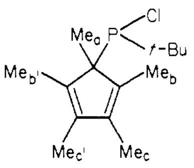
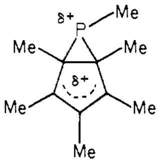
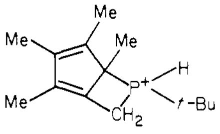


1

$[(Me_5C_5)(t\text{-}Bu)P]^+$ (**2**), is evident from the >70 ppm downfield ^{31}P chemical shift accompanying Cl^- removal: $^{31}P\{^1H\}$ NMR (36.43-MHz) **1** (s, δ +168), **2** (s, δ +240). The corresponding $(Me_3Si)_2CH$-substituted cation, $\{(Me_5C_5)[(Me_3Si)_2CH]P\}^+$ (**3**), was produced by aluminum chloride promoted Cl^- abstraction from $(Me_5C_5)[(Me_3Si)_2CH]PCl$ (**4**):[5] $^{31}P\{^1H\}$ NMR (36.43 MHz) **3** (s, +265.9), **4** (s, +171.0). Corroborative evidence for the **4** → **3** transformation is provided stereochemically. Compound **4** features a chiral center at phosphorus and hence exhibits diastereopy in the $(Me_3Si)_2CH$ group: ^1H NMR (90.0 MHz) Me_3Si (s, 9 H, δ 0.32), Me_3Si' (d, 9 H, δ 0.11, J_{PCSiCH} = 1.5 Hz). Cation **3** lacks such a chiral center; therefore, the Me_3Si groups are equivalent (s, 18 H, δ 0.24). The ring and Me carbons of cations **2** and **3** remain equivalent down to −90 °C in ^{13}C and ^1H NMR experiments: $^{13}C\{^1H\}$ NMR (50.31 MHz) **2**, C_5Me_5 (d, δ 139.7, J_{PC} = 14.7 Hz); C_5Me_5 (s, δ 11.5), **3**, C_5Me_5 (d, δ 137.8 J_{PC} = 13.4 Hz), C_5Me_5 (s, δ 12.6); ^1H NMR (90.0 MHz) **2**, C_5Me_5 (d, 15 H, δ 1.22, J_{PCCH} = 12.0 Hz), **3**, C_5Me_5 (d, 15 H, δ 2.16, J_{PCCH} = 3.0 Hz). These observations are consistent with the η^5 attachment of an RP^+ moiety to the Me_5C_5 ring or with fluxional system possessing lower degrees of hapticity. MNDO calculations[6] on the model cation $[C_5Me_5PMe]^+$ reveal that the global minimum corresponds to an η^2 structure:[7]



When CH_2Cl_2 solutions of **2** were allowed to stand ~5 days at room temperature, the NMR peaks of **2** disappeared gradually and were replaced by those of **5**. The ^{31}P chemical shift and P–H coupling constants of **5** (d, δ 87.1, J_{PH} = 503.6 Hz) are in the range anticipated[8] for phosphonium salts of type $[R_3PH]^+$, thus suggesting the formulation



5

Confirmation of the above formulation is provided by $^{13}C\{^1H\}$ NMR (50.31 MHz) spectroscopy (−35 °C). A doublet at δ 58.0 (J_{PC} = 16.7 Hz) is attributable to the CH_2 group, and the four methyl resonances appear as singlets at δ 10.7, 11.5, 11.9, and 12.0. A large doublet at δ 60.9 (J_{PC} = 132.4 Hz) is assignable to the α ring carbon attached to phosphorus. The β ring carbons appear at δ 144.4 (d, J_{PCC} = 8.8 Hz) and 147.1 (d, J_{PCC} = 10.9 Hz), and the γ ring carbons appear at δ 129.7 and 132.7. Very similar observations were made when CH_2Cl_2 solutions of **3** were allowed to stand at ambient temperature. The rate of production of phosphonium salt **6** from **3** is slightly faster than the **2** → **5** rearrangement (^{31}P NMR (36.43 MHz) for **6**: (d, δ 72.2, J_{PH} = 565.7 Hz)). The conclusion that the P–H bonds in **5** and **6** do not arise from solvent was established by the fact that no P–D bond formation took place when the reactions were conducted in CD_2Cl_2. The products therefore arise from the intramolecular insertion of P^+ into a C–H bond of a Me_5C_5 methyl group. Finally, we draw attention to the striking parallel between our findings and the observation that the bis(pentamethylcyclopentadienyl) compounds of titanium and zirconium also undergo ring methyl to metal hydrogen shifts.[9,10]

