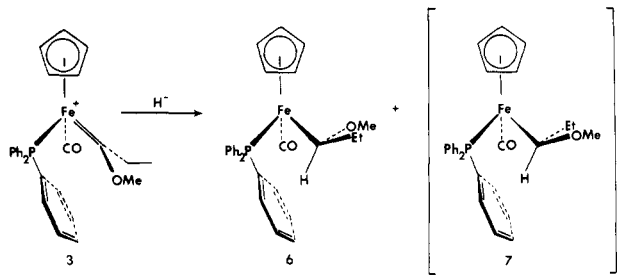
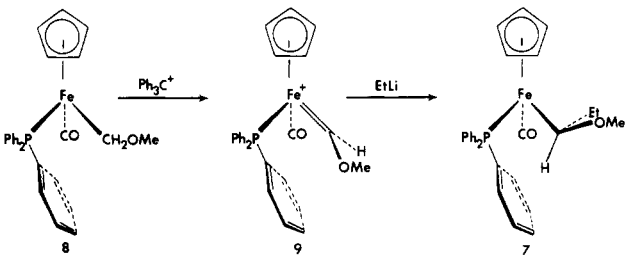
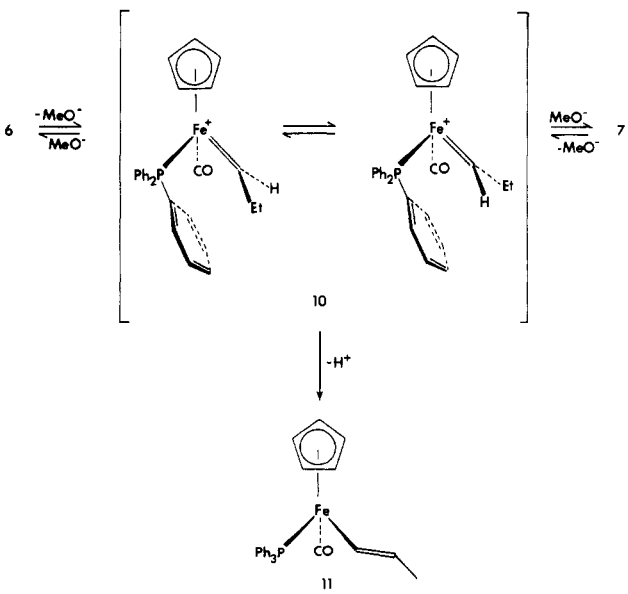
reducing agents such as K[*s*-Bu_3BH] gave only the deprotonated product **5**.[7]



Hydride abstraction from the methoxymethyl complex **8** with the trityl cation generated the carbene cation **9**.[8] Treatment of **9** with ethyllithium in dichloromethane at −78 °C again generated the diastereoisomers **6** and **7**. This time, as expected, **7** was by far the major product (**7**:**6** > 30:1) consistent with attack onto the unhindered face of **9** in the anti (O–O) conformation. Recrystallization gave pure **7**.[7]



Methanol–dichloromethane (1:2) solutions of **6** or **7** at 20 °C undergo epimerization, presumably via reversible loss of methoxide to give the carbene cation **10**. In the presence of methanol-d_4 incorporation of CD_3O into **6** and **7** was observed (CD_2Cl_2/CD_3OD, 2:1; 20 °C; 90% incorporation after 24 h). In methanol solution **6** and **7** slowly undergo methanol loss to generate the stable (*E*)-vinyl complex **11**.



Acknowledgment. We thank the SERC for studentships (G.J.B. and T.R.M.).

Registry No. **3**, 92219-90-4; **4**, 32611-01-1; (*Z*)-**5**, 91594-50-2; **6**, 83802-10-2; **7**, 83860-17-7; **9**, 69621-15-4; **11**, 92219-91-5.

(8) Cutler, A. R. *J. Am. Chem. Soc.* **1979**, *101*, 604.

