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C−H versus C−N Bond Cleavage Promoted by Niobium(II) Amide

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Summary: Despite their close structural similarity, the potassium and lithium salts of dipyridylamine and diphenylamine [$(C_5H_5N)_2N^-$, Ph_2N^-] react with $(TMEDA)_2Nb_2Cl_5Li(TMEDA)$ in THF to afford two different compounds arising from the cooperative oxidative addition of the two metal centers on either the C−H or C−N bond of the amide ligand.

Oxidative addition performed by transition metals into C−H[1] and C−N[2] bonds are two distinct domains of research which embrace important interest from both application and fundamental points of view. Trivalent Nb and Ta complexes, either isolated[3] or in situ generated,[4] perform a wide variety of transformations (dehydrogenation reactions,[5] C−N[2a−b,i] and CO[6] cleavage, cyclometalation reactions,[7] pyridine ring opening,[2h] hydrogenation reactions,[8] dinitrogen fixation[9]) which can be rationalized in terms of the ability of these reactive d^2 systems to perform oxidative additions. By contrast, the chemistry of divalent niobium compounds is limited to the preparation and characterization of a few salts[10] for which only little information is available about chemical behavior.[3a−b,11]

In this paper, we wish to communicate the results of the reaction of $(TMEDA)_2Nb_2Cl_5Li(TMEDA)$[12,13] with two structurally similar anionic amides, dipyridylamide (Py_2N) and diphenylamide (Ph_2N), which led to oxidative cleavage of both the C−H and C−N bonds of the ligands and resulted in the formation of unique examples of diamagnetic and dinuclear Nb(III) amide derivatives.

Reactions of $(TMEDA)_2Nb_2Cl_5Li(TMEDA)$ with dipyridylamide and diphenylamide (either lithium or potassium salts) were carried out in THF, and crystalline solids of both samples were obtained in moderate yield (30% and 33%, respectively) after suitable work up[14] (Scheme 1). Both compounds were diamagnetic, inevi-

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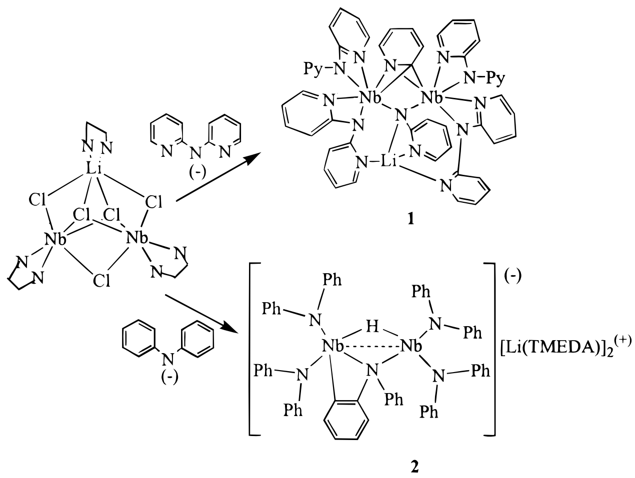
Scheme 1



tably retained lithium, and were correctly analyzed for the formulations elucidated by the X-ray crystal structures.[15]

