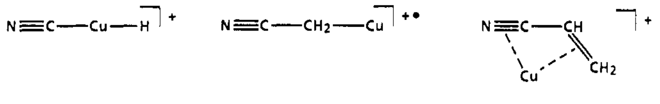
acted with the nitriles (ca. 10^{-3} Torr),[10] and the resulting complex ($^{65}Cu^+$/nitrile) accelerated to 8 keV kinetic energy, mass selected by using BE, and collided with He (ca. 10^{-5} Torr) to induce fragmentation.[11] Spectra were recorded by scanning the second magnet. Organometallic complexes formed via FAB have been shown[3,12] to produce results identical with those for complexes formed through ion/molecule reactions of neutral organic substrates with transition-metal ions; the latter were generated upon electron-impact ionization of suitable organometallic precursors.

The reaction of Cu^+ with the linear nitriles from propyl to hexyl cyanide (Table I; Figure 1) yield three major products (in addition to Cu^+ (m/z 65) which is a simple ligand detachment reaction), corresponding to m/z 92, 105, and 118. Labeling the cyanide group with ^{13}C in hexyl cyanide produces a quantitative mass shift of one unit for all three products. Thus, the cyanide group remains coordinated to the metal throughout the reactions. Proposed and by no means proven[13] structures A–C are shown below. These assignments (which do not rule out isomers, as for example a structure $HCN{-}Cu^+$ as an alternative to A, as suggested by a reviewer) are consistent with the data obtained from the study of deuterium-labeled isotopomers (Table II).



A (m/z 92) B (m/z 105) C (m/z 118)

Labeling the α-positions with deuterium produces a shift of two mass units for B and one for C. No shift in A is observed. Some 30% of C, however, have incorporated two deuterium atoms. Deuterium labeling of the β-positions produces a one deuterium shift for some of A (ca. 30%), although the rest remains unchanged. No mass shift is observed for B, and a shift of two mass units is observed for C. Labeling the γ-positions again produces a shift of one mass unit for ca. 30% of A, while the rest remains unchanged. No mass shifts are observed for either B or C.

The labeling results reveal complex mechanisms *totally distinct* from those earlier proposed for Fe^+, Co^+, and Ni^+.[1-3] For example, the H atom of ion A does not come from the α-position but originates at least from the β- and γ-positions (>60%) and perhaps from C–H bonds even further away from the functional group. Ion B is produced via insertion of the Cu^+ ion into the C(α)–C(β) bond. The α-methylene group is fully retained in the ionic fragment B. Ion C may be formed by allylic C–H activation of the α-positions, followed by β-alkyl transfer and reductive elimination. The alternative sequence is also reasonable with initial C–C cleavage of the C(β)–C(γ) bond followed by a hydrogen atom shift. However, the former pathway may be more favorable due to the initial activation of the allylic C–H position. Another mechanism is "dissociative attachment" whereby Cu^+ associates with a functional group (usually a heteroatom) and causes an organic molecule to rearrange and/or cleave. This mechanism has been proposed for the reactions of Cu^+ with ketones and esters,[14] alkyl chlorides,[15] and alcohols.[16,17] However, as stated by Gross and Peake[5] "dissociative attachment is thought to be a trait of alkali-metal ions".[18,19] We thus reacted Na^+ and Li^+, which bonds more strongly to heteroatoms, with pentyl cyanide and observed essentially no reactions. The only product was due to the loss of the ligand to regenerate Na^+ and Li^+, respectively.

Finally, it should be mentioned that *end-on* coordination of Cu^+ with RCN should also occur from simple electrostatic considerations. This mode is, for example, operative in the protonation and alkylation of nitriles that take place exclusively at the lone pair of the nitrogen.[20] However, this thermochemically favorable coordination most probably generates *unreactive* RCN/Cu^+ complexes. The species, which upon collisional activation[21] give rise to the products A, B, and C, are more likely to have a *side-on* coordination. If the reactive species would possess an *end-on* structure, we should observe products identical with those generated from RCN/M^+ (M = Fe, Co, Ni).