Unprecedented Alkyl Migration in an Iron(II) Alkylidene[1]

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Summary: Protonation of $(\eta^5\text{-}C_5H_5)(CO)_2FeCR(1\text{-norbornyl})OC_2H_5$ (R = H or D) with HBF_4 provides $[(\eta^5\text{-}C_5H_5)(CO)_2Fe(exo\text{-}\eta^2\text{-}1\text{-R-bicyclo}[3.2.1]oct\text{-}2\text{-ene})]^+BF_4^-$ (R = H or D) in nearly quantitative yield. No $[(\eta^5\text{-}C_5H_5)(CO)_2Fe(\eta^2\text{-bicyclo}[2.2.2]oct\text{-}2\text{-ene})]^+BF_4^-$ is formed. This regioselective reaction is thought first to form an $[(\eta^5\text{-}C_5H_5)(CO)_2Fe(1\text{-norbornylmethylidene})]^+$, which undergoes an unprecedented ring enlargement to a $[(\eta^5\text{-}C_5H_5)(CO)_2Fe(\eta^2\text{-bicyclooct-1-ene})]^+$ and then rearranges to the final product.

In an effort to form and trap an unstable[2] bridgehead olefin as a π-complex, we have generated and examined the reaction of [Fp(1-norbornylmethylidene)]$^+$ [A, Fp = $(\eta^5\text{-}C_5H_5)(CO)_2Fe$] in acid solution.

Alkylation[3] of the Fp–acyl, **1**,[4] with $Et_3O^+BF_4^-$ in CH_2Cl_2 at 25 °C gives the yellow ethoxycarbene salt **2**[5] in >90% yield. Reduction of **2** in CH_2Cl_2 at −78 °C with a 1 M solution of $LiEt_3BH$ in THF[6] produces the ethoxyalkyl complex **3**[7] as a yellow oil in 65% yield. Protonation of **3** in CH_2Cl_2 at 25 °C with a slight excess of HBF_4 in $Ac_2O/HOAc/CH_2Cl_2$,[8] concentration, and dilution with Et_2O gives an essentially quantitative yield of a yellow, crystalline Fp(η^2-bicyclooctene)$^+BF_4^-$ identical with that[9]

(1) Presented before the 188th National Meeting of the American Chemical Society, Philadelphia, PA, Aug 1984; American Chemical Society: Washington, DC, 1984; Abstract INOR 79.

(2) Maier, W. F.; Schleyer, P. von R. *J. Am. Chem. Soc.* **1981**, *103*, 1891–1900.

(3) Dry, oxygen-free solvents and Schlenk techniques were employed throughout.

(4) **1**: IR (CH_2Cl_2) 2005, 1955, 1640 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 4.80 (s, 5 H, Cp), 2.21 (b s, 1 H, >CH), 1.49 (m, 10 H, 5 >CH_2); $^{13}C\{^1H\}$ NMR ($CDCl_3$) δ 261.6 (>C=O), 215.0 (–C≡O), 86.1 (Cp), 75.3 (C_1), 41.9 (C_4), 35.8 (C_7), 32.3 (C_2, C_6), 29.6 (C_3, C_5); mp 97–99 °C. Anal. Calcd for $C_{15}H_{16}FeO_3$: C, 60.03; H, 5.37. Found: C, 59.91; H, 5.41. We thank John Lever for the original preparation of this compound.

(5) **2**: IR (CH_2Cl_2) 2057, 2010 cm^{-1}; ^{1}H NMR (CD_2Cl_2, −10 °C) δ 5.42 (s, 5 H, Cp), 5.30 (q, J = 7.5 Hz, 2 H, OCH_2CH_3), 2.40 (b s, 1 H, >CH), 1.90–1.32 (b m, 13 H, CH_3 + 5 >CH_2); $^{13}C\{^1H\}$ NMR (CD_2Cl_2, −10 °C) δ 342.9 (C_8), 209.4 (–C≡O), 88.2 (Cp), 83.9 (C_9), 78.7 (C_1), 44.7 (C_4), 37.3 (C_7), 35.4 (C_2, C_6), 30.0 (C_3, C_5), 14.7 (C_{10}).

(6) Bodnar, T.; Cutler, A. *J. Organomet. Chem.* **1981**, *213* C31–C36.

