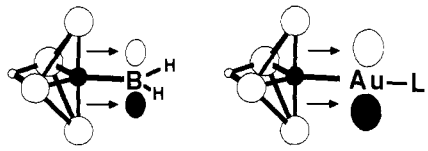
Chart I



(a) (b)

interaction is maximized for the observed cluster structure. The strength of this interaction must be sufficient to prevent the adoption of the presumably preferred 60-electron $[Fe_4(CO)_{12}CC(O)Me]^-$ structure. Clearly the B2p orbital is substantially occupied as even though I was prepared in the presence of THF, no THF is found in the solid state. This, plus the very slow reaction with Lewis bases, shows that the formal acid site on I is sterically and/or electronically inaccessible.

The occupation of the B2p orbital can be estimated from the ^{11}B NMR chemical shift.[16] Considering the boron in I as three coordinate, one expects the ^{11}B shift to lie in the range δ 87 to 7. The low field limit corresponds to compounds with little back donation into the B 2p orbital, e.g., BMe_3, and the high field limit to compounds with large back donation, e.g., BI_3. Hence, the shift of δ 9 observed for I is consistent with strong back donation from the carbido cluster to the boron atom. In addition, the ^{11}B-H coupling constant is typical of that observed for related boranes, e.g., BH_3·THF.

One might also view I as being derived from a hypothetical square-pyramidal carbido cluster cation $[HFe_4(CO)_{12}CBH]^+$ which has opened up into the observed structure on the addition of the base H^- to boron. This reaction has been observed for the all-transition-metal system, $Os_5(CO)_{15}C$, which opens into structure I on the addition of another ligand, e.g., CO.[17] If the osmium cluster is viewed as a $[Os_4(CO)_{12}C]^-$ carbido fragment bound to a $[Os(CO)_4]^+$ fragment, then the latter plays the role of the BH_2 fragment in I, i.e., I is a borane mimic of the "bridged-butterfly" pentaosmium carbido cluster. Note, however, if the boron atom actually occupied a cluster vertex, one would expect a tetrahedral disposition of nearest neighbors rather than the trigonal one observed.

Further, I is structurally very similar to $HFe_4(CO)_{12}CAu(PPh_3)$ with the $[Au(PPh_3)]^+$ fragment replacing the $[BH_2]^+$ fragment.[18] The carbido framework of the gold derivative is that of the 62-electron butterfly despite the fact that the $AuPPh_3$ fragment is formally an exo-ligand to the carbido carbon. As noted by the authors, the structural parameters suggest that a p orbital of the gold atom is interacting with the wing-tip iron atoms, and the bonding situation is analogous to that of I (Chart Ib). If the $[AuPPh_3]^+$ had behaved like $[H]^+$,[19] one would have expected it to bridge a C-Fe edge to yield a structure analogous to that of $HFe_4(CO)_{12}CH$.[8] Hence, one might well view the $[Au(PPh_3)]^+$ as isolobal to the $[BH_2]^+$ fragment. Considering, in addition, the tendency of gold fragments to form Au-Au bonds, this may be a better representation of the bonding capabilities of this fragment.

A more detailed analysis of the properties of the electronic structure of I is required, but it is clear from the information presented here that the use of metal clusters as ligands has the potential for the stabilization of unusual bonding modes particularly where p backbonding as well as steric bulk is advantageous.

Acknowledgment. The support of the National Science Foundation is gratefully acknowledged.

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Registry No. $Fe_2(CO)_9$, 15321-51-4; BH_3·THF, 14044-65-6; $HFe_4(CO)_{12}CBH_2$, 119850-57-6.

Supplementary Material Available: Tables of atom coordinates, bond distances and angles, and anisotropic temperature factors (4 pages); tables of observed and calculated structure factors (9 pages). Ordering information is given on any current masthead page.