Acknowledgment. Generous support from the National Science Foundation (Grant CHE-8205871) and the Robert A. Welch Foundation is gratefully acknowledged. Thanks are also due to Jon Lasch for the MNDO calculations and Dr. B. Shoulders for helpful discussions on NMR spectroscopy.

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(4) The $AlCl_4^-$ anion is characterized by a sharp resonance (w_h ~10–20 Hz) at δ ~103. See: Akitt, J. W. *Annu. Rep. NMR Spectrosc.* **1972**, *5A*, 465.

(5) The new chlorophosphine, $(Me_5C_5)[(Me_3Si)_2CH]PCl$ (**4**) was prepared by the action of $(Me_3Si)_2CHLi$ with $C_5Me_5PCl_2$ in Et_2O solution and characterized by high-resolution mass spectrometry: calcd for $C_{17}H_{34}Si_2PCl$ 360.1625, Found 360.1613.

(6) For MNDO parameters for phosphorus, see: Dewar, M. J. S.; McKee, M.; Rzepa, H. *J. Am. Chem. Soc.* **1978**, *100*, 3607 and references to earlier work cited therein.

(7) For carbocation analogues of this structure, see: (a) Swatton, D. W.; Hart, H. *J. Am. Chem. Soc.* **1967**, *89*, 5075. (b) Childs, R. F.; Winstein, S. *J. Am. Chem. Soc.* **1968**, *90*, 7146. We are indebted to a referee for bringing this work to our attention.

(8) See, for example: Mavel, G. *Annu. Rep. NMR Spectrosc.* **1973**, *5B*.

(9) Bercaw, J. E. *J. Am. Chem. Soc.* **1974**, *96*, 5087. Bercaw, J. E. *Adv. Chem. Ser.* **1978**, *No. 167*, 136.

(10) For related work on ethyl–tungsten compounds, see: Chong, K. S.; Green, M. L. H. *Organometallics* **1982**, *1*, 1586 and references therein.

β-Hydrogen Effects in Alkylalkoxides of Dimolybdenum (M≡M)

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The reaction of a metal alkyl to form a metal hydride and alkene and its reverse, alkene insertion into a metal hydride, are fundamental and important reactions in organo–transition-metal chemistry.[1,2] We here report observations of β-hydrogen effects at a dinuclear metal center that show rather unusual, if not unprecedented, features when compared to those well documented at mononuclear centers.

Addition of alcohols to hydrocarbon solutions of 1,2-$Mo_2R_2(NMe_2)_4$ compounds[3] yields either 1,2-$Mo_2R_2(OR')_4$ or $Mo_2R(OR')_5$ compounds according to eq 1 and 2.[4]

$$Mo_2R_2(NMe_2)_4 + 4R'OH \rightarrow Mo_2R_2(OR')_4 + 4HNMe_2 \quad (1)$$

$$R = Me,\ R' = t\text{-}Bu;\ R = CH_2CMe_3,\ CH_2SiMe_3,\ R' = t\text{-}Bu,\ i\text{-}Pr,\ t\text{-}CH_2Bu,\ Et$$

$$Mo_2R_2(NMe_2)_4 + 5R'OH \rightarrow Mo_2R(OR')_5 + RH + 4HNMe_2 \quad (2)$$

$$R = C_2H_5,\ C_3H_7,\ R' = t\text{-}Bu,\ i\text{-}Pr$$

In both eq 1 and 2, the rate of alcoholysis shows a marked dependence on the steric bulk of R and R'. Alcoholysis of the alkyl ligands, Mo–R + R'OH → MoOR' + RH, is slower than the alcoholysis of the amide ligands. The compounds $Mo_2Me_2(O\text{-}t\text{-}Bu)_4$ is coordinatively unsaturated and reacts with donor ligands. The compound $Mo_2Me_2(O\text{-}t\text{-}Bu)_4(py)_2$, where py = pyridine, has been characterized by a single-crystal X-ray study.[5]

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(1) Kochi, J. K. In "Organometallic Mechanisms and Catalysts"; Academic Press: New York, 1978.