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[10] It should be mentioned that the detailed mechanism by which the RCN/Cu^+ complexes are formed from sputtered Cu^+ (or species of complex compositions) is unknown. The pressure in the ion source, however, is high enough to probably "cool" the complex by multiple collisions. Furthermore, the RCN/Cu^+ complex is big enough so that its lifetime is lengthened by dispersing the internal energy, gained in the complex formation, to the many vibrational modes.

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[13] Proposed structures are fictitious in nearly all cases studied in mass spectrometry; they are based on plausible considerations using as much direct and indirect "evidence" as available from experiments and model considerations. Strictly speaking, without detailed spectroscopic studies and state of the art high level ab initio MO calculations, both of which cannot be applied in the present case due to size of the molecules in question, none of the structures are proven. Nevertheless, the heuristic merits of such structural representations are indeniable in chemistry, as long as speculations remain within acceptable limits.

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[21] In order to study the effects of internal energy, deposited in the collision event in the RCN/Cu^+ complexes, we have performed some initial experiments at lower kinetic energies. It turned out that the production of ions A, B, and C from RCN/Cu^+ is not any longer observed when the kinetic energy of RCN/Cu^+ is less than 4 keV. Similarly, no fragment ions are observed under collision-free conditions.

Regioselective Insertion of Acrylonitrile into the Pt–N Bond of Hydrido(phenylamido)bis(triethylphosphine)platinum(II). A Model Step for Olefin Amination

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Summary: The new hydridoamido complex *trans*-PtH(NHPh)$(PEt_3)_2$ (I) inserts acrylonitrile to form *trans*-PtH[CH(CN)(CH_2NHPh)]$(PEt_3)_2$, which reductively eliminates 3-anilinopropionitrile.

Catalytic amination of olefins to amines under mild conditions has not yet been achieved, although the hydration of olefins to alcohols occurs with as simple a catalyst as H_2SO_4.[1] Transition-metal hydride complexes of Ni, Pd, Pt, Rh, and other platinum group metals have been recognized as intermediates in catalytic reactions such as hydrogenation,[2] hydration,[3] hydrocyanation,[4] olefin isomerization,[5] hydroformylation,[6] and hydrosilation.[7] One model catalytic cycle for olefin amination would proceed by olefin insertion into a hydrido amido complex, followed by reductive elimination of amine product. If one considers that tetrafluoroethylene will insert into a Pt–O bond[8] and that a recent study[9] demonstrates Pt–N bonds should be thermodynamically less stable than Pt–C bonds, then the insertion of olefins into Pt–N bonds may be thermodynamically feasible.

Oxidative addition of water[3] and alcohols[10,11] to bis(phosphine)platinum(0) complexes yields metal hydrides; however, analogous reactions with ammonia are unknown. Oxidative addition of an N–H bond to a platinum(0) species has been observed only for activated amines and amides (–C(O)NHR), where the pK_a's approach those of alcohols.[12] Current research on late transition-metal amide chemistry includes N–H intramolecular oxidative addition to Rh(I) and Ir(I),[12] synthesis of the first alkyl amido complexes of Pt,[13] and preparation of $Cl(PEt_3)_2HIr(\mu\text{-}NH_2)HIrH(PEt_3)_2Cl$ by addition of NH_3 to $Ir(PEt_3)_2(CH_2CH_2)Cl$ in liquid NH_3.[14] A question remains whether the weakness of late transition metal–nitrogen bonds may preclude oxidative addition on thermodynamic grounds.[9] In this report we show the feasibility of an insertion–reductive elimination sequence for the reaction of a hydrido amido complex with an olefin and some qualitative information about the kinetic and thermodynamic stability of a bis(phosphine)platinum(II) hydrido amido complex.