The First High Resolution Direct NMR Observation of an f-Block Element

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The last decade has seen a dramatic increase in the importance of the organometallic chemistry of the lanthanide elements.[1-4] In particular, iodides, cyclopentadienyls, and bis(trimethylsilyl)amides of Sm(II), Eu(II), or Yb(II), due to their unique reactivity (e.g., as selective reducing agents[4-7]), have attracted much attention. Yet high resolution, solution-state NMR, one of the organometallic chemists' most informative tools, has not previously been applied to the direct observation of an f-block element, excluding complexes of the f^0 La(III). The reason for this neglect is probably because the majority of complexes [except those of La(III), Yb(II), and Lu(III)] are paramagnetic, and many of the NMR-active f-block nuclei have large quadrupole moments. Yet for certain of these elements spin-1/2 isotopes exist, and the range of diamagnetic compounds is rapidly increasing. As a case in point, ^{171}Yb is a spin-1/2 nucleus, with a natural abundance of 14.27% and a moderately sized, positive gyromagnetic ratio (4.712×10^7 rad T^{-1} s^{-1}); these features combine to give a receptivity four times greater than that of ^{13}C. Three solid-state, wide-line studies of this nucleus have appeared.[8-10] We now report ^{171}Yb chemical shift solution NMR data for the series of Yb(II) complexes **1-7**, Table I.

NMR experiments were performed on a Bruker WM360 spectrometer. We originally chose $[Yb(\eta\text{-}C_5Me_5)_2(OEt_2)]$ (**1**)[11] as the ^{171}Yb chemical shift standard, due to its good solubility, thermal stability, and low-frequency resonance. {Subsequently a referee has suggested a lower frequency standard having similar characteristics, $[Yb(\eta\text{-}C_5Me_5)_2(THF)_2]$ (**7**), see Table I.} The

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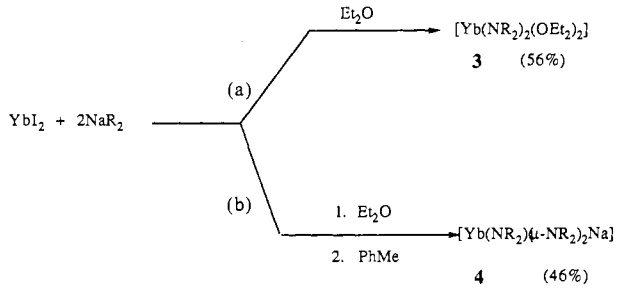
Table I. Some ^{171}Yb NMR Data for the Yb(II) Complexes **1**–**7**

compound	solvent	temp (K)	$\delta(^{171}Yb)$ (ppm)	$w_{1/2}$[a] (Hz)	$\Delta\delta/\Delta T$, ppm/K
$[Yb(\eta\text{-}C_5Me_5)_2(OEt_2)]$, **1**[11]	Et_2O	308	36[b]	90	+0.25
$[Yb(\eta\text{-}C_5Me_5)_2(NC_5H_5)_2]$, **2**[13]	C_5H_5N	338	949	70	+1.46
$[Yb(NR_2)_2(OEt_2)_2]$, **3**[14]	Et_2O	193	614[c]	70	−0.18
$[Yb(NR_2)(\mu\text{-}NR_2)_2Na]$, **4**[14]	PhMe	193	947	190	+0.5
$[\{Yb(NR_2)(\mu\text{-}NR_2)\}_2]$, **5**[17]	PhMe	263	796	90	+0.35
$[Yb(NR_2)_2(dmpe)]$, **6**[18]	PhMe	193	1228	200	+0.51
$[Yb(\eta\text{-}C_5Me_5)_2(THF)_2]$, **7**[22]	THF-d_8	173	87[d]	24	+0.69

[a] The large $w_{1/2}$ values and their inverse dependence on temperature, suggest exchange broadening contributes to the line widths. [b] In a typical experiment, a 0.171 molar solution of **7** in THF, at 173 K, gave a signal:noise ratio of 82:1 after 16 scans. [c] $^1J(^{14}N\text{–}^{171}Yb)$ = 117.6 Hz. [d] Corresponding to 0 ppm at 296 K.

