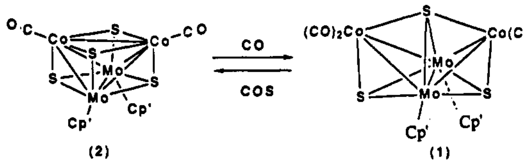
The reactions of **1** mimic desulfurization of thiophene over metal surfaces. Thiophene has been shown to readily undergo stoichiometric desulfurization on single-crystal surfaces such as Mo(100)[14] and Mo(110).[15] The products of these reactions are gaseous dihydrogen, surface carbon, and surface sulfur with no evidence for the formation of hydrocarbons. The deposition of sulfur on the metal surface is analogous to the formation of **2** from **1**. The reaction of **1** with thiophene in the absence of hydrogen also produces a black insoluble material which is presumed to be the carbon-containing residue from the desulfurization of thiophene. It is interesting to note that this residue is not produced when the desulfurization is conducted under H_2.

Conversion of **2** back to **1** would complete the cycle necessary for homogeneous, HDS catalysis. Thus, **2** gave a 20% conversion to **1** after 12 h when heated to 150 °C in toluene under 1000 psi of CO (eq 2). COS was identified via GC–MS as the only gaseous product. Independent experiments have shown that **1** abstracts sulfur from COS to form **2**. Thus, a higher CO pressure would give a higher conversion of **2** to **1**. Neither cluster **1** nor **2** reacts with H_2 (500 psi, 150 °C).



(2)

The reactions depicted in eqs 1 and 2 form the basis for a homogeneous catalytic cycle for the desulfurization of thiophene. However, under actual catalytic conditions (1000 psi of CO, 200 psi of H_2, thiophene), the forward reaction was inhibited by the presence of excess CO and no conversion of **1** to **2** was obtained.

In refluxing toluene, **1** (250 mg) reacted with thiophenol (1.0 mL in 100 mL of toluene) to give an 80% isolated yield of **2** after 7 h. Benzene was identified by GC–MS as the organic product. This reaction is the homogeneous analogue of the reaction of thiophenol on Mo(110)[16] surfaces, which produces adsorbed sulfur and benzene. Cleavage of aryl C–S bonds by transition-metal clusters has been reported,[17] but a mixture of organometallic products results. Under catalytic conditions (1000 psi of CO, 150 °C), PhSH (18 mmol) reacts with **1** (0.16 mmol) in 50 mL of xylene to afford only 0.01 mmol of PhH, 0.06 mmol of PhCHO, 0.04 mmol of PhCOSPh (phenyl thiobenzoate), 0.23 mmol of PhSSPh, and two as yet unidentified species with molecular weights of 134 and 204 (GC–MS, ca 0.03 and 0.02 mmol, respectively).[18] The sum of the yields of desulfurized compounds is approximately equal to the amount of **1** reacted; therefore, the desulfurization does not appear to be catalytic, but the formation of PhSSPh does. This reaction is under further investigation.

tert-Butyl isothiocyanate reacts with **1** in refluxing toluene to give, after 3 h, two products, **3** and **4**, in a 3:2 ratio, that are derived from **2** by substitution of the carbonyl groups by one or two *t*-BuNC ligands, respectively:[19]

$$\underset{\mathbf{1}}{\mathrm{Cp'_2Mo_2Co_2S_3(CO)_4}} + t\text{-BuNCS} \rightarrow \underset{\mathbf{3},\ x = 1;\ \mathbf{4},\ x = 2}{\mathrm{Cp'_2Mo_2Co_2S_4(CO)_{2-\mathit{x}}(CN\text{-}\mathit{t}\text{-}Bu)_{\mathit{x}}}}$$

Sulfur abstraction from isothiocyanates has been observed previously.[20-22]

In summary, it is possible to desulfurize thiophene and thiophenol through the use of a sulfido bimetallic cluster in reactions analogous to those over single-crystal surfaces and in some respects to the Co/Mo/S-catalyzed HDS process. Further studies into the mechanism of these reactions should provide insight into the mechanism of HDS catalysis.

Acknowledgment. This work was supported by the National Science Foundation (Grant No. CHE-8619864).