(2) Parshall, G. W. In "Homogeneous Catalysis: The Applications and Chemistry of Catalysis by Soluble Transition Metal Complexes"; Wiley: New York, 1980.

(3) Chisholm, M. H.; Haitko, D. A.; Huffman, J. C. *J. Am. Chem. Soc.* **1981**, *103*, 4046.

(4) All reactions were carried out in dry and oxygen-free solvents and atmospheres. The new compounds are yellow or orange solids which sublime and yield molecular ions in the mass spectrometer. Satisfactory elemental analyses have been obtained.

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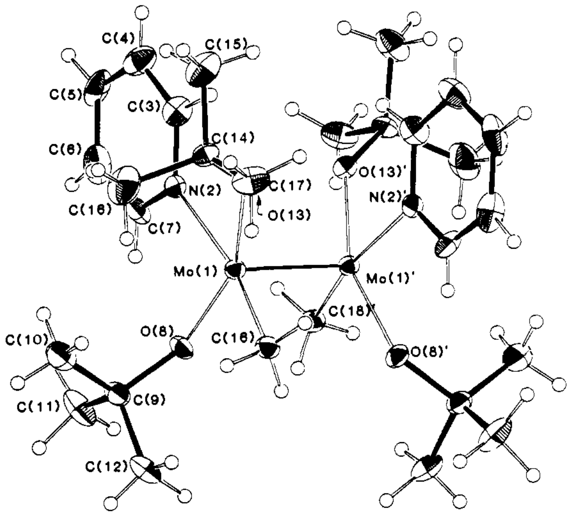

Figure 1. ORTEP view of the $Mo_2Me_2(O\text{-}t\text{-}Bu)_4(py)_2$ molecule showing the roughly square-planar coordination at each metal and the unbridged $Mo\equiv Mo$ bond. The molecule has rigorous C_2 symmetry. Some pertinent bond distances (Å) and angles (°) are Mo–Mo = 2.256 (1), Mo–O = 1.92 (6) (averaged), Mo–C = 2.167 (7), Mo–N = 2.349 (6), Mo–Mo–O = 101.5 (6), Mo–Mo–C = 97.3 (2), Mo–Mo–N = 94.5 (1).

An ORTEP view of this molecule is shown in Figure 1. In the presence of excess *t*-BuOH, $Mo_2Me_2(O\text{-}t\text{-}Bu)_4$ shows rapid exchange on the ^1H NMR time scale (16 °C, 220 MHz) between free alcohol and coordinated alkoxide ligands and slowly reacts further to give $Mo_2(O\text{-}t\text{-}Bu)_6$ and methane via the intermediate $Mo_2Me(O\text{-}t\text{-}Bu)_5$, which can be detected by ^1H NMR spectroscopy. The reaction between $Mo_2Me_2(NMe_2)_4$ and *i*-PrOH proceeds very rapidly to give $Mo_2(O\text{-}i\text{-}Pr)_6$, and it has not been possible to stop the reaction at $Mo_2Me_2(O\text{-}i\text{-}Pr)_4$. With the bulkier alkyl ligands CH_2CMe_3 and CH_2SiMe_3, the alcoholysis of Mo–NMe_2 becomes much slower, and we have been able to isolate $Mo_2R_2(OEt)_4$ compounds and even the mixed alkyl-amido-alkoxide $Mo_2(CH_2CMe_3)_2(NMe_2)_2(O\text{-}i\text{-}Pr)_2$. The spectroscopic data for these new compounds leave no doubt that they are members of a now extensive class of $X_2YM\equiv MX_2Y$ compounds.[3,6]

The fact that the ethyl and propyl compounds, $Mo_2R_2(NMe_2)_4$, reacted with *t*-BuOH and *i*-PrOH to give $Mo_2R(OR')_5$ compounds suggested a β-hydrogen effect, and this was confirmed and investigated further in the following manner.