Scheme I

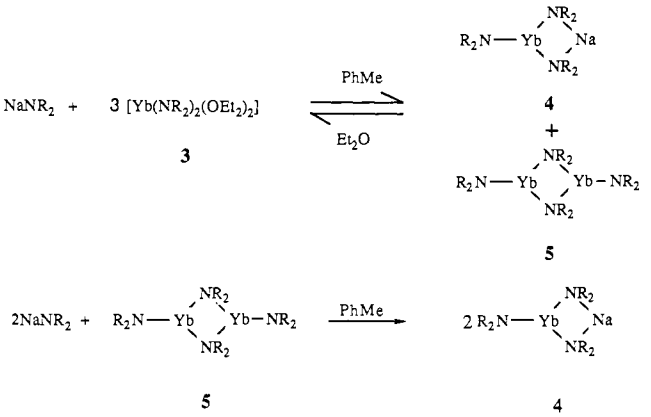


^{171}Yb signal in **7** had an absolute frequency of 63.021 652 3 MHz at 296 K; the protons of $Si(CH_3)_4$ under the same conditions resonated at 360.138 004 MHz. The spin–lattice relaxation time of 1.312 s at 263 K for **7** was measured by the inversion recovery method.[12] Irradiation at the frequency of the protons produced no measurable nuclear Overhauser effect. The temperature dependence of the chemical shift ($\Delta\delta/\Delta$T) for **7** over the range 193–353 K was 0.65 ppm K^{-1}. The bis-pyridine analogue $[Yb(\eta\text{-}C_5Me_5)_2(py)_2]$ (**2**)[13] displayed the largest $\Delta\delta/\Delta$T of the compounds studied.

A series of Yb(II) bis(trimethylsilyl)amides was next examined. The ^{171}Yb NMR signal for $[Yb(NR_2)_2(OEt_2)_2]$ (R = $SiMe_3$) (**3**)[14] comprised a broad quintet, due to coupling to two equivalent ^{14}N nuclei, ^1J = 117.6 Hz. (This was verified by repeating the observation at a lower frequency using a JEOL GSX-270.) The multiplet collapsed with decreasing temperature as relaxation of the ^{14}N became more efficient, resulting in a line width at half height ($w_{1/2}$) of 70 Hz at 193 K. Observation of $^1J(^{14}N\text{–}^xM)$, although unexpected in **3** (^xM = ^{171}Yb) because of the unsymmetrical ^{14}N environment, has been noted for other M(II) amides $M(NR_2)_2$ (^xM = ^{119}Sn or ^{207}Pb).[16] The value for $^1J(^{14}N\text{–}^{171}Yb)$ of 117.6 Hz represents a reduced coupling constant K which is very similar to that in the tin analogue but substantially smaller than in the lead compound: $K(^{14}N\text{–}M)/NA^{-2}\ m^{-3}$: M = ^{171}Yb, 7.71 × 10^{21}; M = ^{119}Sn, 7.58 × 10^{21}; M = ^{207}Pb, 16.5 × 10^{21}.

The ^{171}Yb NMR spectrum of $[Yb(NR_2)(\mu\text{-}NR_2)_2Na]$ (**4**)[14] consisted of a singlet at 947 ppm. Interestingly, the ^{29}Si spectrum of **4** showed two peaks, at −11.5 and −15.0 ppm, of relative intensity 2:1; upon warming to 293 K they collapsed to a singlet (−12.6 ppm), indicating rapid site-switching of the sodium cation among the three amido ligands.

The reaction between YbI_2 and $2NaNR_2$ was said to be solvent dependent, as shown in Scheme I (the yields referred to crystalline products).[14] Using ^{171}Yb NMR, we found that the situation is more complicated (eq 1 and 2). Thus, the PhMe extract, prepared according to (b) in Scheme I, showed the presence of **3**, **4**, and $[\{Yb(NR_2)(\mu\text{-}NR_2)\}_2]$ (**5**), in relative ratios 5:3:2. Removal of PhMe and addition of Et_2O to the solid residue yielded **3** as the exclusive ytterbium(II)-containing product. Evaporation of Et_2O and addition of PhMe restored the above equilibrium mixture of **3**, **4**, and **5**. Finally, heating the PhMe solution to ca. 353 K and evaporation of the solvent, followed by further cycles of PhMe addition/heating/and evaporation, gave solely **4** and **5** (eq 1). Pure **5** was isolated from this PhMe solution by crystallization at −13 °C.[17] A more convenient synthesis of **4**, in quantitative yield, was by addition of $NaNR_2$ to a demimolar portion of **5** (eq 2). [Compound **5** has previously been reported[18] but not as a product of eq 1 or 2.] Further reactions of **5**, related to that of eq 2, are under investigation (see also ref 19).



