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(9) The capped cyclodextrin (**5**) was very stable in alkaline solution (pH 11, aqueous Na_2CO_3, 25 °C, >12 h), in marked contrast to the unstable monotosylate **2**. The capping positions were discussed in ref 2e and 2i.

(10) **3**: ^1H NMR (D_2O) δ 2.0 (3 H, CH_3, s), 2.8 (2 H, CH_2S), 3.2–4.1 (40 H, cyclodextrin protons other than C_1 H), 4.90 (7 H, C_1 H); IR spectrum was very similar to that of β-cyclodextrin. Found: C, 43.55; H, 6.22. Calcd for $C_{43}H_{72}O_{34}S\cdot H_2O$: C, 43.64; H, 6.30. **4**: ^1H NMR (D_2O–Me_2SO-d_6) δ 1.18 (9 H, CH_3, s), 2.9 (2 H, CH_2S), 3.15–4.0 (40 H, cyclodextrin protons other than C_1 H), 4.81 (7 H, C_1 H); IR spectrum was very similar to that of β-cyclodextrin. Found: C 45.08; H, 6.38. Calcd for $C_{46}H_{78}O_{34}S\cdot H_2O$: C, 45.09; H, 6.58.

(11) When fast reactions were being followed, the time necessary to complete the mixing of the reactants and to begin recording of the spectral change did not exceed 6 s.

(12) Although the methyl-substituted ester was bound by β-cyclodextrin more strongly than the nitro-substituted ester, the former was bound by the capped cyclodextrin (5) more weakly than the latter. This may indicate the importance of an interaction between the nitro group and the capping moiety by London dispersion force. The significance of London dispersion forces of a nitro group in guest bindings by α-cyclodextrin was discussed: R. J. Bergeron, M. A. Channing, G. Gibeily, and D. M. Pillor, *J. Am. Chem. Soc.*, **99**, 5146 (1977).

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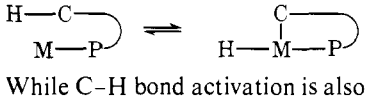
Activation of Carbon–Hydrogen Bonds by $[Rh(diphos)_2]^0$

Sir:

The activation of carbon–hydrogen bonds poses an interesting and important challenge. Despite the apparent similarity to H_2 in terms of bond energy and polarity, the C–H bond, unlike H_2, does not readily undergo activation via oxidative addition. Most examples are restricted to intramolecular metalations[1] (eq 1), in which entropic effects undoubtedly play



(1)

a major role. While C–H bond activation is also indicated by H/D exchange reactions of arenes[2,3] using catalysts such as $PtCl_4^{2-}$ in D_2O/DOAc and NbH_3Cp_2 under D_2, the mechanism of the exchange and details of the activation process remain uncertain. In the present paper, we describe an approach to the activation of C–H bonds which is based on the notion that a highly energetic odd-electron complex can react with C–H bonds to form organic radicals by eq 2, and we demonstrate that this reaction does indeed occur. Implicit in this approach to C–H bond activation is the view that the more energetic the M· species, the more stable the resultant M–H bond, and, when the bond dissociation energies for M–H and C–H are of similar value, reaction 2 may proceed at a useful rate.[4]

$$M\cdot + R\text{-}H \rightarrow M\text{-}H + R\cdot \qquad (2)$$

Previously, we reported[5] the electrochemical reduction of $[Rh(diphos)_2]^+$ in CH_3CN, Me_2SO, and DMA and subsequent formation of the hydride, $RhH(diphos)_2$; the overall reaction consumes two e^-. Through a combination of electrochemical and chemical techniques, we established that the reduction proceeds in one-electron steps, initially yielding $[Rh(diphos)_2]^0$ which reacts with a solvent molecule to form the product hydride and a reducible solvent radical which undergoes the second electron transfer. This sequence is shown in eq 3–5 and represents an ECE mechanism for the reduction. Since eq 4 represents a subset of eq 2, thereby demonstrating its feasibility, we next focused on using a solvent that would preclude (4) and permit us to study the reaction of the Rh^0 species with added substrates.

