established from the NMR data. Accordingly, the crystal structures of the vinyl complexes derived from 2-butyne, **3**, and from di-p-tolylacetylene, **4**, were determined. Both vinyl derivatives had trans stereochemistry (Figure 1). There are precedents for bridging vinyl ligands in clusters but all, crystallographically defined, have cis stereochemistry.[5]

One equivalent of hydrogen converted the vinyl complexes to the corresponding trans olefin and the original dimer, **1**. In a catalytic mode, the product was the trans olefin (see the degradation sequence described below). With the vinyl complexes as the catalyst, the results were the same. Our data provide a firm basis for the catalytic sequence presented in Figure 2. The rate-determining step appears to be hydrogen addition to the vinyl intermediate since the catalytic reaction rate is a sensitive function of H_2 pressure. Olefin elimination directly from this vinyl intermediate does not appear to be a kinetically significant process, at least for diarylacetylenes in either the presence or absence of H_2.

Unfortunately, alkynes also react with the bridged vinyl intermediate.[6] This process, competitive with hydrogen addition to form the trans olefin, leads to the degradation of the dinuclear complex within 5 min under catalytic conditions. The details of the chemistry that ensues vary with the nature of the acetylene; for brevity, the description here is limited to diphenylacetylene chemistry. Addition of the latter to the vinyl complex forms the mononuclear, square-planar complex[7] $Rh[\pi-(H)(R^1)C=C-(R^2)C(R^3)=C(R^4)][P(O-i-C_3H_7)_3]_2$ (**5**), which has been crystallographically defined (details of which will be presented in a separate article) and which has the R^1 and R^2 aryl groups trans and R^3 and R^4 cis. This latter complex was shown to be a catalyst precursor for the hydrogenation of diphenylacetylene to cis-stilbene. Thus, alkyne hydrogenation initiated by **1** transforms from selective trans-olefin to selective cis-olefin formation. Attempts to prevent the effective alkyne competition for the vinyl intermediate by raising the hydrogen pressure from 1 to 100 atm were only partially successful (the degradation rate was suppressed but the overall rate was so greatly enhanced that all alkyne was consumed in ~60 s, and under these conditions, substantial amounts of the first formed olefins were converted to alkanes).

Alkyne hydrogenations catalyzed by other dinuclear and also polynuclear complex precursors have been described,[8-12] but in all these reported systems, cis olefins have been the main products. However, the nuclearity of the actual catalyst intermediates was not defined in these systems although labeling studies[8] for (η^5-$C_5H_5)_2Mo_2(CO)_4(\eta^2-\mu-RC_2R)$ indicated that fragmentation of the dimer was not a significant process. In any case, the presence of reactive, adjacent metal centers is not a sufficient condition[1] for trans-olefin formation in alkyne hydrogenation—the stereochemical outcome obviously depends on the intimate stereochemistry of the intermediates in the catalytic cycle. The precise electronic and steric factors that govern stereochemistry[1] in the formation of the bridging vinyl ligand are not evident from available data. Having demonstrated a principle concerning catalysis at two adjacent metal centers and having reasonably outlined the mechanistic character of the catalytic cycle, we now

seek a robust trans-olefin catalytic system by dispersing and supporting dinuclear metal complexes on metal oxides.

Reduction of CH_3NC and CH_3CN by the Reduced Species of $[Fe_4S_4(SPh)_4]^{2-}$ and $[Mo_2Fe_6S_8(SPh)_9]^{3-}$: Model Reactions to Nitrogenase