The substitution of the two Et_2O ligands of **3** by bis(dimethylphosphino)ethane (dmpe) has been demonstrated to yield $[Yb(NR_2)_2(dmpe)]$ (**6**).[20] We found that there was no reaction upon addition of 1 equiv of dmpe to the red, dilute Et_2O solution of **3** at ambient temperature; only upon concentration of the solution in vacuo was the purple **6** obtained.[21] The ^{171}Yb NMR spectrum of **6** showed no ^{31}P–^{171}Yb coupling, implying either an upper limit of ca. 200 Hz for $^1J(^{31}P\text{–}^{171}Yb)$ or, more probably, ^{31}P decoupling arising from rapid neutral ligand exchange. Addition of excess dmpe to an Et_2O solution of **3** and examination of the ^{171}Yb NMR spectrum at 193 K showed three signals: at 1136 (**6**), 862 (the major peak), and 606 (**3**) ppm, which on warming coalesced to a singlet at 852 ppm. The peak at 862 ppm is assigned to $[Yb(NR_2)_2(dmpe)(OEt_2)]$, which is in equilibrium with **3** and **6**.

(12) T_1 becomes longer with (i) increasing temperature (0.132 s at 173 K and 1.312 s at 263 K) and (ii) decreasing field strength (0.146 s at 8.45 T and 0.351 s at 6.34 T at 183 K).

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(17) The ^{29}Si NMR spectrum of **5** in PhMe showed two singlets (−7.8 and −13.8 ppm) at 193 K of relative intensity 1:1, assigned to the ^{29}Si atoms of terminal and bridging NR_2 ligands, respectively; $NaNR_2$ has $\delta(^{29}Si)$ = −21.9 ppm.

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(21) An ether solution of equimolar dmpe and **3** showed $\delta(^{171}Yb)$ = 614 ppm and $\delta(^{31}P)$ = −186.6 ppm at 302 K due to **3** and uncomplexed dmpe, respectively. Evaporation of the solution yielded the purple **6**, identified in part by its ^{31}P NMR spectra, $\delta(^{31}P)$ = −180.2 ppm.[20]

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The use of ^{171}Yb NMR spectroscopy as a valuable structural and mechanistic probe is expected to catalyze a rapid expansion in Yb(II) chemistry.

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Nucleotide Base Recognition: A Macrocyclic Receptor for Adenine Employing Hydrogen Bonding and Aromatic Stacking Interactions

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Effective molecular recognition requires a precise complementarity between several binding regions on the receptor and the different chemical features of the substrate. We have recently introduced[1] such a *multi-site* approach for the recognition of nucleotide bases in which hydrogen bonding and aromatic stacking groups within a macrocyclic receptor bind simultaneously to the substrate. Varying the hydrogen bonding region has led to selective receptors for thymine[2a] and guanine,[2b] while changing the electronic characteristics of the stacking group results[2c] in different geometries for the aromatic–aromatic interaction.[3] In this paper we report the further development of this approach with the synthesis, structure, and binding properties of a family of receptors for adenine derivatives.[4]

The periphery of adenine offers four readily accessible hydrogen bonding sites, the pyrimidine-N,NH of Watson–Crick and the imidazole-N,NH of Hoogsteen base-pairing.[5] Molecular modelling studies suggested that all four of these could be complexed by a 1,2-bis(2-amino-6-pyridyl)ethane derivative in an anti conformation and with inwardly pointing pyridine and amide groups (Chart I).[5] This particular orientation of hydrogen bonding groups should be favored by incorporating the dipyridylethane into a macrocycle which also contains a suitable π-stacking component.