$$Rh^+ + e^- \rightarrow Rh\cdot \qquad (3)$$

$$Rh\cdot + Sol\text{-}H \rightarrow Rh\text{-}H + Sol\cdot \qquad (4)$$

$$Sol\cdot + e^- \rightarrow Sol^- \qquad (5)$$

To this end, we examined the electrochemical reduction of $[Rh(diphos)_2]^+$ in benzonitrile. Cyclic voltammetry (CV) of $[Rh(diphos)_2](ClO_4)$ in this solvent at a hanging Hg drop electrode (HMDE) shows a reversible one-electron couple at −2.10 V vs. an Ag/0.1 M $AgNO_3$ in benzonitrile reference. Constant potential coulometry (CPC) at −2.20 V yields, as before,[5] a coulometric n value of 2 and the hydride, $RhH(diphos)_2$, as the sole inorganic product. Analysis of the organic distillate by GC–MS reveals that tributylamine is formed in 80–95% conversion with respect to the initial amount of Rh complex. 1-Butene is also produced, although a quantitative analysis of the yield was not obtained because of evaporation.[6] In the previous electrolyses[5] the source of the hydrogen atom was demonstrated to be the solvent. In benzonitrile, however, the primary source of the hydrogen atom for Rh hydride formation is the electrolyte, tetrabutylammonium perchlorate, and not the solvent. The formation of Hofmann degradation products from the reaction of electrochemically generated anionic intermediates with tetraalkylammonium salts is not uncommon.[7] We envision this to be a similar process involving initial attack of Rh^0 on a C–H bond of the electrolyte followed by an electron transfer and subsequent elimination to yield tributylamine and 1-butene.

The viability of a competition between added C–H bond substrates and the electrolyte was demonstrated by the generation of $[Rh(diphos)_2]^0$ in a mixed solvent system. Electrolysis of $[Rh(diphos)_2]^+$ in benzonitrile containing 1.0 M CD_3CN and 0.1 M (n-Bu_4N)ClO_4 yields an 1:1 mixture of $RhH(diphos)_2$ and $RhD(diphos)_2$.[8] Both a statistical correction for available hydrogens and an isotope effect should favor reaction with (n-Bu_4N)ClO_4 and compensate for the differences in molar concentrations of the two substrates. Thus, the observed 1:1 RhH–RhD product composition indicates no dramatic preference of the Rh^0 intermediate for reaction with either substrate.

It was, therefore, of primary interest to explore the reactivity of this Rh^0 complex with *unactivated* C–H bonds. Electrochemical reduction of $[Rh(diphos)_2]^+$ in a 1:1 by volume solution of benzonitrile and cyclohexane containing 0.1 M (n-Bu_4N)ClO_4 reveals[10] the formation of cyclohexyl radical as

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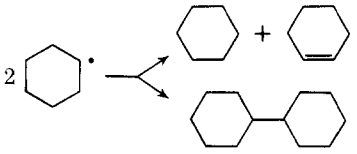
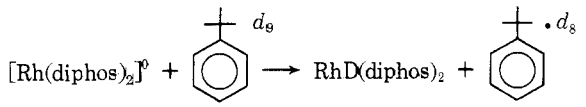
determined by the identification of cyclohexene and bicyclohexyl, the characteristic disproportionation and dimerization products[11] (eq 6). The yield of these bimolecular radical



(6)

products is ~5% with respect to the initial Rh concentration. The direct interaction of $[Rh(diphos)_2]^0$ with cyclohexane is established by reduction of the complex in a 1:1 solution of benzonitrile and cyclohexane-d_{12}. Again, cyclohexene and bicyclohexyl are observed; in addition, $RhD(diphos)_2$ is isolated as ~30% of the rhodium "hydride" product.[7,8,12,13]