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(13) Aryl sulfides are desulfurized with BH_4^- and NiB catalyst (Back, T. S.; Yang, K. *J. Chem. Soc., Chem. Commun.* **1990**, 819) or TiH sponge (from $TiCl_4$ and $LiAlH_4$) (Mukaiyama, T.; Hayashi, M.; Narasaka, K. *Chem. Lett.* **1973**, 291) or $LiAlH_4$ with soluble Ni catalysts (Eisch, J. J.; Im, K. R. *J. Organomet. Chem.* **1977**, *139*, C51). Thiophenes are converted to conjugated dienes with phenylmagnesium bromide and Ni catalysts: Wenkert, E.; Leftin, M. H.; Michelotti, E. L. *J. Chem. Soc., Chem. Commun.* **1984**, 617. RS groups of allylic sulfides, C_3H_5SR, may be replaced by nucleophiles with transition-metal catalysts: Okamura, H.; Takei, H. *Tetrahedron Lett.* **1979**, 3425. Osakada, K.; Chiba, T.; Nakamura, Y.; Yamamoto, T.; Yamamoto, A. *J. Chem. Soc., Chem. Commun.* **1986**, 1589. $Mo(CO)_6$ in acetic acid or on AL_2O_3 desulfurizes organic sulfides, but the metal product is unknown: Alper, H.; Blais, C. *J. Chem. Soc., Chem. Commun.* **1980**, 169.

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(18) The reaction was conducted in a glass-lined Parr reactor. *n*-Heptane was added as an internal GC standard for the quantification of volatiles in solution. The PhSSPh and PhCOSPh were sublimed from the reaction mixture after removal of solvent and volatile components and analyzed by GC–MS.

(19) $Cp'_2Mo_2Co_2S_3(CO)_4$ (0.095 g, 0.040 mmol) was dissolved in 40 mL of toluene, and *t*-BuNCS (0.21 mL, 1.66 mmol) was added. The solution was refluxed under N_2 for 8 h. After removal of the solvent, a ^1H NMR spectrum of the brown solid in C_6D_6 showed a 3:2 ratio of $Cp'_2Mo_2Co_2S_4(CO)(t\text{-}BuNC)$–$Cp'_2Mo_2Co_2S_4(t\text{-}BuNC)_2$. The compounds were identified by comparison of their spectra with those of authentic compounds prepared from the cubane, **2**, and *t*-BuNC. For **3**: ^1H NMR δ 5.31 (m, 2), 5.26 (m, 2), 5.01 (m, 2), 4.98 (m, 2), 1.83 (s, 6), 0.88 (s, 9); IR 2111 m, 1953 s. For **4**: ^1H NMR δ 5.46 (m, 4), 5.11 (m, 4), 2.00 (s, 6), 0.88 (s, 18); IR 2101, 2071 cm^{-1}; MS(FAB), $[P]^+ = 762$.

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Catalytic Asymmetric Arylation of 2,3-Dihydrofuran with Aryl Triflates

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There has been considerable recent interest in the use of Heck-type arylation and alkenylation of olefins for constructing carbon skeletons of biologically important organic compounds.[1,2] Development of "asymmetric Heck reactions" where the carbon–carbon bond formation proceeds with high enantioselectivity would provide new efficient routes to the optically active compounds. Shibasaki and Overman have recently reported their pioneering works on the asymmetric Heck reactions, where in-

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(1) Recent examples and leading references: (a) Torii, S.; Okumoto, H.; Akahoshi, F.; Kotani, T. *J. Am. Chem. Soc.* **1989**, *111*, 8932. (b) Larock, R. C.; Fried, C. A. *J. Am. Chem. Soc.* **1990**, *112*, 5882. (c) Larock, R. C.; Gong, W. H. *J. Org. Chem.* **1990**, *55*, 407. (d) Larock, R. C.; Gong, W. H.; Baker, B. E. *Tetrahedron Lett.* **1989**, *30*, 2603. (e) Larock, R. C.; Baker, B. E. *Tetrahedron Lett.* **1988**, *29*, 905. (f) Larock, R. C.; Song, H.; Baker B. E.; Gong, W. H. *Tetrahedron Lett.* **1988**, *29*, 2919. (g) Zhang, Y.; Negishi, E. *J. Am. Chem. Soc.* **1989**, *111*, 3454. (h) Zhang, Y.; O'Connor, B.; Negishi, E. *J. Org. Chem.* **1988**, *53*, 5588. (i) Negishi, E.; Zhang, Y.; O'Connor, B. *Tetrahedron Lett.* **1988**, *29*, 2915. (j) Nilsson, K.; Hallberg, A. *J. Org. Chem.* **1990**, *55*, 2464. (k) Mori, M.; Kubo, Y.; Ban, Y. *Tetrahedron* **1988**, *44*, 4321. (l) Grigg, R.; Stevenson, P.; Worakun, T. *Tetrahedron* **1988**, *44*, 2049. (m) Daves, G. D., Jr.; Hallberg, A. *Chem. Rev.* **1989**, *89*, 1433.