(1) When $Mo_2(CH_2CD_3)_2(NMe_2)_4$ and $Mo_2(CH(CD_3)_2)_2(NMe_2)_4$ were allowed to react with *t*-BuOH, the eliminated alkanes were CH_2DCD_3 and CD_3CHDCD_3, respectively, as determined by ^2H NMR spectroscopy. The elimination of alkane is thus shown to occur by a reaction in which the β-hydrogen/deuterium atom of one alkyl ligand is transferred to the α-carbon of the other. This has been observed before in the reactions between $Mo_2R_2(NMe_2)_4$ compounds and CO_2, which yield $Mo_2(O_2CNMe_2)_4$ ($M\overset{4}{-}M$) and alkane and alkene when the ligand is ethyl, propyl, or butyl.[7] The elimination of alkane in **2** implies a reductive elimination step, $R{-}M\equiv M{-}R \rightarrow M\overset{4}{-}M$ + alkane + alkene, and as such requires an oxidative addition, $M\overset{4}{-}M + R'OH \rightarrow (M\equiv M)(H)(OR')$ and metal hydride–olefin insertion step to give the final alkyl ligand. This was demonstrated in 2 and 3 below.

(2) When $Mo_2(CH_2CH_3)_2(NMe_2)_4$ was allowed to react with *t*-BuOD, the ethyl ligand initially formed contained D in the β-position, $Mo_2(CH_2CH_2D)(O\text{-}t\text{-}Bu)_5$. The oxidative addition of R'OH to a M–M multiple bond has precedent in the reaction between $W_2(NMe_2)_6$ and *i*-PrOH, which yields $[W_2(\mu\text{-}H)(O\text{-}i\text{-}Pr)_7]_2$.[8] Alkene insertion into transition-metal hydride bonds is well-known,[1,2] and $[W_2(\mu\text{-}H)(O\text{-}i\text{-}Pr)_7]_2$ has been found to react reversibly with 1-alkenes to give either the alkyl derivative or isomerized alkenes.[8]

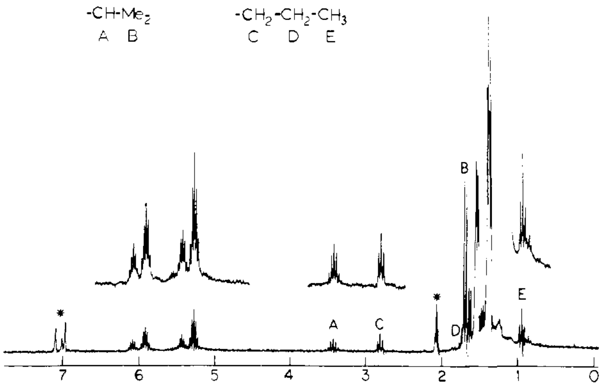

Figure 2. ^1H NMR spectrum (220 MHz, 16 °C) of the mixture of $Mo_2\text{-}i\text{-}Pr(O\text{-}i\text{-}Pr)_5$ (70%) and $Mo_2\text{-}n\text{-}Pr(O\text{-}i\text{-}Pr)_5$ (30%) compounds formed in the reaction between $Mo_2\text{-}n\text{-}Pr_2(NMe_2)_4$ and *i*-PrOH (ca. 6 equiv) in benzene after 15 min. The solvent was stripped and the mixture dissolved in toluene-d_8.

(3) In the presence of added ethylene (20-fold excess), $Mo_2\text{-}i\text{-}Pr_2(NMe_2)_4$ reacted with each of *t*-BuOH and *i*-PrOH to give $Mo_2Et(OR')_5$ compounds, along with propane and propene. This supports the view that both alkane and alkene are eliminated from the dinuclear center during the course of the reaction as was suggested in 1 above.

(4) Both normal and isopropyl compounds, $Mo_2Pr_2(NMe_2)_4$, reacted with each of *t*-BuOH and *i*-PrOH to give initially $Mo_2\text{-}i\text{-}Pr(OR')_5$ compounds which, in the presence of $HNMe_2$, isomerized to give the *n*-propyl compound. This can easily be monitored by NMR spectroscopy as is seen in Figure 2. The formation of the isopropyl ligand must occur by kinetic control and implies a conformational preference in olefin-metal binding prior to the insertion step.