Addition of 1.5 equiv of Na(NHPh) (a solution in THF) to *trans*-$PtH(NO_3)(PEt_3)_2$[15] in benzene followed by evaporation of solvent, extraction with pentanes, concentration, and cooling to –30 °C yields (80%) extremely air-sensitive yellow crystals of $PtH(NHPh)(PEt_3)_2$ (I). These crystals darken within hours at room temperature but may be stored under a N_2 atmosphere at –30 °C for several months. In contrast, toluene solutions of I, while extremely air-sensitive, do not decompose on heating for 0.5 h at 90 °C. Complex I exhibits a $^{31}P\{^1H\}$ NMR spectrum (δ 21.8 (s, $^1J_{\text{Pt-Pt}}$ = 2778 Hz)) consistent with a trans Pt(II) structure. Selective decoupling experiments show the presence of a single hydride ligand cis to the phosphines ($^2J_{\text{P-H}}$ = 14 Hz). The ^1H NMR spectrum (Figure 1) shows, in addition to the hydride resonance, C (δ –14.7 (t, $^2J_{\text{P-H}}$ = 16 Hz, $^1J_{\text{Pt-H}}$ = 885 Hz)), and PEt_3 protons (δ 1.5–0.9), a singlet, B, for the N–H proton at δ 2.2 flanked by platinum satellites ($^2J_{\text{Pt-H}}$ = 22 Hz, ^{195}Pt is 33.7% abundant with $I = {}^1/_2$). The aromatic region of the spectrum, A, exhibits a triplet–doublet–triplet pattern for the anilide ring where the meta and ortho protons, respectively, lie downfield of free aniline while the para triplet moves upfield.

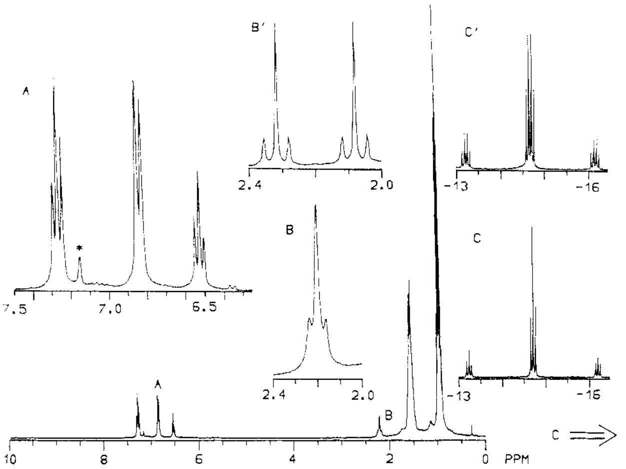

Figure 1. ^1H NMR spectra of *trans*-$PtH(NHPh)(PEt_3)_2$ showing aromatic protons, A, the N–H proton, B, and the hydride resonance, C. Spectra in B′ and C′ show corresponding resonances for *trans*-$PtH(^{15}NHPh)(PEt_3)_2$.

Definitive characterization of the Pt(NHR) moiety is obtained from an analysis of the ^{15}N NMR spectrum of I prepared from 99% enriched ^{15}N aniline. The proton-decoupled spectrum contains a singlet 2.25 ppm upfield of free aniline flanked by ^{195}Pt satellites ($^1J_{\text{N-Pt}}$ = 216 Hz). Coupling to cis ^{31}P was not resolved. As the proton-decoupler power was reduced, additional splittings appear from a single directly bound hydrogen ($^1J_{^{15}\text{N-H}}$ = 71 Hz) and the metal hydride ($^2J_{^{15}\text{N-H}}$ = 20 Hz). This assignment is confirmed from the additional ^{15}N splittings that develop in the ^1H NMR spectrum of the ^{15}N-enriched complex (Figure 1, parts B′ and C′). The low value of $^1J_{^{15}\text{N-H}}$ requires sp^3 hybridization and a pyramidal structure about nitrogen.[16] Therefore, little π donation from N to Pt occurs, as expected for a d^8 metal complex with filled dπ orbitals.