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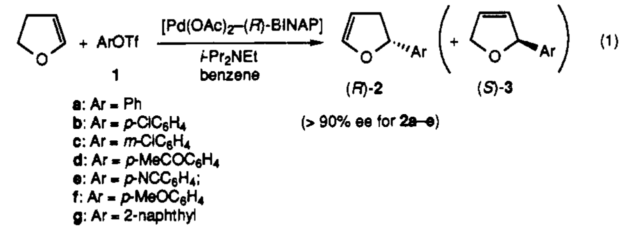
Table I. Asymmetric Heck Reaction of 2,3-Dihydrofuran with Aryl Triflates Promoted by $Pd(OAc)_2$–(*R*)-BINAP Catalyst[a]

ArOTf (**1**)	Ar	time, h	yield,[b] %			% ee[e,f]	
			(*R*)-**2**	(*S*)-**3**	**2**/**3**[d]	(*R*)-**2**	(*S*)-**3**
a	Ph	66	71[c]	7[c]	89/11	93	67
b	*p*-ClC_6H_4	22	(86)		83/17	91	47
c	*m*-ClC_6H_4	29	(82)		82/18	93	56
d	*p*-$MeCOC_6H_4$	24	(76)		80/20	93	53
e	*p*-NCC_6H_4	26	(79)		77/23	93	46
f	*p*-$MeOC_6H_4$	72	(72)		72/28	73	66
g	2-naphthyl	48	(42)		71/29	76	*g*

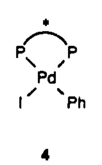
[a] The reaction was carried out in benzene in the presence of diisopropylethylamine under a nitrogen atmosphere. ArOTf/2,3-dihydrofuran/*i*-$Pr_2NEt/Pd(OAc)_2$/BINAP = 1.0/5.0/3.0/0.03/0.06. Reaction temperature: 30 °C (**1a**); 40 °C (**1b–g**). [b] The products were isolated as a mixture of regioisomers **2** and **3** by bulb-to-bulb distillation unless otherwise noted. The total yields of **2** and **3** are given in parentheses. [c] Isolated by column chromatography. [d] The ratio was determined by GLC. [e] Determined by ^1H NMR spectroscopy using optically active NMR shift reagent $Eu(hfc)_3$ after separation of **2** and **3** by preparative GLC. [f] Optical rotation ($[\alpha]^{20}_D$ (*c* 0.6–1.4, $CHCl_3$)): −64.3° (**2a**), −175° (**3a**), −64.8° (**2b**), −128° (**3b**), −49.5° (**2c**), −86.3° (**3c**), −45.7° (**2d**), −134° (**3d**), −40.7° (**2e**), −100° (**3e**), −75.7° (**2f**), −114° (**3f**). [g] Not measured.

tramolecular cyclization provides a new tertiary carbon center of around 45% ee.[2,3] We report here that high enantioselectivity over 90% ee was achieved in catalytic arylation of 2,3-dihydrofuran when aryl triflates (aryl trifluoromethanesulfonates) (**1**) were used as arylating agents.