To probe further the reactivity and radical nature of $[Rh(diphos)_2]^0$, $[Rh(diphos)_2]^+$ was reduced in a 1:1 by volume solution of benzonitrile and *tert*-butylbenzene. Analysis of the solution by GC–MS following the electrolysis indicates that isobutylbenzene is produced in ~10% conversion with respect to the electroactive Rh complex. This result indicates that the classic neophyl rearrangement has occurred, signalling the formation of the neophyl radical. The reduction of $[Rh(diphos)_2]^+$ in the presence of *tert*-butylbenzene-d_9 also generates the neophyl rearranged product and $RhD(diphos)_2$ is isolated as 33% of the "hydride" product.[8,13] Thus, the direct reaction of the Rh^0 complex with *tert*-butylbenzene-d_9 is established as the source of the neophyl radical as shown in eq 7 and 8.



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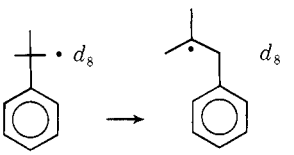

(8)

The sole transition-metal product in all the aforementioned electrolyses is the rhodium(I) hydride, or deuteride, which is isolated in ~90% yield. This complex, $RhH(diphos)_2$, is electrochemically inactive at these potentials and, in general, precipitates from the electrolysis solution. This product can easily be reconverted into the electroactive $[Rh(diphos)_2]^+$ by reaction with 1 equiv of acid. Thus, reaction of the hydride with benzoic acid or *p*-toluenesulfonic acid gives a quantitative yield of the corresponding Rh^I salts; hydrogen is also produced (eq 9). The $RhH(diphos)_2$ is also transformed[14] into $[Rh(diphos)_2]^+OH^-$ in ~80% yield via reaction with O_2 by a mechanism that is not presently understood. These reactions of the Rh hydride suggest a way to make the system electrocatalytic for the activation of C–H bonds.

$$RhH(diphos)_2 + HA \rightarrow [Rh(diphos)_2]^+A^- + H_2 \quad (9)$$

In summary, the labeling experiments demonstrate the transfer of deuterium from substrate to rhodium. The product studies show that the substrate is converted into a free radical by this process. These studies, in conjunction with our previous chemical and electrochemical studies,[5] constitute the basis for our recognition of the occurrence of eq 2. Thus, we have shown that the Rh^0 complex $[Rh(diphos)_2]^0$ is capable of activating sp^3 C–H bonds by hydrogen atom abstraction.[15,17] We are currently working on modifications of this system such that the activation and functionalization of saturated alkanes can be performed catalytically by this indirect electrochemical process.

Acknowledgments. We thank the National Science Foundation (Grants CHE 76-17440 and GP-38786) for partial support of this work and Matthey Bishop Co., Inc., for a generous loan of Rh salts. We also thank Ms. Michele Cree for help in carrying out part of this study.

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(4) The relationship between the reactivity of the M· species and the bond dissociation energy *D*(M–H) is the same as that made for organic free radicals and the corresponding *D*(C–H) of the homolyzed bond. For example, phenyl radical is more reactive to C–H bonds than *tert*-butyl radical; *D*(C–H) for benzene is 110.5 kcal/mol and that for the tertiary C–H bond of isobutane is 92 kcal/mol: S. W. Benson, "Thermochemical Kinetics", 2nd ed., Wiley, New York, 1976, p 309. An analysis of (2) into the component reactions (2a–2d) shows explicitly how the energy content of M· relates directly to the potential at which the odd-electron species is produced:

$$M\cdot \rightarrow M^+ + e^- \quad (2a)$$

$$R\text{–}H \rightarrow R\cdot + H\cdot \quad (2b)$$

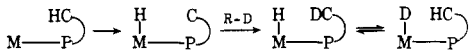
$$H\cdot + e^- \rightarrow H^- \quad (2c)$$

