Table II. Stabilization of Various Formylmetal Complexes by n-Bu_3SnH[a]

formyl complex	yield,[b] %	Bu_3SnH, equiv	^1H NMR[c]	τ,[d] min	decomposition products (% yield)
$Cr(CO)_5CHO^-K^+$	70		15.2	39	$Cr(CO)_5H^-$[e] (10), μ-H-$[Cr(CO)_5]_2^-$[f] (17)
$Cr(CO)_5CHO^-K^+$	92	0.1	15.1	165	$Cr(CO)_5H^-$ (16)
$Cr(CO)_5CHO^-K^+$	90	1	15.1	170	$Cr(CO)_5H^-$ (14)
$Cr(CO)_5CHO^-K^+$	86	2	15.1	172	$Cr(CO)_5H^-$ (35)
$W(CO)_5CHO^-K^+$	74		15.9	16	μ-H-$[W(CO)_5]_2^-$[g] (17)
$W(CO)_5CHO^-K^+$	76	1	15.8	28	$W(CO)_5H^-$[h] (50)
$W(CO)_5CHO^-K^+$	76	2	15.8	60	$W(CO)_5H^-$ (48)
$(CH_3CO)Re(CO)_4CHO^-K^+$	87	2	15.7	78	$(CH_3CO)Re(CO)_4H^-$[n] (32)
$Re(CO)_4BrCHO^-Li^+$[m]	58	1	14.95	130	*i*
$Mn(CO)_3(PPh_3)_2CHO$[j]	77	2	13.45	30	$HMn(CO)_3(PPh_3)_2$[k] (15)
$Mo(CO)_5CHO^-K^+$[l]	40	2	15.4	<10	*i*
$Mn_2(CO)_9CHO^-Li^+$	p	2			*i*

[a] The formylmetal complexes were formed either by the addition of $K(i\text{-}PrO)_3BH$ (1 M in THF) or $Li(C_2H_5)_3BH$ (1 M in THF) to a 1.0×10^{-1} M solution of the parent metal carbonyl and the appropriate amount of n-Bu_3SnH in THF. Experiments performed in degassed, sealed NMR tubes. [b] The yields refer to the conversion of the metal carbonyl to the formylmetal complex. [c] Formyl resonance relative to Me_4Si. [d] Apparent half-life; see footnote 5. [e] Identified by its characteristic ^1H NMR resonance, −7.0 ppm.[6] [f] Identified by its characteristic ^1H NMR resonance, −19.5 ppm.[6] [g] Identified by its characteristic ^1H NMR resonance, −12.5 ppm.[6] [h] Identified by its characteristic ^1H NMR resonance, −4.5 ppm.[6] [i] No hydride resonances could be observed between 0 and −30.0 ppm. [j] The formyl complex was formed by the addition of a slight excess of $Li(C_2H_5)_3BH$ (1 M in THF) to 2×10^{-2} M $Mn(CO)_4(PPh_3)_2^+ PF_6^-$ in CH_2Cl_2. [k] Identified by its characteristic ^1H NMR spectrum (−7.2 ppm, t, J = 33 Hz). [l] In the absence of n-Bu_3SnH, the formyl complex was formed in 17% yield. [m] In the absence of n-Bu_3SnH, $Re(CO)_5H$ (−5.2 ppm) was formed exclusively in 85% yield. [n] Identified by its characteristic ^1H NMR spectrum (−4.7 ppm).[12] In the absence of Bu_3SnH, the hydride was formed in 94% yield. [p] Not stable at room temperature.

Further addition of the tin hydride had no effect on the half-life, in striking accord with the saturation limit described in Figure 1.