(1)

In our initial attempts, we applied chiral diphosphine ligands to the Larock's arylation system of cyclic olefins using aryl iodides,[1c–e] but no or little asymmetric induction has been observed. For example, the reaction of 2,3-dihydrofuran with phenyl iodide in THF containing triethylamine and 3 mol % of $Pd(OAc)_2$–(*R*)-BINAP[4] catalyst at 40 °C for 44 h gave racemic 2-phenyl-2,3-dihydrofuran (**2a**) and 2-phenyl-2,5-dihydrofuran (**3a**) in 23% and 2% yields, respectively. This reaction must involve [PhPd(I)(BINAP)] intermediate (**4**), which is formed by oxidative addition of phenyl iodide to a Pd(0)–BINAP species generated in the system.[5] Coordination of dihydrofuran to **4** and the subsequent insertion and β-hydrogen elimination processes provide arylation products **2a** and **3a**. Recent mechanistic studies on olefin insertion into phosphine-coordinated acylpalladium halide complexes ($RCOPdXL_2$) revealed that the insertion process is preceded by displacement of one of the phosphine ligands (L) with olefin to give an [RCOPd(olefin)XL]-type intermediate.[6] Therefore, we reasoned that the low enantioselectivity in the reaction with phenyl iodide is attributable to partial dissociation of BINAP ligand during the olefin-insertion process.



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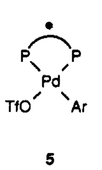

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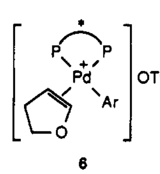

6

For overcoming this problem, we planned to employ an arylpalladium triflate intermediate [ArPd(OTf)(BINAP)] (**5**), which may be generated in the catalytic system by oxidative addition of aryl triflate to a Pd(0) species. Complex **5** having triflate ligand as a good leaving group was expected to form a cationic olefin-coordinated species $[ArPd(olefin)(BINAP)]^+OTf^-$ (**6**) without involving the partial dissociation of BINAP ligand, leading to the highly enantioselective Heck reaction.[7]

Table I demonstrates that the catalytic arylation of 2,3-dihydrofuran with aryl triflates (**1a–g**) actually proceeded in high enantioselectivity (eq 1).[8] Thus, the reaction of phenyl triflate (**1a**) in the presence of 3 mol % of a palladium catalyst prepared in situ from $Pd(OAc)_2$ and (*R*)-BINAP gave (*R*)-**2a** ($[\alpha]^{20}_D$ −64.3° (*c* 1.2, $CHCl_3$)) of 93% ee in 71% yield, together with a small amount (7%) of (*S*)-**3a** (67% ee, $[\alpha]^{20}_D$ −175° (*c* 1.3, $CHCl_3$)).[9] The high enantioselectivity in the formation of (*R*)-**2** was observed in the reaction performed with diisopropylethylamine as a base and benzene as a solvent.[11]

A striking feature of this asymmetric reaction is that the arylation products **2** and **3** have opposite configurations to each other, indicating that a kinetic resolution process enhances the enantiomeric purity of (*R*)-**2** by selective elimination of the S isomer as minor product **3** from the catalytic cycle. Insertion of dihydrofuran into the Ar–Pd bond in **5** followed by β-hydrogen elimination forms hydrido–olefin complex **8** or its diastereoisomer **8′**. It is likely that complex **8** has a preferable structure for further olefin insertion and β-hydrogen elimination processes giving major product (*R*)-**2**, whereas diastereoisomer **8′** readily releases the coordinated olefin ((*S*)-**3**). The overall process gives rise to highly enantioselective formation of (*R*)-**2**.

