to remove some insoluble green material. The filtrate was evaporated to dryness to give complex **2** as an orange, microcrystalline solid. Yield 1.564 g (79 % from Me₂N–C≡C–NMe₂), m.p. 124 °C (decarbonylation to **3**); elemental analysis calcd (%) for C₁₀H₁₂FeN₂O₄ (280.06): C 42.89, H 4.32, N 10.00; found: C 42.79, H 4.41, N 9.71; EI-MS (70 eV): m/z : 280 [M]⁺, 252 [M –CO]⁺, 224 [M –2CO]⁺, 196 [M –3CO]⁺, 168 [M –4CO]⁺, 112 [Me₂NC≡CNMe₂]⁺.

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- [10] Crystal structure determination of **2**: C₁₀H₁₂FeN₂O₄, M_r = 280.06; orange crystals from diethyl ether upon cooling from 20 → -78 °C, monoclinic, space group $P2_1/c$, a = 8.7893(10), b = 9.359(3), c = 15.2880(14) Å, α = γ = 90, β = 98.530(8)°, V = 1243.6(4) Å³, Z = 4, ρ_{calcd} = 1.496 g cm⁻³, T = 180(2) K, $2\theta_{\text{max}}$ = 65.8°, μ = 1.216 mm⁻¹, $F(000)$ = 576, 5571 reflections, 2180 unique reflections, 154 param-

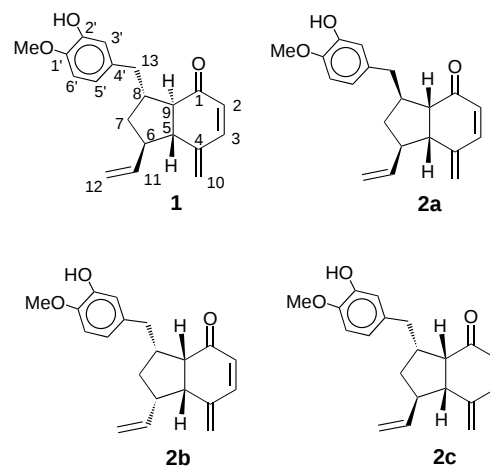
ters, GOF = 1.081, $R_1 = 0.0463$ [$I > 2\sigma(I)$], $wR_2 = 0.1350$, min./max. residual electron density $-0.989/0.599 \text{ e } \text{\AA}^{-3}$. Crystal structure determination of **3+4**: $\text{C}_9\text{H}_{12}\text{FeN}_2\text{O}_3$, $M_r = 252.05$ (**3**), $\text{C}_{16}\text{H}_{24}\text{FeN}_4\text{O}_4$, $M_r = 392.24$ (**4**); orange cocrystals of **3** and **4** (molar ratio 1/1: chromatographic work-up of the product mixture of the reaction of $[\text{Fe}_2(\text{CO})_9]$ with $\text{Me}_2\text{N}-\text{C}\equiv\text{C}-\text{NMe}_2$ containing the complexes **2**, **3**, **4** and $[\text{Fe}_2(\text{CO})_6(\mu-\text{CNMe}_2)_2]$ led to a fraction containing **3** and **4** from which the cocrystals were grown from a pentane solution upon cooling from $20 \rightarrow -30^\circ\text{C}$, triclinic, space group $P\bar{1}$, $a = 8.831(2)$, $b = 8.944(2)$, $c = 21.354(5) \text{ \AA}$, $\alpha = 97.26(3)$, $\beta = 95.31(3)$, $\gamma = 113.20(3)^\circ$, $V = 1519.0(7) \text{ \AA}^3$, $Z = 2$, $\rho_{\text{calcd}} = 1.409 \text{ g cm}^{-3}$, $T = 160(2) \text{ K}$, $2\theta_{\text{max}} = 52.48^\circ$, $\mu = 1.005 \text{ mm}^{-1}$, $F(000) = 672$, 13 771 reflections, 5573 unique reflections, 361 parameters, GOF = 1.008, $R_1 = 0.0461$ [$I > 2\sigma(I)$], $wR_2 = 0.1311$, min./max. residual electron density $-0.504/0.524 \text{ e } \text{\AA}^{-3}$. Crystal structure determination of **5**: $\text{C}_{14}\text{H}_{18}\text{FeN}_4\text{O}_4$, $M_r = 362.17$; violet crystals from pentane upon cooling from $20 \rightarrow -78^\circ\text{C}$, triclinic, space group $P\bar{1}$, $a = 8.6326(12)$, $b = 10.222(2)$, $c = 11.114(3) \text{ \AA}$, $\alpha = 113.15(2)$, $\beta = 105.499(17)$, $\gamma = 99.58(3)^\circ$, $V = 827.2(3) \text{ \AA}^3$, $Z = 2$, $\rho_{\text{calcd}} = 1.454 \text{ g cm}^{-3}$, $T = 180(2) \text{ K}$, $2\theta_{\text{max}} = 53.9^\circ$, $\mu = 0.936 \text{ mm}^{-1}$, $F(000) = 376$, 5758 reflections, 3512 unique reflections, 209 parameters, GOF = 1.068, $R_1 = 0.0451$ [$I > 2\sigma(I)$], $wR_2 = 0.1324$, min./max. residual electron density $-0.631/0.685 \text{ e } \text{\AA}^{-3}$. Crystal structure determination of **6**: $\text{C}_{11}\text{H}_{13}\text{F}_3\text{FeN}_2\text{O}_7\text{S}$, $M_r = 430.14$; yellow crystals upon diffusion of diethyl ether in THF at 20°C , triclinic, space group $P\bar{1}$, $a = 6.6594(17)$, $b = 10.490(3)$, $c = 12.988(4) \text{ \AA}$, $\alpha = 102.87(4)$, $\beta = 100.02(3)$, $\gamma = 95.78(3)^\circ$, $V = 861.8(4) \text{ \AA}^3$, $Z = 2$, $\rho_{\text{calcd}} = 1.658 \text{ g cm}^{-3}$, $T = 180(2) \text{ K}$, $2\theta_{\text{max}} = 50.48^\circ$, $\mu = 1.062 \text{ mm}^{-1}$, $F(000) = 436$, 5674 reflections, 2892 unique reflections, 226 parameters, GOF = 1.109, $R_1 = 0.0776$ [$I > 2\sigma(I)$], $wR_2 = 0.2197$, min./max. residual electron density $-0.725/1.778 \text{ e } \text{\AA}^{-3}$. Instruments: STOE STADI-4 four-circle diffractometer with scintillation counter (**2** and **5**) and STOE-IPDS diffractometer with area detector (**3** and **6**) at $\lambda(\text{MoK}\alpha) = 0.71073 \text{ \AA}$. CCDC-181665–181668 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB21EZ, UK; fax: (+44) 1223-336-033; or deposit@ccdc.cam.ac.uk).