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Recently, we reported that C_2H_2 is reduced by the electrochemically reduced species of $[Fe_4S_4(SPh)_4]^{2-}$ ($[4-Fe]^{2-}$)[1] or $[Mo_2Fe_6S_8(SPh)_9]^{3-}$ ($[Mo-Fe]^{3-}$)[2] catalytically in MeOH/THF to give C_2H_4 selectively without evolving H_2 gas and that C_2D_2 is reduced by the same catalyst in H_2O at pH 6.0 to afford cis-$C_2D_2H_2$ stereoselectively.[3] The close similarity of these reactions to the nitrogenase reaction has driven us to study the reduction of CH_3NC and CH_3CN by the same catalysts. Isonitrile and nitrile molecules seem to be more practical substrates than acetylene for nitrogenase model reactions, since the reductions of CH_3NC to CH_4 and CH_3NH_2[4-9] and of CH_3CN to C_2H_6 and NH_3[8-10] require six electrons as in the reduction of N_2 to NH_3. In the reduction of RNC and RCN catalyzed by some molybdenum complexes reported so far,[11-13] the amounts of CH_3NH_2 and NH_3 formed have not been determined at all. This com-

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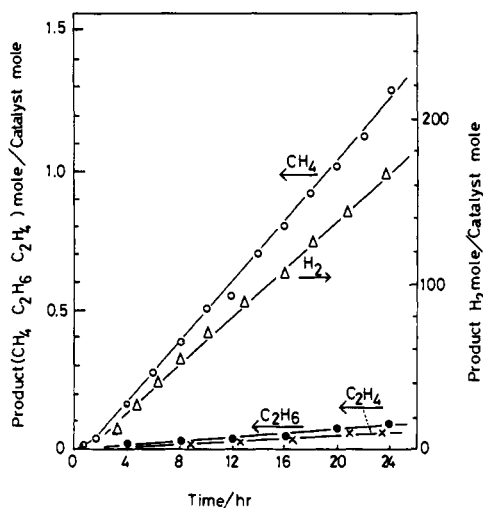(13) Schrauzer, G. N. *Angew. Chem., Int. Ed. Engl.* **1975**, 14, 514.

Communications to the Editor

J. Am. Chem. Soc., Vol. 104, No. 15, 1982 4259

Figure 1. Plots of the amount of products vs. time in the reaction of CH_3NC with $[4\text{-Fe}]^{4-}$ in CH_3OH/THF.

Table I. Relative Amounts[a] of the Products Formed in the Reduction of CH_3NC Catalyzed by $[4\text{-Fe}]^{n-}$ ($n = 3, 4$) or $[Mo\text{-Fe}]^{5-}$ in CH_3OH/THF

	catalyst[b]		
product	$[4\text{-Fe}]^{4-}$	$[4\text{-Fe}]^{3-}$	$[Mo\text{-Fe}]^{5-}$
CH_4	1	1	1
CH_3CH_3	0.050	2.7	0.49
CH_2CH_2	0.033	0	0
H_2	1.4×10^2	4×10^3	2×10^3

[a] The amount of CH_4 produced in each reaction was taken as unity. [b] Relative rates of the reduction by $[4\text{-Fe}]^{4-}$, $[4\text{-Fe}]^{3-}$, and $[Mo\text{-Fe}]^{5-}$ were 17:1.0:1.1 with respect to the amount of CH_4 produced.

munication describes the catalytic reduction of CH_3NC and CH_3CN by the electrochemically reduced species of $[4\text{-Fe}]^{2-}$ or $[Mo\text{-Fe}]^{3-}$ in MeOH/THF and in H_2O as novel nitrogenase model reactions.

The reduction of CH_3NC and CH_3CN was carried out under controlled potential electrolysis conditions with a Hg working electrode at the reduction potentials of $[4\text{-Fe}]^{2-}$ (2–/3–, –1.25 V, 3–/4–, –1.65 V)[14] or $[Mo\text{-Fe}]^{3-}$ (4–/5–, –1.25 V)[14] in a MeOH/THF (1:1 v/v, 40 cm³) solution or in an aqueous suspension[15] containing the $n\text{-Bu}_4N^+$ salt of $[4\text{-Fe}]^{2-}$ (39 μmol) or $[Mo\text{-Fe}]^{3-}$ (34 μmol) and CH_3NC (17 mmol) or CH_3CN (34 mmol). Lithium chloride (24 mmol) and a $NaOH\text{-}H_3PO_4$ buffer were used as supporting electrolytes in the MeOH/THF solutions and aqueous suspensions, respectively. The reaction cells consisted of three compartments: a Hg working electrode and a platinum auxiliary electrode, which were separated by a glass frit, and a SCE reference electrode.[16]