The synthesis of the adenine receptors is shown in Chart II. Protection of 2-amino-6-picoline as its phthalimide derivative **1** followed by NBS bromination gave bromomethylpyridine **2** in 60% yield. Reductive dimerization[6] of **2** using chlorotris(triphenylphosphine)cobalt(I)[7] afforded a 50% yield of **3**[8] which was then

Chart I

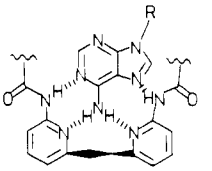
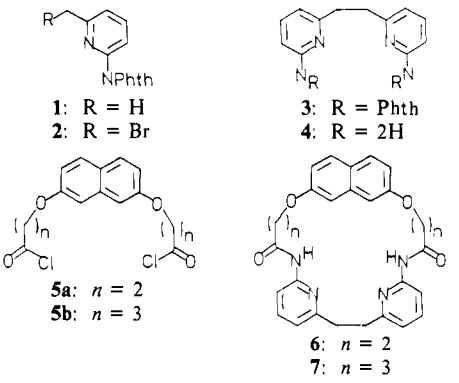


Chart II



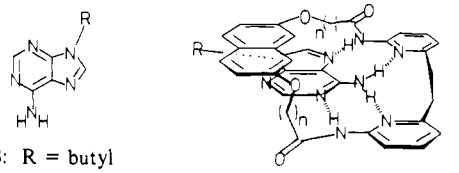
1: R = H
2: R = Br

3: R = Phth
4: R = 2H

5a: n = 2
5b: n = 3

6: n = 2
7: n = 3

Chart III



8: R = butyl

deprotected via hydrazinolysis (70% yield) to diamine **4**.[8] High dilution coupling of **4** (CH_2Cl_2, Et_3N) with the appropriate naphthalene diacid chloride **5a**,**b**[2a,9] gave macrocyclic receptors **6** and **7**[8,10] in 17 and 19% yield, respectively.

The proposed binding orientation of the receptors (Chart I) is supported by the X-ray crystal structure of macrocycle **6** (Figure 1a). Two intramolecular hydrogen bonds between the amide-NHs and ether-Os (H···O, 1.95 Å, 2.00 Å) stabilize a conformation for the macrocycle in which the two amidopyridines are anti to each other and approximately in the same plane. This places the pyr-Ns at 5.04 Å and the amide-NHs at 7.33 Å apart from each other with good binding complementarity to the amino group and purine-Ns of adenine. In contrast, the crystal structure of receptor **7** (Figure 1b) shows a more open conformation with the dipyridylethane unit in a gauche arrangement. A 60° rotation around the central C–C bond is required to form the binding orientation.

The adenine-binding properties of the receptors were followed by ^{1}H NMR and showed a strong dependence on ring size. Titration of **7** in $CDCl_3$ with 9-butyladenine **8**[11] caused large downfield shifts in the receptor-NH (2.0 ppm) and adenine-NH_2 (2.4 ppm) resonances consistent with the formation of a tetrahydrogen bonded complex, as in Chart I. In addition, upfield shifts in the naphthalene-1,8- (0.4 ppm), 4,5- (0.16 ppm), and 3,6- (0.17

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(8) All new compounds gave satisfactory spectroscopic and analytical and/or high resolution mass spectral data.

(9) Prepared by alkylating 2,7-dihydroxynaphthalene with the appropriate bromoester (acetone, K_2CO_3) followed by hydrolysis and acid chloride formation (oxalyl chloride).

(10) **7**: ^{1}H NMR ($CDCl_3$) 8.30 (2 H, br s, NH), 7.95 (2 H, d, J = 7.6 Hz, pyr-2H), 7.59 (2 H, d, J = 8.8 Hz, naphth-4,5H), 7.54 (2 H, t, J = 7.6 Hz, pyr-4H), 7.28 (2 H, d, J = 2.0 Hz, naphth-1,8H), 6.95 (2 H, dd, J = 2.0, 8.8 Hz), 6.82 (2 H, d, J = 7.6 Hz, pyr-5H), 4.21 (4 H, t, J = 5.8 Hz, CH_2O), 2.91 (4 H, s, pyrCH_2), 2.61 (4 H, t, J = 5.9 Hz, CH_2CO), 2.27 (4 H, m, $CH_2CH_2CH_2$).

(11) Synthesized by a modification of Carraway et al. (Carraway, K. L.; Huang, P. C.; Scott, T. G. In *Synthetic Procedures in Nucleic Acid Chemistry*; Zorbach, W. W., Tipson, R. S., Eds.; Interscience: New York, 1986; p 3).

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