The efficacy of the tin hydride to effectively inhibit the radical-chain process, we believe, has an important bearing on the role of other metal hydrides to act in a similar capacity in *catalytic* systems. Accordingly we examined the stabilization by tributyltin hydride of a variety of other formylmetal complexes presented in Table II. Thus the formyltungsten complex $W(CO)_5CHO^-$ can be generated in 74% yield,[7] and it has an apparent half-life of 16 min at 25 °C to afford the μ-hydride $(OC)_5WHW(CO)_5^-$ as the principal decomposition product. In the presence of n-Bu_3SnH, the half-life of $W(CO)_5CHO^-$ is prolonged substantially, and the principal product is the tungsten hydride $W(CO)_5H^-$, as given in Table II. Similarly the half-life of the mixed formylacetylrhenium complex[12] $CH_3CORe(CO)_4CHO^-$ can be increased from 8 min to 78 min in the presence of 2 equiv of n-Bu_3SnH. Even haloformylrhenium complexes such as $BrRe(CO)_4CHO^-$, which are unstable at room temperature,[13] could be stabilized by 2 equiv of n-Bu_3SnH (τ 130 min at 25 °C). Likewise, the neutral formyl complex $Mn(CO)_3(PPh_3)_2CHO$,[14] which decomposes readily at 0 °C, could be seen at room temperature (τ 30 min) in the presence of 2 equiv of n-Bu_3SnH.

It is noteworthy that there are some formylmetal complexes such as $(OC)_5MnMn(CO)_4CHO^-$[15] and $Mo(CO)_5CHO^-$[7] whose stabilities are not noticeably affected by the presence of n-Bu_3SnH. Since these formyl complexes do not afford the corresponding hydrides as decomposition products, we infer that the radical-chain mechanism is mainly applicable to those formylmetal complexes which are readily converted to their hydride derivatives. However there are other formylmetal complexes such as $CH_3CORe(CO)_4CHO^-$ and $BrRe(CO)_4CHO^-$ (vide supra), which are stabilized but afford the corresponding carbonylmetal hydrides in considerably reduced yields in the presence of n-Bu_3SnH. Clearly the isolation and identification of the products formed under these conditions will provide important insights into the nonchain processes for the decomposition of formylmetal species.

Our results thus indicate that a multiplicity of pathways are available for the reaction of formylmetal intermediates. In particular we have identified the existence of additional nonchain pathways by which formylmetal intermediates can disappear.[16] The delineation of these molecular processes (with the aid of retarders described in this study) will doubtlessly lead to additional insight into the role of formylmetal intermediates in the catalytic reduction of carbon monoxide.

Acknowledgment. We wish to thank the National Science Foundation and the United States–France (NSF–CNRS) cooperative program for financial support.

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(13) See ref 12. Note that $BrRe(CO)_4CHO^-$ has been stabilized by the addition of LiBr. The decomposition products will be reported at a later time.
(14) Tam, W.; Lin, G. Y.; Gladysz, J. A. *Organometallics* **1982**, *1*, 525.
(15) Tam, W.; Marsi, M.; Gladysz, J. A. *Inorg. Chem.* **1983**, *22*, 1413.
(16) See, e.g., ref 1e and: Davies, S. G.; Hibberd, J.; Simpson, S. J. *J. Chem. Soc., Chem. Commun.* **1982**, 1404. Compare also: Lin, Y. C.; Milstein, D.; Wreford, S. S. *Organometallics* **1983**, *2*, 1461.

(*R*,*R*)-2,3-Butanediol as Chiral Directing Group in the Synthesis of (α*S*)-α-Chloro Boronic Esters

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Summary: (*R*,*R*)-2,3-Butanediol dichloromethaneboronate is a convenient reagent for attaching a chiral carbon to a Grignard or lithium reagent via zinc chloride catalyzed rearrangment of the intermediate borate complex, which yields (α*S*)-α-chloro boronic esters with

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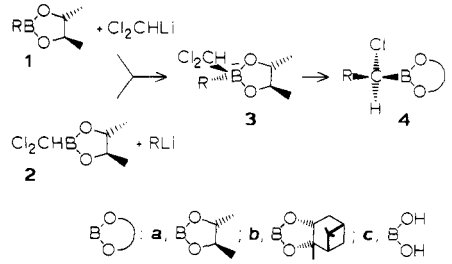
91–96% diastereoselectivity. The esters are easily hydrolyzed to crystalline boronic acids.

Homologation of (+)-pinanediol boronic esters by lithiodichloromethane with zinc chloride catalysis of the rearrangement of the intermediate borate salt has yielded (αS)-α-chloro boronic esters with diastereoselectivities generally >99%.[1] An (*R,R*)-2,3-butanediol acetal has shown 92% diastereoselectivity in a cationic olefin cyclization.[2] We have now found that homologations of (*R,R*)-2,3-butanediol boronic esters (**1**) to (αS)-α-chloro boronic esters (**4a**) readily yield diastereoselections in the 91–96% range and present certain other advantages that offset the lower initial chiral purity.