The reduction product of CH_3NC by $[4\text{-Fe}]^{4-}$ in MeOH/THF consists of CH_4 and small amounts of C_2H_6 and C_2H_4 (Figure 1) together with a trace of C_3H_8, similar to the reduction by nitrogenase, though the concomitant H_2 evolution in the present reaction is large compared with that in the nitrogenase reaction ($H_2/CH_4 \simeq 3$).[5] There is seen an almost linear relation between the amount of the reaction products and the reaction time after the initial induction period of 1 h (Figure).[17] The number of moles of CH_4 evolved in 24 h reached ca. 130% of that of $[4\text{-Fe}]^{2-}$ present in the working electrode cell, indicating that the reduction proceeds catalytically.[18] Similar results were obtained also in the reduction
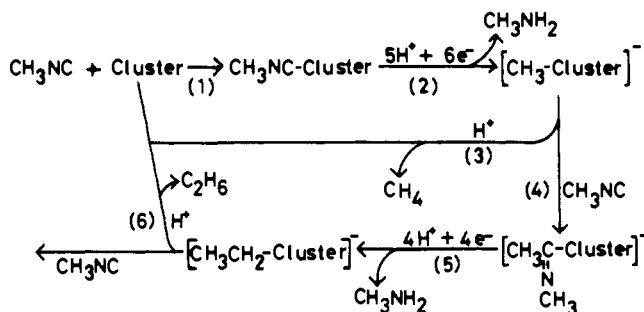
(14) The potentials in MeOH/THF (1:1 v/v).
(15) The same potentials as in MeOH/THF were applied for an aqueous suspension.
(16) The volumes of the solution placed in the working, auxiliary, and reference electrode cells are 16, 16, and 8 cm³, respectively.
(17) A similar induction period was observed also in the reduction of C_2H_2 (ref 3).

Scheme I

of CH_3NC catalyzed by $[4\text{-Fe}]^{3-}$ and $[Mo\text{-Fe}]^{5-}$. Relative amounts of the products are summarized in Table I, which indicates that CH_4 is a major product in the reactions catalyzed by $[4\text{-Fe}]^{4-}$ and $[Mo\text{-Fe}]^{5-}$, while $[4\text{-Fe}]^{3-}$ affords C_2H_6 as a main product. The formation of C_2H_6 from CH_3NC may result from the insertion reaction of CH_3NC into the $[CH_3\text{-cluster}]^-$, as shown in the Scheme I, which has been modified from that of the RNC reduction by nitrogenase.[9] The first step in the scheme is adduct formation between CH_3NC and the cluster. The formation of such an adduct in solution has already been characterized for the $[Fe_4S_4(SR)_4]^{2-}\text{-R'NC}$ system (R = C_2H_5, $C_6H_5CH_2$; R' = $t\text{-}C_4H_9$, $n\text{-}C_4H_9$).[19] The second step stands for the reduction of coordinated CH_3NC, which requires 5 H^+ and 6 e^-, producing CH_3NH_2 and $[CH_3\text{-cluster}]^-$, the latter of which may readily undergo an insertion reaction of either H^+ (step 3) or additional CH_3NC (step 4). The insertion of H^+ produces CH_4, while that of CH_3NC affords $[CH_3C(=NCH_3)\text{-cluster}]^-$, which can give C_2H_6 in the subsequent reactions (steps 5 and 6). Thus, the ratio of the amount of CH_4 and C_2H_6 produced from CH_3NC may primarily depend on the relative ease of the insertion reactions of H^+ (step 3) and CH_3NC (step 4). It is therefore suggested that the rate of the insertion of CH_3NC is faster than that of H^+ in the reaction catalyzed by $[4\text{-Fe}]^{3-}$ and vice versa by $[4\text{-Fe}]^{4-}$ and $[Mo\text{-Fe}]^{5-}$.