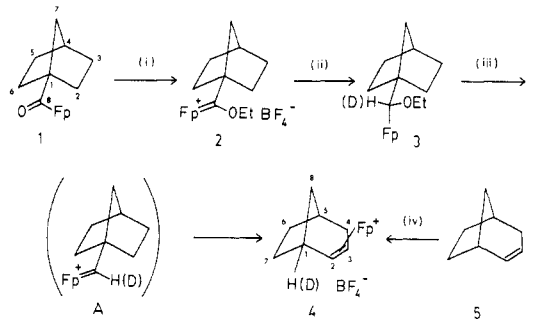
(7) **3**: (a) IR (CH_2Cl_2) 1995, 1937 cm^{-1}; ^{1}H NMR ($CDCl_3$, −10 °C) δ 5.10 (s, 1 H, FpC*H*(OR)C<), 4.80 (s, 5 H, Cp), 3.57 (d of q, $J_{ac} \approx$ 6.6 Hz, $J_{ab} \approx$ 2 Hz, 1 H, >C*OC*H*^{a}H^{b}CH$_3^c$), 3.26 (d of q, $J_{bc} \approx$ 6.6 Hz, $J_{ba} \approx$ 2 Hz, 1 H, >C*OCH^a*H*^{b}CH$_3^c$), 2.11 (b s, 1 H, >CH), ~1.9–0.9 (b m, ~10 H, 5 >CH_2) superimposed upon a triplet at 1.07, $J_{cb} \approx J_{ca}$ = 6.6 Hz, ~3 H, CH_3); $^{13}C\{^1H\}$ NMR ($CDCl_3$, −20 °C)[7b] δ 218.9, 216.8 (–C≡O), 85.9 (Cp), 85.3 (C_8), 65.8 (C_9), 61.6 (C_1), 45.1 (C_7), 36.6, 35.1 (C_2, C_6), 32.2 (C_4), 31.1, 30.4(C_3, C_5), 15.4 (C_{10}); MS m/e 330 [M]$^+$·, 302 [M − CO]$^+$·, 274 [M − 2CO]$^+$·, 153 [M − Fp]$^+$·. (b) These ^{13}C assignments were made by using the refocused "insensitive nucleus enhancement through polarization transfer" (INEPT) technique [Morris, G. A.; Freeman, R. *J. Am. Chem. Soc.* **1979**, *101*, 760–762].

(8) (a) Jolly, P. W.; Pettit, R. *J. Am. Chem. Soc.* **1966**, *88*, 5044–5045. (b) Green, M. L. H.; Ishaq, M.; Whiteley, R. N. *J. Chem. Soc. A* **1967**, 1508–1515.

(9) **4**: IR (CH_2Cl_2) 2079, 2038 cm^{-1}; ^{1}H NMR (CD_2Cl_2) δ 5.50 [b s, ~5 H, Cp superimposed on an ~1 H multiplet at ~5.4–5.5 due to Fp$^+$(η^2-=C(3)*H*CH$_2$–)], 5.30 [s, 1 H, Fp$^+$(η^2-C(2)*H*=)], 2.73 (m, 1 H, >C(1)*H*), 2.48 (perturbed d, 1 H, =CHC(4)*H*(endo)HCH<), 2.05–2.25 (m, 3 H, >C(5)*H* superimposed upon –C(6)H*H*(exo)C(7)H*H*(exo)–), ~1.86 (m, 2 H, –C(6)*H*(endo)HC(7)(endo)H–), 1.45 (m, 1 H, =CHC(4)H*H*(exo)CH<), 1.39 (br, perturbed doublet, $J \approx$ ' 16 Hz, 1 H, >C(8)*H*(anti)H), 0.58 (d, J = 16 Hz, 1 H, >C(8)H*H*(syn); $^{13}C\{^1H\}$ N 1 R (CD_2Cl_2, −10 °C)[7b] δ 211.1, 210.8 (–C≡O), 89.1 (Cp), 86.6 (C_2), 76.4 (C_3), 38.8 (C_1), 35.1 (C_8), 33.4 (C_4), 32.5 (C_5), 32.3, 30.5 (C_6, C_7); mp 92.5–93.5 °C dec. Anal. Calcd. for $C_{15}H_{17}O_2FeBF_4$: C, 48.44; H, 4.61. Found: 48.09, H, 4.66.