The C_2 symmetry of the butanediol is significant because the intermediate borate complex **3** is identical whether formed from a boronic ester **1** and lithiodichloromethane or from (*R,R*)-2,3-butanediol dichloromethaneboronate (**2**) and an alkyllithium. Thus, **2** is a convenient reagent for adding a chiral carbon to any common lithium or Grignard reagent,[3] and the resulting (αS)-α-chloro boronic esters (**4**) are versatile chiral synthetic intermediates.[1,4-6] It is advantageous that (*R,R*)-2,3-butanediol is commercially available[7,8] and that its boronic esters, in contrast to the exceedingly refractory esters of pinanediol (**4b**),[4] hydrolyze readily on contact with water to form crystalline boronic acids (**4c**).

To measure diastereomer ratios, the 2,3-butanediol α-chloroboronates (**4a**) were transesterified to **4b** with (+)-pinanediol[4] (100% ee) and the 200-MHz NMR spectra were examined, especially near δ 1.0–1.2 where one of the pinanediol protons often differentiates diastereomers.[1,9] The spectra of both epimers were already known[1,5] except for R = *i*-Pr, for which the (−)-pinanediol ester was also made. Estimated diastereomeric purities (±2%) are recorded in Table I.

Table I. Diastereoselectivities in Rearrangements of (*R,R*)-2,3-Butanediol (Dichloromethyl)(organyl)borates (**3**) to (αS)-α-Chloro Boronic Esters (**4**) in the Presence of Zinc Chloride

R	route[a]	yield, %	% αS[b]	chem shifts, δ proton[c]	αS	αR
Me	C	90	95	pinyl	1.171	1.157
i-Pr	A		91	BCHCl	3.83	3.31
i-Pr	B	88	96	BCHCl	3.33	3.31
Bu	A	80	95	NH[d]	9.40	9.06
Bu	C	92	96	pinyl	1.182	1.173
Ph	B	94	95	pinyl	1.141	1.029
$PhCH_2$	B	81	95	BCHCl	3.652	3.634

[a] A: $R-BO_2(CHMe)_2$ + $LiCHCl_2$. B: $Cl_2CH-BO_2(CHMe)_2$ + RMgX. C: $Cl_2CH-BO_2(CHMe)_2$ + RLi. [b] Based on 200-MHz NMR of (+)-pinanediol ester derived from crude 2,3-butanediol ester. [c] "Pinyl" refers to unobstructed doublet, J = 11 Hz, which appears in the NMR of all pinanediol esters and usually differs for diastereomers. [d] The pinanediol ester of the α-acetamido derivative was prepared, mp 140–141 °C.[17] **4a**, R = Bu, was also treated with methyllithium and oxidized to (*S*)-2-hexanol,[4] $[\alpha]^{22}_D$ +9.21° (EtOH). This contained 6.5% butanol (from **1**) by GC analysis, and therefore the ee is 90%, diastereoselectivity 95%.

2,3-Butanediol partitions preferentially into water and the α-chloro boronic acids (**4c**) into ether, from which they can be crystallized by partial concentration and addition of hexane. (*S*)-1-Chloro-2-phenylethaneboronic acid (**4c**, R = $PhCH_2$) after one recrystallization and esterification to **4b** with pinanediol showed no (>0.5%) *R* isomer by 200-MHz NMR analysis.

Epimerization[9] of the relatively unhindered 2,3-butanediol α-chloroboronates (**4a**) does not limit diastereoselectivity. (*R,R*)-2,3-Butanediol (*S*)-chlorophenylmethaneboronate (**4a**, R = Ph) showed $k = 4.4 \times 10^{-5}$ s^{-1} for epimerization in 0.176 M lithium chloride in THF, only about twice the k for the corresponding pinanediol ester.[9] The usual precautions to avoid epimerization with reactive benzylic or allylic products[4] are sufficient.