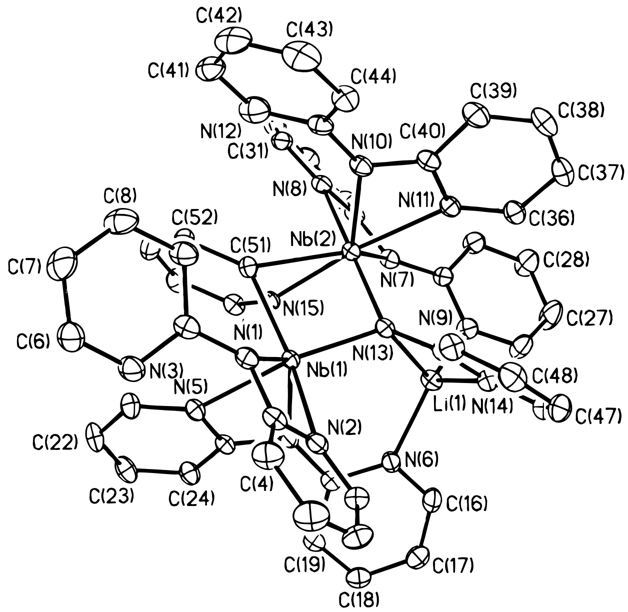


Figure 1. ORTEP drawing of **1**. Thermal ellipsoids are drawn at the 30% probability level. Bond distance are in angstroms (Å) and angles in degrees. Nb(1)−N(13) = 2.011(3), Nb(1)−N(15) = 2.125(3), Nb(1)−C(51) = 2.164(3), Nb(1)−N(1) = 2.184(3), Nb(1)−N(4) = 2.244(3), Nb(1)−N(2) = 2.277(3), Nb(1)−N(5) = 2.315(3), Nb(2)−N(15) = 2.125(3), Nb(2)−C(51) = 2.174(3), Li(1)−N(6) = 2.003(7), N(13)−Nb(1)−C(51) = 97.19(12), C(51)−Nb(1)−N(1) = 91.72(12), C(51)−Nb(1)−N(4) = 120.37(11), C(51)−Nb(1)−N(2) = 150.64(12), N(1)−Nb(1)−N(2) = 59.30(11), C(51)−Nb(1)−N(5) = 89.50(11), N(13)−Nb(2)−N(15) = 94.55(11), N(15)−Nb(2)−C(51) = 38.86(11), N(13)−Nb(2)−N(7) = 108.78(11), C(51)−Nb(2)−N(7) = 121.83(11).

The compound obtained from the first reaction is formulated as $[(py_2N)_2Nb]_2(\mu\text{-}C_5H_4N)(\mu\text{-}Npy)[Li(THF)_2]\cdot(THF)_2\cdot[Li_2Cl_2(THF)_4]$ (**1**) and arises from the ligand C−N bond cleavage. The structure is dinuclear and is formed by two similar $(py_2N)_2Nb$ moieties (Figure 1) bridged by one $[NC_5H_4]$ fragment and one pyridylimido moiety $[(C_5H_4N)N]$. The bridging NC_5H_4 fragment is placed on one side of the Nb−Nb vector and perpendicular between the two metal centers, which, to the best of our knowledge, is an unprecedented type of bonding mode for this moiety. Complex **1** is thermally robust and chemically inert with H_2 at ambient conditions.

Ligand C−H bond cleavage and formation of a metallacycle/hydride was the result of the second reaction. The reaction yielded dinuclear $\{[(Ph_2N)_2Nb]_2[\mu\text{-}NPh(\mu\text{-}\eta^1{:}\eta^2\text{-}C_6H_4)](\mu\text{-}H)\}\{Li\ (TMEDA)_2\}\cdot$toluene (**2**). In this case, two different ionic units (Figure 2) form the dinuclear structure. The anionic moiety is dinuclear with two very similar $(Ph_2N)_2Nb$ moieties connected by one bridging amide group. One of the two phenyl rings of the bridging amide is bent toward both Nb atoms and exhibits two different types of interactions (σ and π) with the two metals. A short bonding contact between one metal center and one of the two ring *ortho* carbon atoms indicates the presence of a Nb−C σ-bond and consequent metalation of the aromatic ring.