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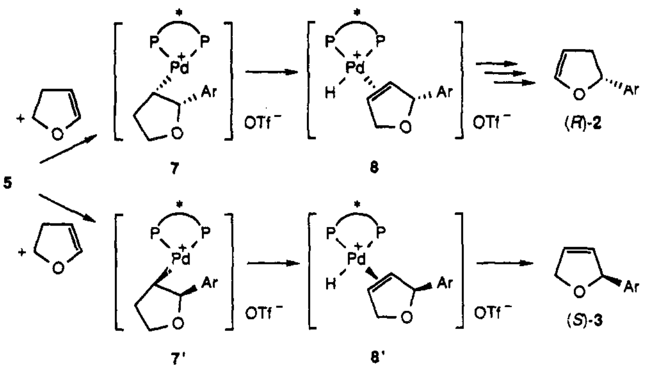
(9) The enantiomeric purities of **2** and **3** were determined by ^1H NMR analysis with optically active NMR shift reagent $Eu(hfc)_3$. The absolute configuration of (−)-**2a** (84% ee) was determined by converting it with Jones reagent into known γ-butyrolactone derivative, (*R*)-(+)-3-phenyl-2-oxacyclopentanone (**9a**, 84% optical purity, $[\alpha]^{20}_D$ +27.4° (*c* 1.1, $CHCl_3$)).[10] The absolute configuration of (−)-**3a** was determined as follows. Hydrogenation of (*R*)-(−)-**2a** (93% ee) with Wilkinson catalyst in benzene at room temperature under an atmospheric pressure of dihydrogen gave (*R*)-(+)-2-phenyltetrahydrofuran (**10a**, $[\alpha]^{20}_D$ +45.0° (*c* 1.1, $CHCl_3$)), while hydrogenation of (−)-**3a** (59% ee) under similar conditions gave (*S*)-(−)-**10a** ($[\alpha]^{20}_D$ −17.0° (*c* 0.92, $CHCl_3$)). The absolute configurations of **2b–g** and **3b–f** were assigned by similarity in shifts using the chiral shift reagent.

(10) (*S*)-(−)-**9a** ($[\alpha]_D$ −32.5° (*c* 4.3, $CHCl_3$)): Manzocchi, A.; Casati, R.; Fiecchi, A.; Santaniello, E. *J. Chem. Soc., Perkin Trans. 1* **1987**, 2753.

(11) Optical purities of (*R*)-**1a** formed in the reactions of phenyl triflate (0.64 mmol), 2,3-dihydrofuran (3.2 mmol), and Et_3N (1.9 mmol) in various solvents (2 mL) in the presence of 3 mol % of $Pd(OAc)_2$–(*R*)-BINAP catalyst at 40 °C are as follows: benzene (71% ee), THF (67% ee), CH_2Cl_2 (57% ee), and DMF (63% ee).



Acknowledgment. This work was supported by a Grant-in-Aid for Scientific Research (Grant No. 02453089) and a Special Grant-in-Aid for Promotion of Education and Science in Hokkaido University from the Ministry of Education, Science and Culture, Japan. Partial financial support from Sapporo Bioscience Foundation is gratefully acknowledged.

Supplementary Material Available: Experimental details and spectroscopic data for **2a–g**, **3a–f**, **9a**, and **10a** (4 pages). Ordering information is given on any current masthead page.

Control of Back Electron Transfer from Charge-Transfer Ion Pairs by Zeolite Supercages

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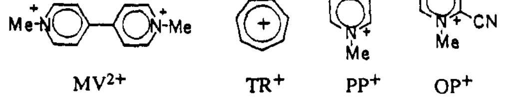
Charge-transfer activation of the precursor complexes[1] from nucleophiles (D) and electrophiles (A) is a viable formulation for a variety of organic and organometallic reactions, i.e.,

$$D + A \rightleftharpoons [D,A] \underset{k_{-1}}{\overset{CT}{\rightleftharpoons}} [D^{\bullet+},A^{\bullet-}] \xrightarrow{k_2} \text{etc.} \qquad (1)$$

where CT includes both photochemical ($h\nu_{CT}$) and thermal (adiabatic) processes.[2] The critical assessment of charge-transfer efficiency depends largely on the rate of back electron transfer (k_{-1}) relative to that of the followup reaction (k_2), especially as evaluated in photoactivated systems.[3] Since the available methodology for the general control of back electron transfer is limited,[4] we describe in this report how the zeolite supercage can be used to strongly modulate the magnitude of k_{-1}.

