Inorg. Chem. **1984**, *23*, 3263–3265

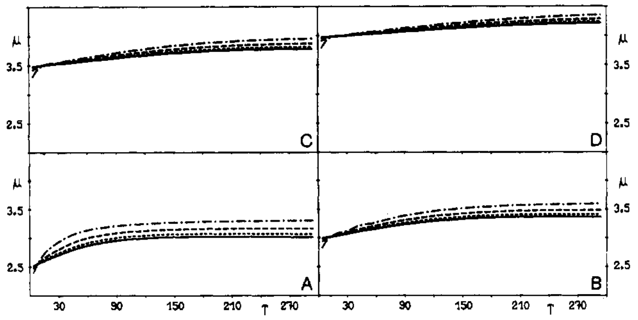

Figure 2. Calculated values of magnetic moments for [NiBr(Me$_6$tren)]Br for different values of $k_\parallel$ and $k_\perp$. The curves refer to $k_\perp$ = 0.25 (—), 0.50 (···), 0.75 (---), and 1.0 (-·-) with $k_\parallel$ = 0.25 (A), 0.50 (B), 0.75 (C), and 1.0 (D). The AO and Racah parameters are those reported in Tables I and II.

Table II. Best-Fit Parameters[a] for Magnetic Moments and Susceptibilities for [NiBr(Me$_6$tren)]Br and [NiNCS(Me$_6$tren)]SCN·H$_2$O

	B^b	C^b	ζ^c	k_x	k_y	k_z
Br	850	3250	649	0.93	0.93	0.34
SCN	850	3250	649	0.90	0.94	0.50

[a] The AO parameters are those reported in Table I. [b] Racah parameters in cm^{-1}. [c] Spin–orbit coupling constant in cm^{-1}.

e_π parameters for the nitrogen donor atoms of the tripod ligand were assumed to be zero. The bromide derivative has crystal C_3 symmetry, while the thiocyanate has only C_1 symmetry. For the calculation the geometrical parameters seen in the crystal structure were used, but no attempt was made to use different e_σ parameters for the three equatorial donors of the thiocyanate derivatives. The best-fit values are shown in Table I. In order to reproduce the experimental magnetic susceptibility data, we first attempted to use isotropic ζ, the spin–orbit coupling constant, and k, the Stevens orbital reduction factor.[10,11] In this hypothesis, for any set of e_λ, k, and ζ values the calculated magnetic moment decreases only slightly from room temperature to the 4.2 K value, as shown by the graphics in Figure 2, in agreement with previous calculations.[5] We tried then to use anisotropic k values, as is common practice in EPR spectroscopy.[12] We found, as shown in Figure 2, that in order to have the magnetic moment distinctly smaller than the spin-only value at liquid-helium temperature with room-temperature magnetic moments close to the observed values it is necessary to have small $k_\parallel$ values. Increasing $k_\parallel$ raises all the curves and decreases the difference in μ at room and liquid-helium temperature. The calculated magnetic moments and susceptibilities are shown in Figure 1, and the best-fit values are given in Table II. $k_\parallel$ is required to be slightly higher for the less symmetric thiocyanate derivative.

Although anisotropic k values are commonly used in the literature,it is by no means common to have such a large difference in $k_\perp$(k_x and k_y) and $k_\parallel$(k_z), with such a small $k_\parallel$. Recently Gerloch observed that in ordder to reproduce the experimental magnetic anisotropy of pseudotetrahedral nickel(II) complexes anomalously low values of k and ζ must be used,[13,14] while normal values are required for the corresponding cobalt(II) complexes. He suggested that the anomaly could be explained either by the Ham[15] effect or by the admixture of the ligand orbitals into the ground state. While, in the cases he considered, the latter could be an explanation, since he considered bromo and iodo derivatives, in our case, when essentially similar results are found for the bromo and the thiocyanate derivatives, the first explanation seems more feasible.