$$M^+ + H^- \rightarrow M\text{–}H \quad (2d)$$

$$\overline{M\cdot + R\text{–}H \rightarrow M\text{–}H + R\cdot} \quad (2)$$

Only (2a) and (2d) depend on the nature of the complex, the former corresponding to the 1-electron oxidation of M· and the latter corresponding to a ligation step. Reaction 2d will always be exothermic. The ΔG for (2a), however, will vary widely depending on the identity of the metal, the redox couple and the types of ligands present. Therefore, there is no reason to believe that the sum of the ΔG's for (2a) and (2d) will be constant as the nature of the metal complex varies, and, in fact, one would expect *D*(M–H) to vary widely with the nature of the complex. Furthermore, by this analysis, the net reaction 2 becomes favorable only when reaction 2a is strongly exothermic.
(5) J. Sofranko, R. Eisenberg, and J. Kampmeier, *J. Am. Chem. Soc.*, **101**, 1042 (1979).
(6) The yield of 1-butene was not quantitatively obtained because of continuous argon purging during the electrolysis.
(7) A. Merz and G. Thumm, *Tetrahedron Lett.*, **No. 7**, 679 (1978); R. Alvarado de la Torre and J. Sease, *J. Am. Chem. Soc.*, **101**, 1687 (1979).
(8) The ratio of RhH:RhD was determined by comparison of the absorbances, $A = \log T_0/T$, of the RhH and RhD stretches at 1870 and 1364 cm^{-1}, respectively, to the phenyl stretch of the ligand at 1420 cm^{-1}. Comparison of authentic samples[9] of $RhH(diphos)_2$ and $RhD(diphos)_2$ shows a ratio of absorbances, RhH:RhD, of 2.0 ± 0.1. In the analysis this ratio was of empirical significance only.
(9) A. Sacco and R. Ugo, *J. Chem. Soc.*, 3274 (1964).
(10) Electrolysis was performed at −2.2 V vs. an 0.1 M $Ag/AgNO_3$ reference on a Hg pool under continuous argon flow. The solutions were 2 mL in volume and 30–50 mM in $[Rh(diphos)_2]^+$.
(11) W. A. Cramer, *J. Phys. Chem.*, **71**, 1171 (1967).
(12) When $RhD(diphos)_2$ is heated at 65 °C for 1 h in a solution of 1:1 benzonitrile and cyclohexane, $RhD(diphos)_2$ is isolated in quantitative yield with no RhH/RhD exchange.
(13) The differences between the amounts of Rh deuteride isolated and radical products observed relates mainly to the fact that the substrate radical, once formed, has paths available to it other than radical product formation or rearrangement. The most notable of these is H· abstraction from the electrolyte, leading to substrate regeneration and electrolyte decomposition.
(14) The compound, $[Rh(diphos)_2]^+OH^-$ is a 1:1 electrolyte as determined by conductivity measurements. Its CV trace is identical with that of $[Rh(diphos)_2]^+ClO_4^-$ with the addition of an irreversible oxidation wave at −0.5 V corresponding to OH^- oxidation on mercury.
(15) While eq 2 is the unambiguous net result of the reaction between $[Rh(diphos)_2]^0$ and R–H, and we believe this reaction to proceed directly as an H atom abstraction, there also exists the possibility of oxidative addition to form a rhodium(II) alkyl hydride followed by homolysis of the Rh–R bond. In this context we note that the $[Rh(diphos)_2]^0$ system appears to be unreactive with aromatic C–H bonds. When an electrolysis is performed in benzonitrile–benzene-d_6 (1:1 v/v), we see no evidence of rhodium deuteride produced. This observation contrasts with the recent report of Tolman et al.[16] who find that $[Fe(dmpe)_2]^0$ [dmpe = bis(dimethylphosphino)ethane], generated in situ, preferentially attacks aromatic C–H bonds. Substituted and unsubstituted benzenes readily add to this Fe^0 species to form aryl hydride complexes, and, with toluene as a substrate, no evidence is reported for attack on the methyl C–H bonds. With the present Rh system, we observe rhodium deuteride formation when $C_6H_5CD_3$ is used as a substrate. It is a well-known fact that the benzylic hydrogen atoms of toluene are significantly more reactive to radical abstraction than are the aromatic H atoms. Our results and those of Toman et al.[16] are totally consistent if *one* assumes the reactive d^8 Fe^0 complex in their report (*and* its Ru analogue) activates C–H bonds by an oxidative addition mechanism with significant stability obtained from M–aryl bond formation, whereas in the present study the d^9 Rh^0 system activates C–H bonds by direct radical abstraction, as indicated by the fact that sp^3 C–H bonds are attacked while stronger sp^2 C–H bonds are not.