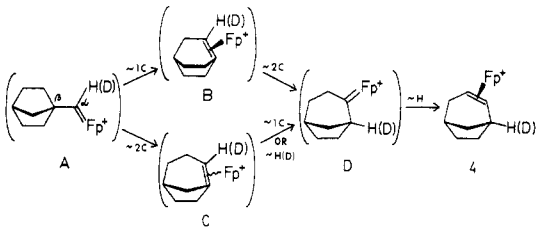
0276-7333/84/2303-1765$01.50/0

Scheme I



(i) $Et_3O^+BF_4^-$, CH_2Cl_2, 25 °C; (ii) $LiEt_3BH(D)$, CH_2Cl_2, −78 °C; (iii) HBF_4, $Ac_2O/HOAc/CH_2Cl_2$, 25 °C; (iv) $FpBF_4$, CH_2Cl_2, 25 °C

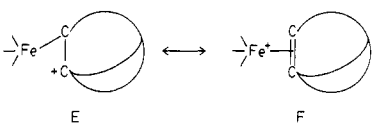
Scheme II



formed from the reaction of bicyclo[3.2.1]oct-2-ene (**5**) and $FpBF_4$ in CH_2Cl_2 at 25 °C (Scheme I).[10] From the known steric bias of bicyclo[3.2.1]oct-2-enes for exo addition[11] and a comparison of the 400-MHz ^1H NMR spectra of **4**[9] and **5**[12] with those of norbornene[13] and its *exo*-η^2-Fp^+ complex,[14] we represent the complex as the exo diastereomer **4**.[15] There is no evidence of any other π-complex or its reaction product(s)[16] in the reaction mixture. When **2** is reduced instead with $LiEt_3BD$, ^1H and ^{13}C NMR spectra indicate that the rearrangement occurs without significant loss of label to produce a product, **4**, which is deuterated exclusively at C(1).

While details of the regioselective rearrangement are not yet clear, the logistics are—the initial alkylidene shifts an ethano or methano bridge to form a complexed, bridgehead bicyclooctene that then isomerizes to a single π-complex. Our present data permit no distinction among the possibilities outlined in Scheme II.

The rearrangement of a transition-metal alkylidene to a π-complex by the shift of a β-alkyl has not been previously reported.[17] Three factors should favor it here: the carbenoid carbon is especially electrophilic,[18] there are no β-hydrogens to shift in preference to carbon,[6,19] and it relieves some of the strain associated with the norbornyl substituent.[2,20] If substantial positive charge develops at the probridgehead carbon during the ring enlargement (cf. E), methano bridge migration (~1 C) should be favored[21] for the bridgehead bicyclo[2.2.2]octyl cation is 1–3 kcal/mol more stable than the [3.2.1].[22] If back-bonding is substantial so that the charge remains primarily on iron (cf. F) ethano migration (~2 C) is expected and the rearrangement product should be π-complexed (*Z*)-bicyclo[3.2.1]oct-1-ene as this olefin is much more stable than either the *E* isomer or bicyclo[2.2.2]oct-1-ene.[2]



The rearrangement of the intermediate bridgehead π-complex B and/or C to product does not follow either of the paths suggested earlier for the Pt(0)- or Pd(II)-catalyzed bridgehead-to-nonbridgehead isomerizations of bicyclononenes.[23] Our label study rules out the transfer of an allylic hydrogen to the bridgehead or the substantial dilution of label that these mechanisms would entail. Instead we suggest that the rearrangement occurs by way of a second alkylidene (Scheme II)—an intermediate not formed in the Pt(0) or Pd(II) cases.[23] Normally, such isomerizations are from alkylidene to π-complex[6,19,24] but here the inherent strain of the bridgehead olefin[2] and

(10) Reger, D. L.; Coleman, C. J.; McElligott, P. J. *J. Organomet. Chem.* **1979**, *171*, 73–84.

(11) (a) Sauers, R. R.; How, H. M.; Feilich, H. *Tetrahedron* **1965**, *21*, 983–987. (b) Goering, H. L.; Kantner, S. S. *J. Org. Chem.* **1984**, *49*, 422–426.