Although benzene solutions of I are stable for hours in vacuo, the addition of CO or PEt_3 results in immediate reductive elimination of aniline and the formation of $Pt(CO)_2(PEt_3)_2$ or $Pt(PEt_3)_4$, as identified by comparison of their ^1H and ^{31}P NMR spectra with those of authentic samples.[17] Addition of C_2H_4, PhC≡CPh, or *cis*-stilbene to a benzene solution of I yields free aniline and the corresponding bis(phosphine)platinum(0) complex[18] on

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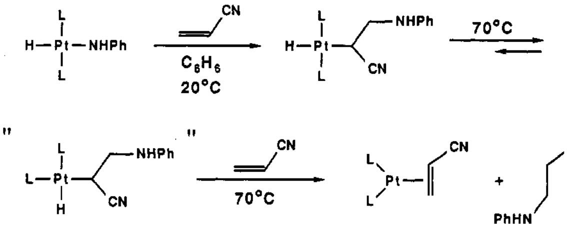
standing for several days or on heating to 150 °C. Insertion products with these unactivated olefins were not detected. Insertion of CO_2, however, proceeds rapidly at room temperature to yield *trans*-$PtH(OCONHPh)(PEt_3)_2$.[19]

Acrylonitrile undergoes a regioselective 1,2-insertion into the Pt–N bond to generate a new hydrido alkyl species, as identified by ^{31}P and ^{1}H NMR spectroscopies (eq 1).[20]



(1)

The ^{31}P NMR spectrum shows complete conversion of the starting material to the insertion product after 48 h of reaction at room temperature. When the sample is heated to 70 °C for another 48 h, reductive elimination occurs (eq 1) to yield $Pt(PEt_3)_2(\eta^2\text{-}CH_2{=}CHCN)$, as identified by ^{31}P NMR spectroscopy.[21] The organic product 3-anilinopropionitrile was identified by ^{1}H NMR spectroscopy and by comparison with an authentic sample purchased from Pfaltz and Bauer. The other isomer for the 1,2-addition process could not be detected, so the regioselectivity of addition must be high (>95%). If the reaction between I and acrylonitrile is performed at 70 °C, insertion–reductive elimination is observed with a total reaction time less than 48 h.

Hydrogenolysis of the Pt–N bond of I, eq 2b, also occurs and may proceed by oxidative addition of H_2 to I via an intermediate six-coordinate Pt(IV) species. Consistent

$$PtH_2(PEt_3)_2 \overset{K \approx 1}{\rightleftharpoons} \text{“}Pt(PEt_3)_2\text{”} + H_2 \qquad (2a)$$

$$PtH(NHPh)(PEt_3)_2 + H_2 \overset{K' \gg 1}{\rightleftharpoons} PtH_2(PEt_3)_2 + H_2NPh \qquad (2b)$$

$$PtH(NHPh)(PEt_3)_2 \overset{KK' \gg 1}{\rightleftharpoons} \text{“}Pt(PEt_3)_2\text{”} + H_2NPh \qquad (2c)$$

with this mechanism D_2 adds to I to produce NDHPh, in addition to $PtD_2(PEt_3)_2$ (from ^{2}H NMR spectral analysis). The equilibrium constant for eq 2b must lie far to the right because addition of a 50-fold excess of aniline to a benzene solution of $PtH_2(PEt_3)_2$[17] led to no detectable reaction. Since reductive elimination of H_2 from $PtH_2(PEt_3)_2$ is favored in the absence of a hydrogen atmosphere (2a),[22] these results require that *trans*-$PtH(NHPh)(PEt_3)_2$ is less thermodynamically stable toward reductive elimination than the dihydride species (eq 2c). Since Pt–N bonds are predicted to be weaker than Pt–H bonds,[9] the kinetic stability of I in solution is unexpected.

Our results show that a hydrido amido complex can be used to model an insertion–reductive elimination sequence, which might be involved in the addition of an N–H bond to an olefin. Unfortunately, as for C–H activation with platinum phosphine complexes[23] oxidative addition of unactivated NH bonds to the intermediate PtL_2 is thermodynamically unfavorable so a catalytic cycle is not completed. The kinetic inertness of the hydrido amido complexes prepared by metathesis may arise from a high barrier to N–H reductive elimination caused by the instability of the cis configuration necessary for reductive elimination. In platinum chemistry, strong trans directors (e.g. H, PR_3, and CH_3) prefer weak trans influence ligands opposite them (e.g. O and N donors).[24] Thus there may be a strong electronic preference for the NHR ligand to be trans to hydride, which shuts down the rate of isomerization, and ultimately leads to a greatly decreased rate of elimination in I. Accelerated reductive elimination on addition of CO or PEt_3 probably occurs by formation of a d^8 trigonal-bipyramidal complex. Molecular orbital theory predicts[25] the strong σ-donor H and PEt_3 ligands should occupy axial sites forcing the axial H and equatorial NHPh ligands into in the required cis geometry for reductive elimination to occur. Alternatively, reductive elimination may proceed by dissociation to free amide and proton transfer.