(16) C. A. Tolman, S. D. Ittel, A. D. English, and J. P. Jesson, *J. Am. Chem. Soc.*, **101**, 1742 (1979).
(17) An alternative explanation of the observed reaction chemistry has been put forth by one reviewer, and involves a ligand based radical according to the sequence shown. This proposal can be ruled out based on the following observations: (1) the product rhodium hydride complex is electrochemically inactive at the potential employed for electrolysis [−2.20 V vs. Ag/Ag(NO$_3$)]; (2) in dry CD$_3$CN as solvent, only the rhodium *deuteride* species is formed as the inorganic product in the electrolysis; and (3) once formed, the rhodium hydride or deuteride does not undergo exchange when placed in deuterated or nondeuterated solvents, respectively. The consequence of (1) is that each Rh center undergoes reaction only once. If the scheme above involving the ligand radical is correct, then the final step or equilibrium should lead to significant amounts of rhodium hydride product when deuterated solvents are used. However, this is in contradiction with (2). If H/D exchange is invoked to remove the contradiction, then a contradiction with (3) is necessitated. Thus, we can rule out the intermediacy of a ligand based radical scheme shown above, even though the absence of deuterium in the diphos ligands after electrolysis was not verified directly.



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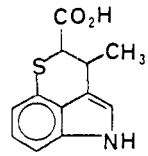
Total Synthesis of the Unique Indole Alkaloid Chuangxinmycin. Application of Nitro Group Displacement Reactions in Organic Synthesis

Sir:

Chuangxinmycin is a new antibiotic produced by the microorganism *Actinoplanes tsinanenis* n.sp., obtained from a soil sample collected in Tsinan, Shantung Province, China.[1] Chemical studies carried out by the Chuangxinmycin Research Group at the Institute of Materia Medica in Peking have led to the following structural assignment for this compound (the



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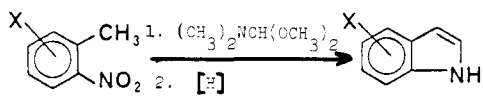
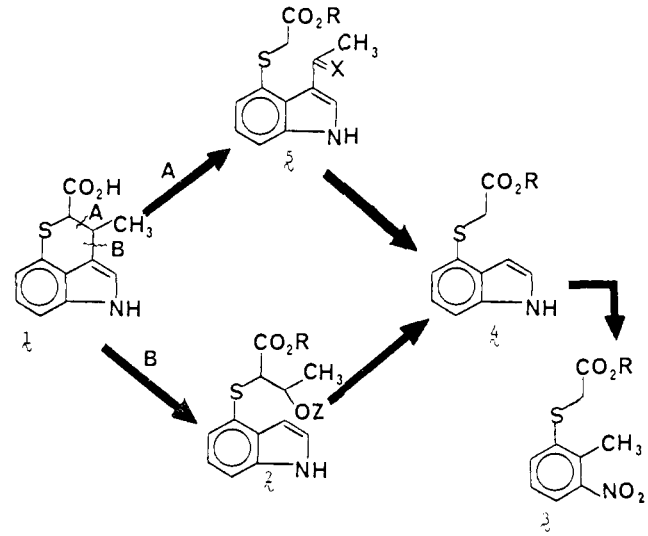
relative stereochemistry of the asymmetric centers was not established with any certitude). Preliminary clinical studies at the Chinese Academy of Medical Science have, moreover, shown chuangxinmycin to be 78% effective in the treatment of septicaemia and urinary and biliary infections.