The formulation of **2** as a trivalent niobium hydride could not irrefutably be argued from X-ray analysis but rather from its diamagnetism. It should be noticed that

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(14) Complex **1**. A freshly prepared solution of lithium 2,2′-dipyridylamide (1.33 g, 5.5 mmol) in THF (100 mL) was reacted with $(TMEDA)_2Nb_2Cl_5Li(TMEDA)$ (1.0 g, 1.4 mmol). The mixture was stirred for a few minutes and allowed to stand at room temperature for 4 days. Intensely colored dark brown crystals of **1** separated (0.64 g, 0.4 mmol, 30%). Anal. Calcd (Found) for $C_{66}H_{71}N_{15}Nb_2Li_2O_4Cl$: C, 57.71 (57.74); H, 5.21 (5.14); N, 15.30 (15.89). IR [Nujol, cm^{-1}] ν: 1581(s), 1548(s), 1483(sh), 1425(vs), 1377(vs), 1302(s), 1284(s), 1245(s), 1149(s), 1105(m), 1052(s), 1008(s), 989(m), 939(w), 896(m), 877(w), 846(m), 800(m), 771(s), 735(s), 644(m). ^1H NMR [500 MHz, THF-d_8, 23 °C] δ: 8.37(m, 2H), 8.15(m, 4H), 8.12(d, 1H), 7.62(d, 2H), 7.24(m, 2H), 7.21(d, 2H), 6.91 (2 m, 4H), 6.73(3 m, 3H), 6.64(m, 1H,) 6.59(d, 3H), 6.58(m, 2H), 6.35(d, 2H), 6.33(2 m, 4H), 6.11(dd, 2H), 5.71(m, 2H), 5.68(2 m, 3H), 5.42 (t, 1H), 3.55(m, 16H, THF), 1.41(m, 16H, THF). ^{13}C NMR [127.7 MHz, THF-d_8, 23 °C] δ: 201.3 (quaternary) 165.6, 163.5, 163.3, 161.1 (quaternary), 148.7 (quaternary), 147.4, 146.9, 144.9, 137.5, 137.2, 136.9, 136.4, 128.5, 128.3, 128.2, 128.1, 128.0, 127.9, 127.8, 119.8, 115.6, 115.2, 114.0, 113.9, 112.9, 111.3, 111.2, 111.1, 109.0. Complex **2**. A solution of $(TMEDA)_2Nb_2Cl_5Li(TMEDA)$ (1.0 g, 1.4 mmol) in THF (70 mL) was treated with Ph_2NK (1.55 g, 5.6 mmol). Upon allowing the homogeneous solution to stand undisturbed overnight at room temperature, a small amount of dark purple insoluble crystalline material separated (0.18 g). The solid was filtered off, and the mother liquor was evaporated to dryness. The residual solid was redissolved in toluene (250 mL), and the resulting solution was filtered to remove a small amount of insoluble material. The resulting solution was allowed to stand at −30 °C overnight, upon which dark orange crystals of **2** separated (0.36 g, 0.28 mmol, 33%). Anal. Calcd (Found) for $C_{79}H_{90}N_9Nb_2Li$: C, 69.85 (69.36); H, 6.68 (6.61); N, 9.28 (9.01). IR [Nujol, cm^{-1}] ν: 1589(s), 1307(m), 1287(m), 1261(s), 1172(m), 1079(m), 1027(s), 921(m), 887(m), 856(m), 798(s), 748(s), 727(s), 694(s). ^1H NMR [500 MHz, THF-d_8, 23 °C] δ: 8.38 (d, 1H, bridging amide terminal aromatic ring), 7.71 (d, 1H, bridging amide metalated ring), 7.30 (d, 2H, bridging amide terminal aromatic ring), 7.17 (m, 5H, toluene), 6.84 (m, 2H, bridging amide metalated ring), 6.80 (d, 1H, bridging amide metalated ring), 6.78 (pseudoquadruplet, 2H, bridging amide terminal aromatic ring), 6.64 and 6.43 (pseudotriplet, 40H, terminal amides rings), 2.31 (s, 8H, CH_2 TMEDA), 2.15 (s, 24H, CH_3 TMEDA), 2.08 (s, 3H, toluene), −8.24 (br s, 1H, hydride). ^{13}C NMR [127.7 MHz, THF-d_8, 23 °C] δ: 157.5, 154.6, 154.4, 152.5, 151.2 (quaternary carbon), 142.4, 121.7, 120.9 (bridging amide terminal aromatic ring), 122.7, 127.9, 126.0, 124.6 (bridging amide metalated ring), 123.9−119.7 (series of lines, aromatic ring terminal amides), 137.5, 128.8, 128.0, 125.2 (toluene), 58.93 (CH_2 TMEDA), 46.18 (CH_3 TMEDA), 20.04 (CH_3 toluene).