(5) Pure $Mo_2\text{-}i\text{-}Pr(OR')_5$ compounds, where R' = *t*-Bu and *i*-Pr, are stable with respect to isomerization to the *n*-propyl derivative for days in hydrocarbon solvents in the absence of donor ligands such as $HNMe_2$. Nor do $Mo_2\text{-}i\text{-}Pr(OR')_5$ compounds react with added ethylene.

(6) $Mo_2\text{-}i\text{-}Pr(O\text{-}i\text{-}Pr)_5$ reacts rapidly to give $Mo_2\text{-}n\text{-}Pr(O\text{-}i\text{-}Pr)_5$ in the presence of donor ligands ($HNMe_2$, pyridine, PMe_3). $Mo_2\text{-}i\text{-}Pr(O\text{-}t\text{-}Bu)_5$ is isomerized much more slowly. In organo-transition-metal chemistry, alkyl ligand isomerization generally occus by a reversible β-hydrogen elimination, metal hydride–olefin insertion sequence.[1,2] The insertion step leading to the isomerized alkyl ligand must occur in the direction opposite to that in which it is formed: $M{-}R \rightleftarrows M(H)(\text{alkene}) \rightleftarrows M(H)(\text{alkene})' \rightleftarrows M{-}R'$. A similar phenomenon can be invoked to account for the observed isomerization in $Mo_2R(OR')_5$ compounds. However, this sequence must be promoted by Lewis base association reactions, which to our knowledge has no precedent in mononuclear transition-metal chemistry.[9]

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(5) Crystal data for $Mo_2Me_2(O\text{-}t\text{-}Bu)_4(py)_2$ at −161 °C: a = 19.536 (6) Å, b = 10.514 (2) Å, c = 18.032 (5) Å, β = 117.22 (1)°, Z = 4 in space group $C2/c$. Of the 2314 reflections collected by using Mo Kα radiation, 5° < 2θ < 50°, the 1696 having $F > 2.33\sigma(F)$ were used in the full-matrix refinement. Final residuals are R_F = 0.041 and R_{wF} = 0.040.

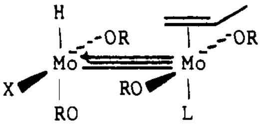
(6) Chisholm, M. H.; Folting, K.; Huffman, J. C.; Rothwell, I. P. *Organometallics* **1982**, *1*, 251.

(7) Chetcuti, M. J.; Chisholm, M. H.; Folting, K.; Haitko, D. A.; Huffman, J. C. *J. Am. Chem. Soc.* **1982**, *104*, 2138.

(8) Akiyama, M.; Chisholm, M. H.; Cotton, F. A.; Extine, M. W.; Haitko, D. A.; Leonelli, J.; Little, D. *J. Am. Chem. Soc.* **1981**, *103*, 779.

(9) In mononuclear organo-transition-metal chemistry, the β-hydrogen abstraction step is well-known to require the presence of a vacant coordination site. Lewis base dissociation may thus be rate determining, and added Lewis bases may suppress the overall rate of reaction by reducing the concentration of the coordinatively unsaturated complex. For examples of β-hydrogen abstraction, see: (i) $Au(t\text{-}Bu)Me_2(PPh_3) \rightarrow Au(i\text{-}Bu)Me_2(PPh_3)$: Tamaki, A.; Kochi, J. K. *J. Chem. Soc., Chem. Commun.* **1973**, 423. (ii) $CpFe(CO)(PPh_3)R \rightarrow CpFe(CO)(PPh_3)H$ + alkene: Reger, D. L.; Culbertson, E. C. *J. Am. Chem. Soc.* **1976**, *98*, 2789. $CpW(CO)_3(n\text{-pentyl}) \rightarrow CpW(CO)_2(1\text{-pentene})(H)$: Kazlauskas, R. J.; Wrighton, M. S. *Ibid.* **1980**, *102*, 1727; **1982**, *104*, 6005.