The Ham effect requires that the first-order orbital angular momentum matrix elements be reduced by vibronic coupling.[15] In D_{3h} symmetry, the orbital E′ level has first-order orbital angular momentum only parallel to z; therefore, the vibronic quenching will occur only parallel to the axis, thus determining a small $k_\parallel$ value. Since no orbital angular momentum is predicted parallel to x and y, the vibronic quenching will not operate in this case and $k_\perp$ will take the normal values expected for a nickel(II) complex. When the symmetry is lower than D_{3h}, the above considerations are no longer strictly valid, but they will keep much of their validity as long as the symmetry does not depart too much from D_{3h}. As a matter of fact, we find smaller k_z values for the more symmetric bromide as compared to those for the thiocyanate derivative, suggesting a larger quenching for the former.

It seems safe to conclude therefore that the Ham effect is responsible for the low magnetic moments of high-spin trigonal-bipyramidal nickel(II) complexes, yielding a large vibronic quenching of the z component of the orbital angular momentum.

Acknowledgment. Thanks are due to Prof. A. Vacca, ISSECC, CNR, Florence, Italy, and Prof. R. D. Willett, Washington State University, for allowing us to measure the magnetic susceptibility of the compounds in the range 4.2–295 K.

Registry No. [Ni(Me$_6$tren)Br]Br, 57036-60-9; [Ni(Me$_6$tren)-NCS]SCN·H$_2$O, 38883-26-0.

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Synthesis of Sodium and Tetra-*n*-butylammonium Dicyanodihydroborates

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The cyanotrihydroborate ion, BH$_3$CN$^-$, has been extensively employed as a highly selective reducing agent in organic synthesis.[1] Its utility is derived in part by the electron-withdrawing cyano group, moderating its reducing ability and imparting enhanced stability in protic solvents and acidic media. A further replacement of hydride by cyano to give BH$_2$(CN)$_2^-$ would be expected to lead to further moderation of reducing ability. We have now prepared the BH$_2$(CN)$_2^-$ anion as the sodium and tetra-*n*-butylammonium salts and have carried out some preliminary experiments to assess its reducing capability in contrast with sodium cyanotrihydroborate.

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Experimental Section

General Procedures. IR spectra were recorded on a Perkin-Elmer 297 spectrometer. Proton NMR spectra were recorded on a Varian EM 360A spectrometer whereas boron NMR spectra were obtained on a JEOL FX 90Q spectrometer. Elemental analyses were performed by Galbraith Laboratories, Inc., Knoxville, TN. The following compounds were purchased and used without further purification: $C_6H_5NH_2$ (Eastman Kodak), $NaBH_3CN$ (Aldrich), $(C_2H_5)_4NCl$ (Aldrich), *n*-Bu_4NHSO_4 (Aldrich), NaCN (Aldrich). THF solvent was dried over KH and distilled prior to use. Aniline hydrochloride ($C_6H_5NH_2\cdot HCl$) was prepared by slowly adding concentrated HCl to a stirred aniline/C_6H_6 solution at 0 °C and purified by recrystallization from C_2H_5OH. Aniline–cyanoborane ($C_6H_5NH_2\cdot BH_2CN$) was prepared in 62% yield by reacting $C_6H_5NH_2\cdot HCl$ and $NaBH_3CN$ as previously described.[2]

Preparation of $NaBH_2(CN)_2$. To a solution of $C_6H_5NH_2\cdot BH_2CN$ (19.80 g, 0.15 mol) in 400 mL of dry THF in a 1-L flask was added NaCN (7.4 g, 0.15 mol), followed by an additional 100 mL of dry THF under N_2. The mixture was stirred at room temperature for $^1/_2$ h and then heated at reflux for 72 h at which time the mixture had turned a slightly grayish color. The mixture was cooled and filtered under N_2. The solvent was removed under vacuum to give a semisolid material. To this crude $NaBH_2(CN)_2$ was added CH_2Cl_2 until it was barely cloudy, and the solution was kept in the refrigerator to give extremely hygroscopic white needles: 9.4 g, (71.1%); IR (THF) 2380 (s, ν_{BH}), 2200 (s, ν_{CN}) cm^{-1}; ^{11}B NMR (Me_2SO-d_6, $BF_3\cdot Et_2O$) δ −36.35 (t, J_{B-H} = 92.8 Hz). An analytical sample was recrystallized from THF/CH_2Cl_2. Anal. Calcd for $C_2H_2N_2BNa$: C, 27.34; H, 2.29; N, 31.88. Found: C, 27.19; H, 2.30; N, 31.68.