(12) **5**: ^1H NMR (CD_2Cl_2) δ ~5.9 (perturbed t, 1 H, =C*H*CH_2–) ~5.4 (m, 1 H, >CHC*H*= superimposed upon a $CHDCl_2$ solvent peak), 2.3–2.45 (m, 3 H, >C(1)*H* superimposed upon =CHC(4)*HH*CH<), 1.8–1.95 (complex m, 3 H, >C(5)H + >C(8)*HH*), 1.6–1.8 [complex m, 2 H, –C(6)H*H*-(exo)C(7)H*H*(exo)–], 1.4–1.6 [complex m, 2 H, –C(6)*H*(endo)HC(7)*H*-(endo)H–].

(13) Cf. Jackman, L. M.; Sternhell, S. "Applications of Nuclear Magnetic Resonance Spectroscopy in Organic Chemistry", 2nd ed.; Pergamon Press: Oxford, 1969; pp 229–233.

(14) Cutler, A.; Ehntholt, D.; Giering, W. P.; Lennon, P.; Raghu, S.; Rosan, A.; Rosenblum, M.; Tancrede, J.; Wells, D. *J. Am. Chem. Soc.* **1976**, *98*, 3495–3507.

(15) The strong deshielding of a single C(8) hydrogen ($\Delta\delta \approx -0.5$ ppm) and a single C(4) hydrogen ($\Delta\delta \approx -0.85$) that occurs when **5** is converted to **4** (Scheme I) suggests that it is exo as shown, cf. ref 13 and 14.

(16) (a) Grootveld, H. H.; Blomberg, C.; Bickelhaupt, F. *J. Chem. Soc., Chem. Commun.* **1973**, 542–543. (b) Wolf, A. D.; Jones, M., Jr. *J. Am. Chem. Soc.* **1973**, *95*, 8209–8210. (c) Warner, P.; LaRose, R.; Lee, C.-m; Clardy, J. C. *J. Am. Chem. Soc.* **1972**, *94*, 7607–7609. (d) Warner, P. M.; LaRose, R. C.; Palmer, R. F.; Lee, C.-m.; Ross, D. O.; Clardy, J. C. *J. Am. Chem. Soc.* **1975**, *97*, 5507–5512.

(17) Some alkyl migrations that occur during the metal-catalyzed valence isomerization of certain strained polycyclic hydrocarbons may be of this type (cf. (a) Gassman, P. G.; Atkins, T. J.; Williams, F. J. *J. Am. Chem. Soc.* **1971**, *93*, 1812–1813. (b) Gassman, P. G.; Atkins, T. J. *J. Am. Chem. Soc.*, **1971**, *93*, 4597–4599. (c) Paquette, L. A.; Zon, G. *J. Am. Chem. Soc.* **1974**, *96*, 203–215), but alternate paths are sometimes possible,[17a,b] there is some uncertainty about the nature of the actual intermediate (cf. (d) Cardin, D. J.; Cetinkaya, B.; Doyle, M. J.; Lappert, M. F. *Chem. Soc. Rev.* **1973**, *2*, 99–144. (e) Bishop, K. C., III *Chem. Rev.* **1976**, *76*, 461–486), and a π-complex is not the final product.

(18) The complex is cationic; the alkylidene carbon is bonded to a late transition metal unstabilized by strongly donating phosphine ligands. See: Casey, C. P. In "Reactive Intermediates"; Jones, M., Jr., Moss, R. A., Eds.; Wiley: New York, 1981; Vol. II, pp 135–174 for a discussion.

(19) (a) Cutler, A.; Fish, R. W.; Giering, W. P.; Rosenblum, M. *J. Am. Chem. Soc.* **1972**, *94*, 4354–4355. (b) Labinger, J. A.; Schwartz, J. *J. Am. Chem. Soc.* **1974**, *97*, 1596–1598. (c) Brookhart, M.; Tucker, J. R.; Husk, G. R. *J. Am. Chem. Soc.* **1983**, *105*, 258–264.

(20) Methyl migration is not observed in $[(\eta^5\text{-}C_5H_5)(\eta^2\text{-}Ph_2PCH_2CH_2PPh_2)Fe{=}CHC(CH_3)_3]^+$ probably because of stabilization by the "diphos" ligand[18] but the absence of notable steric strain may also be a factor [Davison, A.; Selegue, J. P. *J. Am. Chem. Soc.* **1980**, *102*, 2455–2456].