We have found that Lewis base association to $Mo_2(OR)_6$ compounds which give $Mo_2(OR)_6L_2$ compounds is very sensitive to the steric properties of L and the alkoxy group and, furthermore, the binding of L is cooperative: the coordination of one ligand is followed by a more rapid coordination of the second. It is thus possible to rationalize the aforementioned reactions by invoking a common cooperative β-hydrogen elimination/alkene insertion mechanism in which a key intermediate is depicted by I.



I

Elimination of alkane occurs during alcoholysis when L = $HNMe_2$ and X = alkyl. For electronic reasons, the preferred alignment of the alkene should be along the M–M bond, and for steric reasons, the methyl group would be favored distal to the M–M bond. Thus, insertion to give the isopropyl ligand can occur when X = OR. Providing the olefin can gain access to a position in which the methyl substituent is over the Mo–Mo bond, ultimately the presence of L will allow for isomerization of the alkyl ligand, *i*-Pr → *n*-Pr.

Further studies are in progress.[10]

Supplementary Material Available: Listing of fractional coordinates and isotropic thermal parameters (2 pages). Ordering information is given on any current masthead page.

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(10) We thank the Deparment of Energy, Office of Basic Research, Chemical Sciences Division, for financial support and the National Science Foundation for Instrument Grant CHE 81-05004.

Boronic Ester Homologation with 99% Chiral Selectivity and Its Use in Syntheses of the Insect Pheromones (3*S*,4*S*)-4-Methyl-3-heptanol and *exo*-Brevicomin

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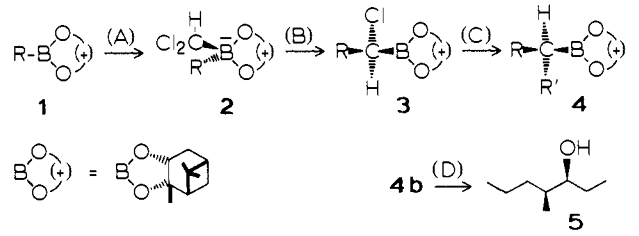
Received December 27, 1982

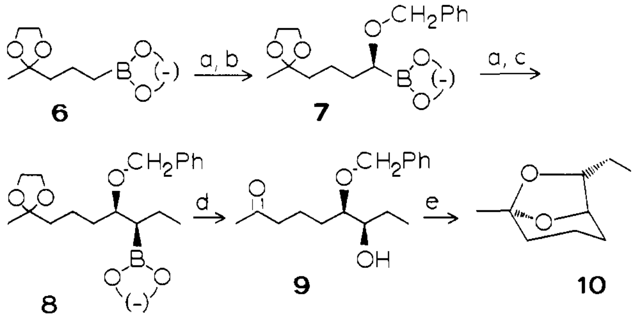
Our directed chiral synthesis of α-chloro boronic esters (**3**) from pinanediol boronic esters (**1**; Scheme I)[1-3] has been dramatically improved by zinc chloride catalysis of the rearrangement of the intermediate borate complexes (**2**). Diastereoselectivities are 98.5–99.5% (except R = CH_3), yields 82–99% (Table I). The utility is demonstrated by efficient syntheses of two insect pheromone components, (3*S*,4*S*)-4-methyl-3-heptanol (**5**) and *exo*-brevicomin (**10**). This chemistry is compatible with a wide variety of structural types and may allow the controlled sequential construction of an unlimited number of adjacent chiral centers.

Scheme I[a]



[a] (A) $LiCHCl_2$, −100 °C; (B) $ZnCl_2$; (C) R'MgBr; (D) H_2O_2/NaOH.