Enantioselection has been one of Hoffmann's goals in his studies of stereoselective allylboronic ester/aldehyde reactions.[10] Based on our first results with **1** (R = Bu),[11] we suggested that he might try 2,3-butanediol as the directing group for the vinyl to α-chloroallyl homologation, and we are pleased that it has proved even better than anticipated.[12]

Experimental Data. (*R,R*)-2,3-Butanediol esters (**1**) were prepared from the boronic acids and diol in ether/hexane as described for pinacol esters.[5] Solutions were dried over magnesium sulfate before distillation.[13] The route to (*R,R*)-2,3-butanediol dichloromethaneboronate (**2**) began with either Rathke's preparation of the boronic acid[14] and isolation of its butyl ester[15] or the Brown–Cole

(1) (a) Matteson, D. S.; Sadhu, K. M. *J. Am. Chem. Soc.* **1983**, *105*, 2077–2078; correction (compound identification) 6195. (b) Matteson, D. S.; Sadhu, K. M., manuscript in preparation.

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(6) Matteson, D. S.; Majumdar, D. *Organometallics* **1983**, *2*, 1529–1535.

(7) It is a fermentation product. We used material purchased from Alfa, $[\alpha]^{22}_D$ −12.3° (neat), no impurity evident in the 200-MHz NMR spectrum.

(8) Both enantiomers of *threo*-2,3-butanediol can be made from tartrate esters: (a) Mori, K.; Tamada, S. *Tetrahedron* **1979**, *35*, 1279–1284. (b) Schurig, V.; Koppenhoefer, B.; Buerkle, W. *J. Org. Chem.* **1980**, *45*, 538–541. For the pure *S,S* isomer, $[\alpha]^{25}_D$ equals +13.38° (neat), and the possibility of some enantiomer contaminating the fermentation product was suggested, though harmless water seems more likely to us.

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(10) (a) Herold, T.; Hoffmann, R. W. *Angew. Chem., Int. Ed. Engl.* **1978**, *17*, 768–769. (b) Hoffmann, R. W.; Ladner, W. *Tetrahedron Lett.* **1979**, 4653–4656. (c) Hoffmann, R. W.; Landmann, B. *Ibid.* **1983**, *24*, 3209–3212.

(11) Sadhu, K. M. Ph.D. Thesis, Washington State University, 1983, pp 37–38, 53–54.

(12) Hoffmann, R. W.; Landmann, B., publication planned in *Angew. Chem.* We thank Professor Hoffmann for informing us of his interesting results prior to publication.

(13) R = Bu, bp 43–45 °C (5 torr);[17] R = Ph, bp 78–80 °C (2 torr);[17] R = *i*-Pr, bp 52 °C (20 torr).

(14) Rathke, M. W.; Chao, E.; Wu, G. *J. Organomet. Chem.* **1976**, *122*, 145–149.

direct route to its diisopropyl ester.[16] Mixing with an equimolar amount of (*R,R*)-2,3-butanediol and distilling yielded **2**: bp 68–70 °C (5 torr); $[\alpha]^{25}_{546}$ +10.2° (*c* 3.5, toluene) (hydrolyzed rapidly by atmospheric moisture).[17] To make **4a**, 5 mmol of **2** in 10 mL of THF was cooled to −78 °C and 5 mmol of Grignard or lithium reagent (purity checked by titration) was added dropwise. After the solution was stirred 5 min, ~480 mg (3.5 mmol) of anhydrous zinc chloride[1] was added and the mixture was kept at 20 °C overnight (except for R = Ph, 2 h at −30 to +20 °C). The solvent was evaporated under vacuum, and the residue was triturated with 3 × ~50 mL of petroleum ether. The solution was stirred with 10 g of anhydrous magnesium sulfate, filtered, and concentrated to yield **4a**, which showed only minor impurities in the NMR. Treatment of **4a** with an equivalent of optically pure (+)-pinanediol[1] in hexane followed by chromatography[1] (no isomer separation) yielded **4b** on which the isomer ratios were determined (Table I).

Acknowledgment. We thank the National Institutes of Health for Grant GM27109, the National Science Foundation for Grant CHE-8025229, and the Boeing Corp. for a gift in support of departmental purchase of the Nicolet NT-200 200-MHz NMR instrument.

(15) The reaction mixture[14] was extracted with butanol/ether and distilled; bp 68–70 °C (0.1 torr).
(16) Brown, H. C.; Cole, T. E. *Organometallics* **1983**, *2*, 1316–1319.
(17) Satisfactory analyses for all elements except O were obtained.