When CH_3OH/THF is used as solvent, the other reduction product CH_3NH_2 reacts with HCHO, which is one of the oxidation products of MeOH at the counter Pt electrode,[20] giving a variety of amines and related compounds such as $(CH_3)_2NH$ and CH_3NHCH_2OH[21] under the present experimental conditions. So that the net amount of CH_3NH_2 produced in the catalytic reaction could be determined, the electrolysis of CH_3NC was carried out at –1.65 V (vs. SCE) for an aqueous suspension of $[4\text{-Fe}]^{2-}$ at pH 7.2.[22] The mole ratios of the reaction products in this case were $CH_4/C_2H_6/C_2H_4/H_2 = 1.0\text{:}0.29\text{:}0.16\text{:}320$, which are not very different from those obtained in MeOH/THF (Table I). The amount of CH_3NH_2 was about 10 times larger than the number of moles of the hydrocarbon formed. The total amount of hydrocarbons and CO_2 detected in the gas phase, however, is in harmony with the amount of CH_3NH_2. Aryl- or alkylisonitriles are known to react with $[Fe_4S_4L_4]^{n-}$ (L = SC_2H_5, $SCH_2C_6H_5$, etc; $n = 2, 4$) in the presence of excess alkyl mercaptan to give various thioformidates,[19] which are readily hydrolyzed to yield aryl- or alkylamines. In addition, alkylisonitriles undergo hydrolysis in acidic media to give alkylamines and HCOOH in equimolar amounts.[23] Carbon dioxide produced in the present

(18) The addition of CH_3NC to $[4\text{-Fe}]^{2-}$ in MeOH/THF (1:1 v/v) weakened the absorption band due to $[4\text{-Fe}]^{2-}$ centered at 450 nm (ε 17 000 M⁻¹ cm⁻¹); see ref 19. The spectrum of $[4\text{-Fe}]^{2-}$ has, however, not changed between before and after the controlled potential electrolysis at –1.65 V for 24 h, suggesting that the Fe_4S_4 core essentially remains without decomposition during electrolysis.
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reaction may, therefore, result from the oxidation of HCOOH, which would be produced in the course of reactions, because the reaction of [4-Fe]$^{2-}$ with excess HCOOH in an aqueous suspension of pH 7.0 at room temperature has evolved a stoichiometric amount of CO_2 (based on [4-Fe]$^{2-}$) for 4 h. This result suggests that the [4-Fe]$^{2-}$ cluster is a possible model compound to formate dehydrogenases.[24]

Acetonitrile was similarly reduced in an aqueous suspension[25] of [4-Fe]$^{4-}$ or [Mo–Fe]$^{5-}$ formed electrochemically at pH 7.0 to produce C_2H_6, C_2H_4 (0.1 or 0.05 to C_2H_4), and NH_3. The amount of NH_3 produced in the reaction was 95% or 96% of the number of total moles of C_2H_6 and C_2H_4, and no $C_2H_5NH_2$ has been detected in the reaction systems. In addition, [4-Fe]$^{4-}$ and [Mo–Fe]$^{5-}$ were completely inactive to the reduction of $C_2H_5NH_2$ under the same conditions. These results indicate that the reduction of CH_3CN in the present reaction does not proceed by a stepwise mechanism, which requires two electrons in each step and should produce $C_2H_5NH_2$ as an intermediate.