(15) Crystal Data. **1**: $C_{66}H_{71}ClLi_2N_{15}Nb_2O_4$, $M_w = 1373.53$, triclinic $P\bar{1}$ $a = 13.9589(7)$ Å, $b = 14.2761(7)$ Å, $c = 18.778(1)$ Å, $\alpha = 96.992(1)°$, $\beta = 93.911(1)°$, $\gamma = 118.336(1)°$, $V = 3235.1(3)$ Å^3, $Z = 2$, $D_{calc} = 1.410$ Mg/m^3, absorption coefficient 0.456 mm^{-1}, $F(000) = 1418$, number of reflections collected 20 099, number of independent reflections 14 086, GOF = 1.057, $R = 0.0512$, w$R^2 = 0.1316$. **2**: $C_{79}H_{90}N_9Nb_2Li$, $M_w = 1358.36$, monoclinic $P2_1/n$, $a = 13.4635(8)$ Å, $b = 39.259(2)$ Å, $c = 14.4266(9)$ Å, $\beta = 94.936(1)°$, $V = 7597.0(8)$ Å^3, $Z = 4$, $D_{calc} = 1.187$ Mg/m^3, absorption coefficient 0.348 mm^{-1}, $F(000) = 2844$, number of reflections collected 27 521, number of independent reflections 8705, GOF = 1.008, $R = 0.0913$, w$R^2 = 0.2505$.

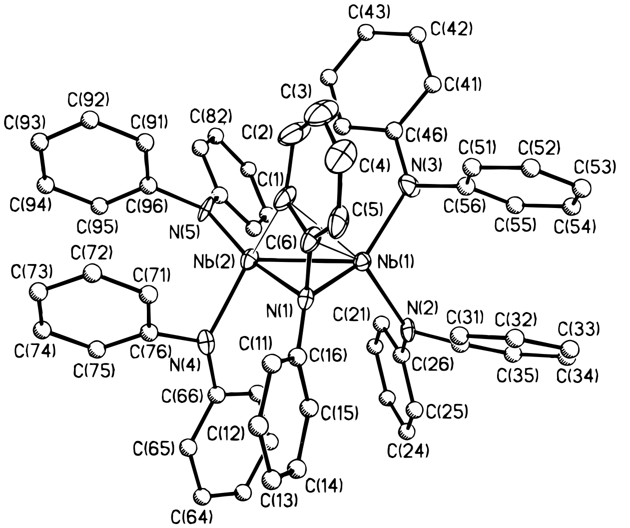

Figure 2. ORTEP drawing of **2**. Thermal ellipsoids are drawn at the 30% probability level. Bond distance are in angstroms (Å) and angles in degrees. Li(1)−N(101) = 2.12(3), Li(1)−N(102) = 2.16(4), N(101)−Li(1)−N(102) = 100(2), Nb(1)−N(1) = 2.177(9), Nb(2)−N(1) = 2.377(9), Nb(1)−N(2) = 2.107(9), Nb(1)−N(3) = 2.108(10), Nb(2)−N(4)−C(66) = 136.1(7), Nb(2)−N(4)−C(76) = 103.1(6), C(66)−N(4)−C(76) = 120.3(8), Nb(1)−N(1) = 2.177(9), Nb(2)−N(1) = 2.377(9) Nb(1)−N(1)−Nb(2) = 68.5(3), N(1)−C(16) = 1.448(11), Nb(1)−N(1)−C(16) = 138.2(7), Nb(2)−N(1)−C(16) = 128.2(6), Nb(1)−N(1)−C(6) = 87.1(7), Nb(2)−N(1)−C(6) = 93.3(6), Nb(2)−C(1) = 2.197(12), Nb(1)−C(1) = 2.573(12), Nb(1)−C(6) = 2.549(16), Nb(1)−C(1)−C(6) = 72.9(7), Nb(1)−C(6)−C(1) = 105.0(9).