(21) (a) Wiberg, K. B.; Lowry, B. R. *J. Am. Chem. Soc.* **1963**, *85*, 3188–3193. (b) Wilt, J. W.; Schneider, C. A.; Dabek, H. F., Jr.; Kramer, J. F.; Wagner, W. J. *J. Org. Chem.* **1966**, *31*, 1543–1551. (c) Wilt, J. W.; Parsons, C. F.; Schneider, C. A.; Schultenover, D. G.; Wagner, S. J.; Wagner, W. J. *J. Org. Chem.* **1968**, *33*, 694–708. (d) Chenier, P. J.; Kiland, P. J.; Schmitt, G. D.; VanderWegen, P. G. *J. Org. Chem.* **1980**, *45*, 5413–5417.

(22) (a) Bingham, R. C.; Schleyer, P. von R. *J. Am. Chem. Soc.* **1971**, *93*, 3189–3199. (b) Bly, R. S.; Quinn, E. K. "Abstracts of Papers", 153rd National Meeting of the American Chemical Society, Miami Beach, FL, April 1967; American Chemical Society: Washington, DC, 1967; No. 0-91. (c) See also, Sauers, R. R.; Ahlstrom, D. H. *J. Org. Chem.* **1967**, *32*, 2233–2236.

(23) (a) Stamm, E.; Becker, K. B.; Engel, P.; Keese, R. *Helv. Chim. Acta* **1979**, *62*, 2181–2185. (b) Godleski, S. A.; Valpey, R. S.; Grundlach, K. B. *Organometallics* **1983**, *2*, 1254–1257.

(24) (a) Casey, C. P.; Albin, L. D.; Burkhardt, T. J. *J. Am. Chem. Soc.* **1977**, *99*, 2533–2539. (b) Bates, D. J.; Rosenblum, M.; Samuels, S. B. *J. Organomet. Chem.* **1981**, *209*, C55–C59. (c) Casey, C. P.; Miles, W. H.; Tukada, H.; O'Connor, J. M. *J. Am. Chem. Soc.* **1982**, *104*, 3761–3762. (d) Miller, G. A.; Copper, N. J. "Abstracts of Papers" 187th National Meeting of the American Chemical Society, St. Louis, MO, April 1984; American Chemical Society: Washington, DC, 1984; INOR 188.

probable unsymmetric nature of the π-complex[25] may render the converse true.[26]

We continue to investigate the mechanism, generality and synthetic utility of this new regioselective rearrangement.

Acknowledgment. R.S.B. thanks Professor Charles P. Casey for helpful discussions and suggestions and the NSF for partial support under its EPSCOR program.

Registry No. 1, 92471-94-8; 2, 92471-96-0; 3, 92471-97-1; 3 deuterated isomer, 92471-98-2; 4, 92472-00-9; 4 deuterated isomer, 92472-02-1; 5, 823-02-9.

(25) (a) Kerber, R. C.; Ehntholt, D. J. *J. Am. Chem. Soc.* **1973**, *95*, 2927–2934. (b) Chang, T. C. T.; Foxman, B. M.; Rosenblum, M.; Stockman, C. *J. Am. Chem. Soc.* **1981**, *103*, 7361–7362.

(26) Eisenstein, O.; Hoffman, R. *J. Am. Chem. Soc.* **1980**, *102*, 6149–6151.

Novel Catalytic and Stoichiometric Approaches to Azetidine-2,4-diones from α-Lactams Using Rhodium and Cobalt Complexes, Respectively

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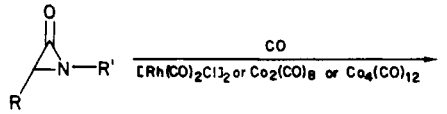
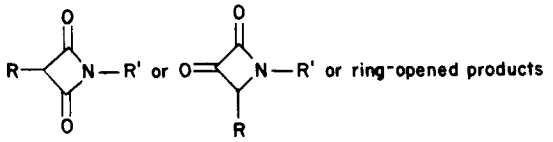
Summary: Although α-lactams are regiospecifically converted to azetidine-2,4-diones in fine yields with use of rhodium(I) [e.g., $[Rh(CO)_2Cl]_2$] and cobalt(0) [e.g., $Co_2(CO)_8$] complexes, the two processes are significantly different: the rhodium reaction occurs under carbon monoxide and is catalytic; the cobalt reaction is inhibited by carbon monoxide and is not catalytic.