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Total Synthesis of ($\pm$)-Otteliones A and B**

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The isolation of the two diastereomeric otteliones A and B from the widely occurring but little studied fresh water plant *Ottelia alismoides*, and the determination of their structures, which include a unique 4-methylenecyclohex-2-enone substructure, was reported in 1998.^[1] Collaborative efforts between US and Egyptian scientists, who employed high-field NMR spectroscopy techniques and modeling studies, led to the stereostructure **1** for ottelione B. However, the



structure of ottelione A could not be assigned unambiguously, and both **2a** and **2b** were considered as likely formulations, the former being more likely.^[1] In 2000, scientists at Rhône-Poulenc Rohrer reinterpreted^[2] the NMR spectroscopic data and proposed an alternate stereostructure **2c** for ottelione A (RPR 112378). Otteliones have attracted much attention as they exhibit remarkable, broad-ranging biological activity.^[1–4] Chinese scientists have reported the antitubercular effect of extracts of *Ottelia alismoides*, which is rich in otteliones, and have shown in clinical trials that two cases of bilateral tuberculosis of the cervical lymph gland were cured in three months.^[3] At the National Cancer Institute, in vitro screening against a panel of 60 human cancer cell lines showed that otteliones exhibited cytotoxicity at nM–pM levels.^[1, 4] More recent results have shown that ottelione A is an efficient inhibitor of tubulin polymerization ($\text{IC}_{50} = 1.2 \text{ }\mu\text{M}$) and is able to disassemble preformed microtubules in a manner reminiscent of the colchicines, vinblastine, and vincristine.^[2] The cytotoxicity of otteliones can be attributed to the presence of

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