in the absence of hydride the complex would be a *paramagnetic* mixed-valence Nb(II)/Nb(III) species. The resonance of the hydride was clearly visible in the ^{1}H NMR spectrum as a broad singlet [$\delta = -8.24$ ppm] which did not correlate with any carbon atom in HQMC. The assignment as a bridging hydride was further substantiated by the fact that the resonance decreased in intensity upon exposure of a THF solution to D_2 gas (99.8), while the resonance of the corresponding Nb−D appeared at −8.24 ppm in the ^{2}H NMR spectrum. The characteristic triplet of DH and the singlet of H_2 appeared at 4.55 and 4.70 ppm, respectively. Although the IR spectrum did not show any resonance in the 2500−1690 cm^{-1} region that could definitely be assigned to a terminal Nb−H, a broad and intense band was observed at 1070 cm^{-1} that we tentatively assign to the bridging hydride. Unfortunately, the tendency of **2** to thermally rearrange to the mixed-valence $(Ph_2N)_2Nb\{[\mu\text{-}NPh(\eta^6\text{-}C_6H_5)]_2Nb\}\cdot$ether[16] prevented carrying out isotopic labeling experiments to conclusively identify the position of the Nb−H resonance.

The formation of both **1** and **2** arises from the formal oxidative addition of the two metal centers into either the C−H or C−N bonds of the two different ligands. In both cases, the two electrons necessary for the oxidative additions are provided by the one-electron oxidation of the two Nb(II) centers. It is tempting to conjecture that in both cases, the reaction starts with the formation of similar $(amide)_5Nb_2Li$ intermediates. In the case of **1,** the reaction proceeds with the oxidative cleavage of the CN bond and formation of the resulting $[NC_5H_4]$ and $[N(NC_5H_5)]$ fragments. In the case of **2**, an analogous intermediate cleaves the C−H bond of one aromatic ring forming the corresponding aryl−hydride structure.

Both **1** and **2** are diamagnetic. This, in combination with the short intermetallic distances [Nb1−Nb2 = 2.7022(4), 2.569(12) Å for **1** and **2**, respectively] might be consistent with the presence of Nb−Nb double bonds. However, we observed that while in the case of **2** the intermetallic distance is perhaps in agreement with this proposal, in the case of **1** the distance is significantly longer and comparable to that of the face-sharing bioctahedral $Nb(II)Cl_3$ derivatives which are instead paramagnetic.[17] This suggests the presence of a significant participation of the bridging moieties in the Nb−Nb interactions. Theoretical work will be necessary to assess the nature of the Nb−Nb bond.

The different nature of the two transformations performed by Nb(II) with the two organic ligands (C−N versus C−H bond cleavage) emphasizes the high reactivity and also the selectivity provided by $(TMEDA)_2Nb_2Cl_5Li(TMEDA)$ during these reactions. At this early stage it is obviously premature to draw conclusions about which factors determine this noticeable difference of behavior and how to use this information for HDN catalysis purposes. So far we simply observe that, as a rather general trend,[12,13,16] this complex always performed two- or four-electron oxidative additions on the same substrate via cooperative interaction of the two metal centers.

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Supporting Information Available: Tables of atomic coordinates, thermal parameters, and bond distances and angles for **1** and **2**. Ordering information is given on any current masthead page.

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[16] More details about the thermolysis of **2** and denitrogenation reactions promoted by $(TMEDA)_2Nb_2Cl_5Li(TMEDA)$ will be reported elsewhere. (a) Tayebani, M.; Feghali, K.; Gambarotta, S.; Yap, G. *Organometallics*, in press. (b) Tayebani, M.; Gambarotta, S.; Yap, G. *Angew. Chem., Int. Ed.*, in press.

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