The unique structural features (a new type of heterocyclic system) of chuangxinmycin, combined with its very promising biological activity, prompted us to attempt the total synthesis of this natural product.

Our synthetic approach to this compound was guided by our experience with the preparation of variously substituted indoles by the Leimgruber-Batcho methodology.[2] This process consists of simply heating an *o*-nitrotoluene with *N,N*-dimethylformamide dimethyl acetal to yield an *o*-nitrophenylacetaldehyde enamine. Subsequent reduction of the nitro group [Fe(II), H_2, dithionite, etc.] directly furnishes the indole. This chemistry thus resembles the Reissert indole synthesis, but bypasses the final decarboxylation step.



Scheme I. Retrosynthetic Analysis of Chuangxinmycin



Several retrosynthetic pathways from the target structure now become apparent based on the utilization of this chemistry. Two possibilities, displayed in Scheme I, lead to the sulfur-bearing nitrotoluene **3** as the required starting material.

Our preliminary efforts to prepare **3** were founded on an observation recorded by Piers and co-workers in their synthesis of 4-mercaptoindole.[3] They had shown that the halogen atom of 2-bromo-6-nitrotoluene could be displaced by potassium benzylmercaptide in DMF to afford the corresponding thioether in 26% yield. We, in fact, attempted to carry out this experiment, using the commercially available 2-chloro-6-nitrotoluene with HMPA as solvent. To our initial surprise, displacement of the nitro group had occurred instead. This result was, however, quite in line with previously reported data concerning the relative rates of displacement of various activated aromatic groups with several different nucleophiles ($Me_2S^+ > Me_3N^+ > F \simeq NO_2 > Cl$).[4] This information moreover suggested that the commercially available 2,6-dinitrotoluene might serve as a suitable precursor to the thioether **3**.[5] Indeed, simply adding powdered lithium hydroxide to an HMPA solution of methyl thioglycolate and 2,6-dinitrotoluene at room temperature and stirring for 1 day afforded **3** in good yield (70%): mp 46–47 °C; IR ($CHCl_3$) 1736, 1523, 1352 cm^{-1}; NMR ($CDCl_3$) δ 6.90–7.70 (m, 3 H), 3.76 (s, 3 H), 3.63 (s, 2 H), 2.60 (s, 3 H).

The conversion of this product into the corresponding indole now required treatment with *N,N*-dimethylformamide dimethyl acetal. As might have been anticipated, while enamine formation did take place with this substrate, reaction occurred exclusively at the more acidic methylene group rather than at the methyl substituent. To shift the regiochemical course of this reaction, the methylene site was deactivated by converting the ester into the potassium salt (KOH, MeOH) of the corresponding acid, thereby enabling the reaction to occur exclusively at the site of the methyl group. The enamine produced was hydrolyzed directly to acid aldehyde by treatment with cold 6 N HCl, and this crude product immediately was reduced with $FeSO_4/NH_4OH$.[6] The crude indole acid was esterified with diazomethane and chromatographed on silica gel to afford in 43% overall yield for these four steps[7] (Scheme II) the ester **4**: mp 83–85 °C; IR ($CHCl_3$) 1730 cm^{-1}; NMR ($CDCl_3$) δ 7.20 (m, 4 H), 6.65 (m, 1 H), 3.70 (s, 2 H), 3.67 (s, 3 H).

While this indole **4** could readily be transformed to a 1:1 diastereomeric mixture of alcohols **2** (Scheme I, Z = H; LDA, CH_3CHO), all attempts to effect ring closure of this mixture by converting the hydroxyl group into a good leaving group

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