Generation and Reactions of Allylidene Complexes of the (η^5-Cyclopentadienyl)dicarbonyliron and (η^5-Cyclopentadienyl)(trimethyl phosphite)carbonyliron Systems

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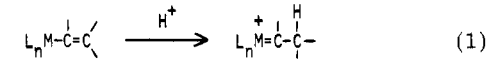
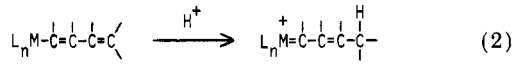
Summary: A series of four dienyliron complexes, Cp(CO)(L)FeCH=CHC(R^1)=CHR^2 [L = CO or $P(OCH_3)_3$; R^1 = H, R^2 = CH_3 or R^1 = CH_3, R^2 = H], is prepared by acylation of $[Cp(CO)_2Fe]Na$ with the appropriate dienoyl chlorides followed by photochemical decarbonylation and, when desired, ligand exchange of carbon monoxide by trimethyl phosphite. Treatment of these complexes with strong acid at low temperature produces the corresponding cationic allylidene complexes $[Cp(CO)(L)Fe{=}CHCH{=}C(R^1)CH_2R^2]^+$, which have been characterized by NMR and by their reaction behavior. Included among their reactions are rearrangement to an η^2-diene complex, nucleophilic addition of triphenylphosphine and of sodium methylmercaptide, and alkene cyclopropanation.

Transition-metal carbene complexes are now recognized as possible intermediates in many metal-promoted reactions,[1] including the cyclopropanation, metathesis,[2] and polymerization of alkenes[3] as well as Fischer–Tropsch syntheses.[4] Carbene complexes of the (η^5-cyclopentadienyl)dicarbonyliron system were first reported by Pettit[5] and by Green[6] several years ago and since that time have been studied extensively by several other groups.[7] With the goal of developing routes to alkylidene complexes in general, we have previously reported methylene,[8] ethylidene,[9] and isopropylidene complexes[10] in this series of iron compounds. We now wish to describe an entry into the corresponding *allylidene* (or vinylcarbene) complexes.[11]

The protonation of alkenylmetal complexes to produce the corresponding carbene complexes (eq 1) has been reported for various metals including the above-mentioned iron system.[12,13] This reaction serves as the basis of one of the routes to iron alkylidenes developed previously by us[10] and by Casey.[14] Analogous reactions of alkynylmetal compounds afford vinylidene complexes.[15] Continuing this analogy, one may expect the protonation of dienyl complexes to form allylidene complexes (eq 2) which are somewhat less common but potentially quite useful.[16] Indeed, this reaction forms the basis of the present report.





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(16) At issue in this approach is whether the protonation will occur at the β- or the δ-position of the dienyl ligand to give the desired allylidene complex or a 4-butylidene complex, respectively; see ref 23. For some examples of allylidene complexes of various metals see: (a) Fischer, E. O.; Kalder, H.-J.; Frank, A.; Koehler, F. H.; Huttner, G. *Angew. Chem., Int. Ed. Engl.* **1976**, *15*, 623. (b) Doetz, K. H. *Chem. Ber.* **1977**, *110*, 78. (c) Casey, C. P.; Polichnowski, S. W.; Shusterman, A. J.; Jones, C. R. *J. Am. Chem. Soc.* **1979**, *101*, 7282. (d) Wood, C. D.; McLain, S. J.; Schrock, R. R. *Ibid.* **1979**, *101*, 3210. (e) Nesmeyanov, A. N.; Petrovskaya, E. A.; Rybin, L. V.; Rybinskaya, M. I. *Bull. Acad. Sci. USSR, Div. Chem. Sci.* **1979**, *28*, 1887. (f) Mitsudo, T.; Watanabe, H.; Watanabe, K.; Watanabe, Y.; Kafuku, K.; Nakatsu, K. *Chem. Lett.* **1981**, 1687. (g) Klimes, J.; Weiss, E. *Angew. Chem., Int. Ed. Engl.* **1982**, *21*, 205. (h) McCullough, L. G.; Listemann, M. L.; Schrock, R. R.; Churchill, M. R.; Ziller, J. W. *J. Am. Chem. Soc.* **1983**, *105*, 6729.

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