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(18) These products all exhibit ^{31}P NMR (C_6D_6) spectra and Pt–P coupling constants expected[17] for unsaturated molecules symmetrically bound to a zerovalent PtL_2 species: δ 21.0 (s, $^{1}J_{Pt-P}$ = 3517 Hz, C_2H_4), 12.0 (s, $^{1}J_{Pt-P}$ = 3301 Hz, C_2Ph_2), 16.5 (s, $^{1}J_{Pt-P}$ = 3387 Hz, *cis*-HPhC=CPhH). Additional evidence arises from the coupling of ^{195}Pt to the olefinic protons of the ethylene and *cis*-stilbene reaction products. ^{1}H NMR (C_6D_6): δ 2.1 (s, $^{2}J_{Pt-H}$ = 57 Hz, C_2H_4), 4.1 (s, $^{2}J_{Pt-H}$ = 60 Hz, *cis*-HPhC=CPhH).

(19) $^{31}P\{^{1}H\}$ NMR (C_6D_6): δ 25.3 (s, $^{1}J_{Pt-P}$ = 2854 Hz). ^{1}H NMR (C_6D_6): δ 9.1 (s, NH), 8.2 (d, ortho), 7.3 (t, meta), 6.9 (t, para), –22.0 (t, Pt–H, $^{1}J_{Pt-H}$ = 1200 Hz, $^{2}J_{P-H}$ = 16 Hz). $^{13}C\{^{1}H\}$ NMR (C_6D_6): δ 160.1 (s, OCONHR). IR (C_6D_6): 1625 cm^{-1} (s, C=O), 2210 cm^{-1} (m, Pt—H). Solutions of this complex are stable only under an atmosphere of CO_2.

(20) $^{31}P\{^{1}H\}$ NMR (C_6D_6): δ 11.7 (s, $^{1}J_{Pt-P}$ = 2740 Hz). ^{1}H NMR (C_6D_6): δ 3.97 (m) and 3.46 (m) (CH_2CN diasteriotopic protons), 4.27 (s, NH), 2.46 (m, $^{2}J_{Pt-H}$ = 75 Hz, PtCH), –9.54 (t, $^{1}J_{Pt-H}$ = 785 Hz, $^{2}J_{P-H}$ = 18 Hz, Pt–H). Anal. Calcd for $C_{21}H_{40}N_2P_2Pt$: C, 43.66; H, 6.99; N, 4.85. Found: C, 44.04; H, 7.30; N, 4.77.

(21) $^{31}P\{^{1}H\}$ NMR (C_6D_6): δ 16.8 ($^{1}J_{Pt-P}$ = 3299 Hz, $^{2}J_{P-P}$ = 36 Hz), 18.3 ($^{1}J_{Pt-P}$ = 3723 Hz, $^{2}J_{P-P}$ = 36 Hz). For related olefin complexes see ref 17.

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Oxidative Addition/Reductive Elimination of Aldehydes and Ketones at Rhodium. Synthesis and Characterization of Cis Hydrido Acyl Complexes of Rhodium(III)

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Summary: The Rh(I) complex $(np_3)RhH$ [np_3 = $N(CH_2CH_2PPh_2)_3$] reacts with Me^+ from $MeOSO_2CF_3$ to give, following the elimination of CH_4, the 16-electron $(np_3)Rh^+$ moiety. The latter reacts with RCHO molecules (R = H, Me, Ph) yielding stable cis hydrido acyl complexes. By contrast, the isoelectronic PP_3Rh^+ system [PP_3 = $P(CH_2CH_2PPh_2)_3$] is unable either to directly activate aldehydes or to keep hydride (or alkyl) and acyl groups in mutually cis positions of the coordination sphere.

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