Scheme II[a]



[a] (a) A + B of Scheme I; (b) $LiOCH_2Ph$;[3] (c) EtMgBr, −78 °C; 36 h at 20 °C; (d) H_2O_2/NaOH, H^+/SiO_2; (78%); (e) H_2/Pd.[12b]

Table I. Homologation of Boronic Esters (**1**) to α-Chloroboronic Esters (**3**)

	yield, %[a]		diastereoselectivity, %		
$RBO_2C_{10}H_{16}$ (**1**)	no $ZnCl_2$	with $ZnCl_2$	no $ZnCl_2$	with $ZnCl_2$	anal. method[b]
1a		87		99	A
1c	57[c]	83	74[c]	95.7	B
1d	61[c]	92[d]	90[c]	98.5	A
1e	30[e]	89	88[e]	99.5	B
1f	75	99	92.5	99.5	B
6		93		99.5[f]	B
7		82		99.5	B

[a] Isolated **3** or **4**. [b] (A) Rotation of derived alcohol;[1] (B) 200-MHz NMR analysis of **3** or NHAc derivative[2,4] compared with epimeric mixture. [c] Reference 1. [d] Data by Mark Peterson. [e] Reference 4. [f] Derived **7**, 98%.

Failure of the original procedure[1] to give satisfactory yields from pinanediol 2-methylpropane-1-boronate (**1e**)[4] prompted a search for better conditions. Noting that lithium chloride catalyzes[1,5] and mercuric chloride inhibits[5] epimerization of **3**, we tested the effects of several metal salts capable of complexing chloride ion in the preparation of **3**. Zinc chloride,[6] 0.5–0.65 equiv, was added to **2** at −100 °C and the mixture kept for 6–8 h at 20–25 °C to yield the results summarized in Table I.

The improved procedure was immediately applicable to the synthesis of (3*S*,4*S*)-4-methyl-3-heptanol (**5**; European elm bark beetle, *Scolytus multistriatus*;[7] Scheme I). (+)-Pinanediol propane-1-boronate (**1a**)[8,9] was homologated to **4a** ≡ **1b** and then to **4b**, which was oxidized to **5**: overall yield 58–63%; $[\alpha]^{24}_{D}$ −19.8° (*c* 4.4, hexane) [lit.[7c] $[\alpha]^{22}_{D}$ −21.7° (*c* 0.57, hexane)]; proton NMR and IR spectra in accord with reported data.[7] The diastereomeric impurity in **5** was not detectable by 200-MHz NMR, even of the

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(1) (1) Matteson, D. S.; Ray, R. *J. Am. Chem. Soc.* **1980**, *102*, 7590–7591. Matteson, D. S.; Ray, R.; Rocks, R. R., manuscript in preparation.

(2) Matteson, D. S.; Sadhu, K. M.; Lienhard, G. E. *J. Am. Chem. Soc.* **1981**, *103*, 5241–5242.

(3) Matteson, D. S.; Majumdar, D. *J. Am. Chem. Soc.* **1980**, *102*, 7588–7590.

(4) Matteson, D. S.; Jesthi, P. K., manuscript in preparation.

(5) Matteson, D. S.; Erdik, E., manuscript in preparation.

(6) Zinc chloride was dried to a fine powder at 0.01–0.02 torr at up to 100 °C with magnetic stirring. Other experimental procedures resembled those already described,[1-3] except that **3** were isolated by concentration below 30 °C, workup with saturated aqueous ammonium chloride and hexane, and concentration to crude **3** satisfactory for preparation of **4**. Products **3**–**9** were chromatographed on silica.

(7) (a) Pearce, G. T.; Grove, W. E.; Silverstein, R. M.; Peacock, J. W.; Cuthbert, R. A.; Lanier, G. N.; Simeone, J. B. *J. Chem. Ecol.* **1975**, *1*, 115. (b) Mori, K. *Tetrahedron* **1977**, *33*, 289–294. (c) Mori, K.; Iwasawa, H. *Ibid.* **1980**, *36*, 2209–2213.

(8) Boronic acids are readily prepared from Grignard reagents: Washburn, R. M.; Levens, E.; Albright, C. F.; Billig, F. A. "Organic Syntheses"; Wiley: New York, 1963; Collect. Vol. IV, pp 68–72. Esterification: ref 1 and 2. Hydroboration routes to boronic acids are of potential interest: Brown, H. C.; Jadhav, P. K.; Desai, M. C. *J. Am. Chem. Soc.* **1982**, *104*, 4303–4304.

(9) Pinanediol: Ray, R.; Matteson, D. S. *J. Indian Chem. Soc.* **1982**, *52*, 119–123. Enantiomeric purification: ref 2.

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