Transition-metal organometallics are useful reagents and catalysts for the synthesis and manipulation of heterocyclic nitrogen compounds.[1-3] Recently, one of us reported the first example of ring expansion and carbonylation of an aziridine to a β-lactam, using chlorodicarbonylrhodium(I) dimer as the catalyst.[4] What would be the consequence of a significant structural change in the aziridine moiety? α-Lactams (aziridinones) (1) are fascinating strained ring



1



2 3

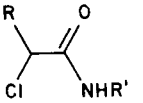
systems[5] that are easy to synthesize[6] and undergo some interesting organic transformations.[5,7] If one were to attempt to carbonylate such a reactant, then ring expansion or ring cleavage may take place. In the event that the four-membered ring compound is formed, then the question arises as to whether the azetidine-2,4-dione(2) or azetidine-2,3-dione (3) or both will be produced in the metal(I)-catalyzed reactions. While some elegant work has been published on carbonylation reactions involving amides as reactants (i.e., amidocarbonylation),[8,9] there have been no publications on the carbonylation of α-lactams. This communication describes the regiospecific, catalytic rhodium(I) and the stoichiometric cobalt(0), ring expansion of α-lactams. Although the products are the same in both cases, there are fundamental differences using the two types of metal complexes.

Table I. Yields of Azetidine-2,4-diones Obtained from the Rhodium(I)-Catalyzed or the Cobalt(0)-Induced Carbonylation of 1[a]

2, R =, R′ =	ML_n	yield,[b] %	mp, °C
$(CH_3)_3C$, $(CH_3)_3C$	$[Rh(CO)_2Cl]_2$	100	38–40
	[1,5-HDRhCl]$_2$	70	
	[1,5-CODRhCl]$_2$	75	
	$Co_2(CO)_8$	90	
	$Co_4(CO)_{12}$	84	
$(CH_3)_3C$, 1-adamantyl	$[Rh(CO)_2Cl]_2$	51	75–85
	$Co_2(CO)_8$	51	
1-adamantyl, $(CH_3)_3C$	$[Rh(CO)_2Cl]_2$	90	68–70
1-adamantyl, 1-adamantyl	$[Rh(CO)_2Cl]_2$	80	200–204
	$Co_2(CO)_8$	95	
	$Co_4(CO)_{12}$	100	

[a] Satisfactory C, H, N analyses were obtained in all cases. [b] Yields are of pure materials.

When 1,3-di-*tert*-butylaziridinone [1, R = R′ = $(CH_3)_3C$][6] was reacted with carbon monoxide and a catalytic amount of chlorodicarbonylrhodium(I) dimer [10:1 ratio of 1/Rh(I)] in dry benzene at 40 °C and 30 atm (overnight), the azetidine-2,4-dione [2, R = R′ = $(CH_3)_3C$] was obtained in quantitative yield. This transformation can also be achieved at atmospheric pressure, but it is a slower reaction. Other rhodium(I) catalysts can be used including the dimers of chloro(1,5-hexadiene)rhodium(I) and chloro(1,5-cyclooctadiene)rhodium(I); however, the rhodium(0) complex $Rh_6(CO)_{16}$ and tetrakis(triphenylphosphine)palladium(0) were inert. The use of dry benzene must be stressed, since the presence of any water results in conversion of some of the α-lactam to the chloro amide 4.[10]



4

Fine yields of azetidine-2,4-diones (2) were obtained by using a number of other α-lactams as substrates. The

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(10) A very acidic solution (pH 2) is formed on treatment of $[Rh(CO)_2Cl]_2$ with water (or wet benzene). It is known that α-chloro amides are obtained on reaction of α-lactams with hydrochloric acid.[2,4] If one assumes that HCl is the acidic species formed when the rhodium catalyst reacts with water, the acid would convert 1 to 4 [the use of stoichiometric quantities of the rhodium(I) complex results in complete conversion of 1 to 4]. The presence of water in effect poisons the rhodium(I) catalyst.

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