Registry No. CH₃NC, 593-75-9; [4-Fe]$^{4-}$, 66213-39-6; [4-Fe]$^{3-}$, 52627-89-1; [Mo–Fe]$^{5-}$, 76125-83-2.

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Bis[bis(trifluoromethanesulfonyl)imido]xenon: A New Compound Possessing Xenon–Nitrogen Bonds

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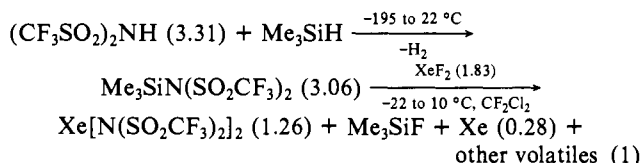*Clemson, South Carolina 29631*
Received March 22, 1982

Since the first report of the synthesis of a stable xenon–nitrogen compound,[2] no other compound of this type has been successfully prepared with a different nitrogen ligand. Until now, only $FXeN(SO_2F)_2$, $Xe[N(SO_2F)_2]_2$, and $[(FSO_2)_2NXe]_2F^+AsF_6^-$ were known.[3,4] In this communication, we report a new xenon–nitrogen compound utilizing a new ligand designed expressly for this purpose.

Reflection on the properties of the $N(SO_2F)_2$ group led us to conclude that a likely ligand for formation of a bond to xenon would be $N(SO_2CF_3)_2$ from the bis(perfluoroalkylsulfonyl)imides, $HN(SO_2R_f)_2$.[5] Several examples of these were known, but the parent member of the series, $(CF_3SO_2)_2NH$, had not been isolated. This compound was of particular interest because of the obvious comparisons one can make with $(FSO_2)_2NH$ and the fact that the CF_3SO_2 group is probably the most electron-withdrawing group in chemistry. This acid was subsequently prepared by us[6,7] and found to give a xenon-containing product upon reaction with XeF_2. This product was postulated to be $Xe[N(SO_2CF_3)_2]_2$, but there were several problems in characterizing the material. The same xenon-containing species was observed regardless of initial reaction stoichiometry. Attempts at the synthesis of a 1:1 product only resulted in poorer yields of the postulated disubstituted compound. The best xenon analyses were only 80-90% of the expected values. The product always seemed to be contaminated
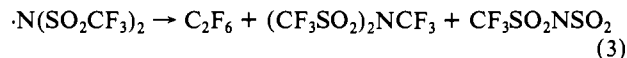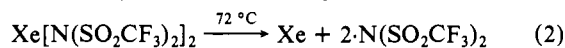
with the starting acid, and the best yields never exceeded 50%.

As an alternate approach to obtaining the pure compound, we investigated the use of a trimethylsilyl group to replace the acid proton. An expected advantage of this approach was the elimination of product HF, which was thought to be a major problem in promoting side reactions. Reaction of $(CF_3SO_2)_2NH$ with an excess of trimethylsilane afforded the appropriate reagent. Reaction of the silylimide with XeF_2 afforded the desired compound in high yield, as shown in the eq 1 (mmol).

$$(CF_3SO_2)_2NH \ (3.31) + Me_3SiH \xrightarrow[-H_2]{-195 \ to \ 22 \ ^\circ C}$$
$$Me_3SiN(SO_2CF_3)_2 \ (3.06) \xrightarrow[-22 \ to \ 10 \ ^\circ C, \ CF_2Cl_2]{XeF_2 \ (1.83)}$$
$$Xe[N(SO_2CF_3)_2]_2 \ (1.26) + Me_3SiF + Xe \ (0.28) +$$
$$\text{other volatiles} \quad (1)$$

The other products include unreacted XeF_2 and small amounts of N_2-, CF_4-, and SO_2-containing materials. The yield of the xenon compound is 75% starting from the acid, including purification of the intermediate silyl compound via pumping under dynamic vacuum for 0.5 h at 22 °C. The xenon product is purified by pumping under dynamic vacuum at 22 °C for 3 h. This illustrates the first successful use of a silyl derivative to form a bond to xenon. The reactivity can be compared to $B(OTeF_5)_3$ in the preparation of TeF_5O derivatives of xenon from xenon fluorides. The Si–N and B–O bonds have comparable reactivity and Me_3SiF and BF_3 are relatively innocuous byproducts.[8]