The stepwise assembly of various charge-transfer complexes directly within the zeolite (Y) supercage has recently been achieved by the prior ion exchange of NaY with the different cationic electrophiles (A^+) MV^{2+}, TR^+, PP^+, and OP^+ followed by exposure of the doped zeolite to arene donors (D = Ar) dissolved in organic solvents.[5] Importantly the correlation of the charge-transfer bands in Figure 1 establishes the intermolecular complexes $[A^+,Ar]$ that are formed within the zeolite-Y supercage to be the same as those produced in solution.[6] Indeed the latter relate to the cofacial pairs of A^+ and Ar in the π-complexes (established by X-ray crystallography[7]) that can be accommodated



MV^{2+} TR^+ PP^+ OP^+

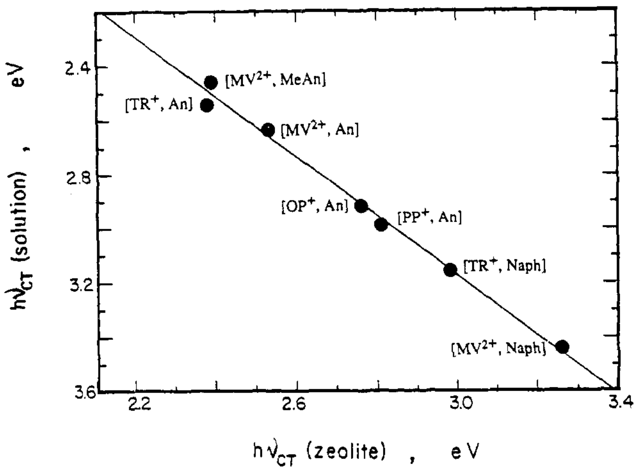


Figure 1. Charge-transfer spectra ($h\nu_{CT}$) of various charge-transfer complexes (as indicated) in zeolite-Y supercages by diffuse reflectance relative to the absorption spectra of the same complexes in acetonitrile solution.

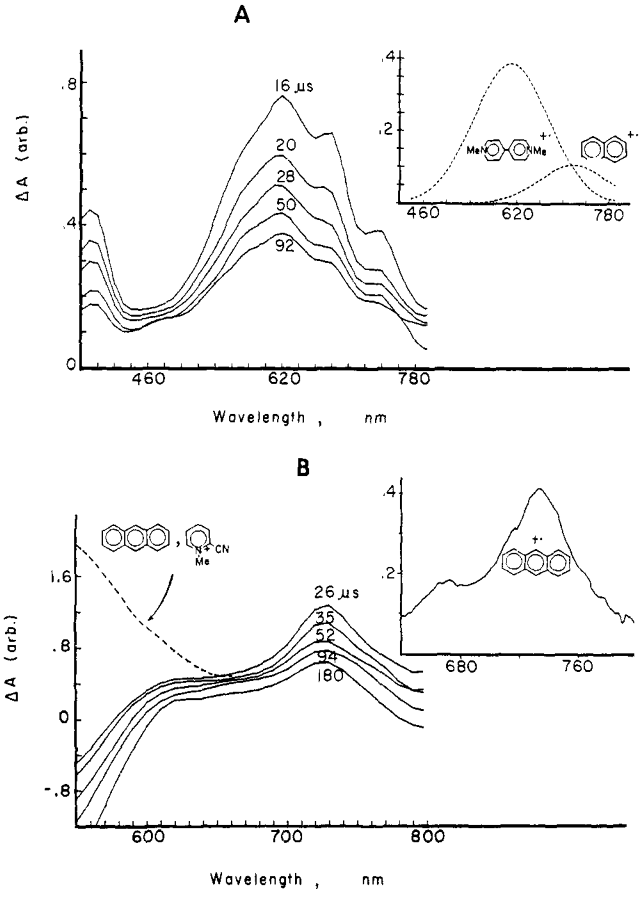


Figure 2. Time-resolved diffuse reflectance spectra obtained at microsecond intervals following the 10-ns laser pulse at (A) 355 nm of $[MV^{2+},Naph]$ and (B) 532 nm of $[OP^+,An]$ immobilized in zeolite-Y. The insets show the transient spectra of the ion-radicals $MV^{\bullet+}$ (smoothed), $Naph^{\bullet+}$, and $An^{\bullet+}$ in acetonitrile solutions.

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[6] See, e.g.: (a) Nakahara, A.; Wang, J. H. *J. Phys. Chem.* **1963**, *67*, 496. (b) Hamity, M.; Lema, R. H. *Can. J. Chem.* **1988**, *66*, 1552. (c) Atherton, S. J.; Hubig, S. M.; Callan, T. J.; Duncanson, J. A.; Snowden, P. T.; Rodgers, M. A. J. *J. Phys. Chem.* **1987**, *91*, 3717 and references therein.

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