$Xe[N(SO_2CF_3)_2]_2$ is a fine white solid, stable at 22 °C under nitrogen or vacuum for several days without appreciable decomposition. A small sample exposed to the air decomposed after ~1 h. A quantitative xenon analysis for the formula $Xe[N(SO_2CF_3)_2]_2$ was realized after heating 0.327 g in a glass vessel to 72 °C, at which point it cleanly and abruptly decomposed. The products obtained were xenon, C_2F_6, a volatile solid and liquid, and a very small amount of a nonvolatile oil. The separation of the Xe from the C_2F_6 was difficult. Repeated transfers through a −172 °C trap gave a xenon value greater than 95 mol%. Mass spectral analysis showed little C_2F_6 in the Xe but considerable Xe in the C_2F_6, and the total Xe is nearer 99%. The volatile liquid was shown to be $(CF_3SO_2)_2NCF_3$ by its mass spectrum (CI, base *m/e* 350 amu) and its ^{19}F NMR. The volatile solid displays a singlet in the ^{19}F NMR, and its IR and mass spectrum indicate it is a form of the previously reported dimer $(CF_3SO_2NSO_2)_2$.[9] No evidence was found for $[N(SO_2CF_3)_2]_2$, a theoretical product.[3] These decomposition products are easily rationalized by eq 2 and 3. The instability of the $\cdot N(SO_2CF_3)_2$ radical compared to

$$Xe[N(SO_2CF_3)_2]_2 \xrightarrow{72 \ ^\circ C} Xe + 2\cdot N(SO_2CF_3)_2 \quad (2)$$
$$\cdot N(SO_2CF_3)_2 \rightarrow C_2F_6 + (CF_3SO_2)_2NCF_3 + CF_3SO_2NSO_2 \quad (3)$$

$\cdot N(SO_2F)_2$[3] is surprising, but this has been confirmed by the photolysis of $ClN(SO_2CF_3)_2$[7] which forms CF_3Cl and $CF_3SO_2-NSO_2$ quantitatively.

The mass spectrum of $Xe[N(SO_2CF_3)_2]_2$ by direct solid inlet shows no parent ion or xenon-containing fragment other than xenon. Analysis of the products evolving from a gradually heated sample by direct gas inlet gave the parent ions of Xe, $CF_3SO_2-NSO_2$, and $CF_3N(SO_2CF_3)_2$ along with the expected fragments as observed in independent spectra of each product.

The Raman spectrum clearly shows the bands of a covalent $(CF_3SO_2)_2N$ group,[10] with no evidence of XeF bonds. However, a band at 826 cm^{-1} is of surprising intensity relative to other covalent $(CF_3SO_2)_2N$ derivatives.

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(8) The use of $B(OTeF_5)_3$ with other xenon fluorides and oxyfluorides is known, as well as with other nonmetal fluorides: Lentz, D.; Seppelt, K. *Angew. Chem., Int. Ed. Engl.* **1978**, *5*, 356. Jacob, E.; Lentz, D.; Seppelt, K.; Simon, A. *Z. Anorg. Allg. Chem.* **1981**, *472*, 7.

(9) Roesky, J. W.; Aramaki, M.; Schonfelder, L. *Z. Naturforsch., B* **1978**, *338*, 1072.

(10) The Raman spectrum in general is very similar to other covalent $(CF_3SO_2)_2NX$ derivatives that we have made, including $(CF_3SO_2)_2NH$ and $(CF_3SO_2)_2NCl$ and dissimilar to ionic derivatives such as $CsN(SO_2CF_3)_2$.