Preparation of *n*-$Bu_4NBH_2(CN)_2$. A suspension of *n*-Bu_4NHSO_4 (16.98 g, 0.05 mol) in 25 mL of water was treated with a solution of NaOH (3.6 g, 0.09 mol) in 20 mL of water, and $NaBH_2(CN)_2$ (4.83 g, 0.055 mol) in 20 mL of water was added. A milky suspension was formed as soon as the aqueous solution of $NaBH_2(CN)_2$ was added to the NaOH and *n*-Bu_4NHSO_4 solution. The suspension was stirred magnetically for 2 h at room temperature and repeatedly extracted with CH_2Cl_2 (3 × 60 mL). The organic extracts were washed with water (3 × 50 mL) and dried over $MgSO_4$, and the solvent was removed under vacuum to give thick oily *n*-$Bu_4NBH_2(CN)_2$: 12.90 g (83.92%); IR (neat) 2360 (s, ν_{BH}), 2200 (s, ν_{CN}) cm^{-1}. The oily material failed to crystallize in CH_2Cl_2/hexane, C_2H_5OH/hexane, and CH_2Cl_2/Et_2O solvent systems. The oily material left after removing solvent was vacuum pumped for approximately 1 month, after which time an oily solid material was obtained. Anhydrous ether was added to this material and the resultant mixture kept in the freezer. After a few days, white crystalline material was obtained and this was repeatedly washed with ether and vacuum pumped: IR ($CDCl_3$) 2995 (s, ν_{CH}), 2380 (s, ν_{BH}), 2250 (s, ν_{CN}), 2200 (w, ν_{CN}) cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 1.0 (distorted t, CH_3), 1.25–1.80 (m, CCH_2CH_2C), 3.0–3.33 (m, CCH_2N); ^{11}B NMR ($CDCl_3$, $BF_3\cdot Et_2O$) δ −41.44 (t, J_{B-H} = 92.8 Hz). Anal. Calcd for $C_{18}H_{38}N_3B$: C, 70.35; H, 12.46; N, 13.67; B, 3.52. Found: C, 70.15; H, 12.62; N, 13.58; B, 3.64.

Preparation of $(C_2H_5)_4NBH_2(CN)_2$ and Attempted Isolation. A solution of $NaBH_2(CN)_2$ (0.9664 g, 0.011 mol) and $(C_2H_5)_4NCl$ (1.6571 g, 0.010 mol) in 30 mL of water was stirred at room temperature for 1 h. To this solution was added 50 mL of CH_2Cl_2, and the mixture was stirred for an additional 1 h. The organic layer was separated, and the aqueous layer was extracted with (3 × 50 mL) CH_2Cl_2. The combined organic layer was washed with 50 mL of water, dried over $MgSO_4$, and evaporated by a rotary evaporator. An oily $Et_4NBH_2(CN)_2$ was obtained: 1.29 g (76.3%); IR (CH_2Cl_2) 2375 (s, νBH), 2200 (m, ν_{CN}) cm^{-1}. Attempts to crystallize this oily $Et_4NBH_2(CN)_2$ in different solvents were unsuccessful.

Stability of $NaBH_2(CN)_2$ in HCl. $NaBH_2(CN)_2$ is not stable in 1 N HCl and suffers considerable decomposition with the evolution of H_2 gas. A small amount (ca. 0.5 g) of $NaBH_2(CN)_2$ was treated with 10 mL of 1 N HCl and stirred at room temperature for 20 h. H_2 gas was evolved slowly. The material was extracted with THF/CH_2Cl_2 and dried, and the solvent was removed. The IR spectrum of this oily material showed medium-intensity bands due to νBH (2390 cm^{-1}), νC≡N (2210 cm^{-1}), and boric acid (3200 cm^{-1}, br). The oily material was again treated with 25 mL of 1 N HCl and stirred overnight. No further gas evolution was noticed. After usual workup, the oily material obtained had IR bands at 1660 (br s) and 3300 cm^{-1} (br s) in addition to very weak B—H and C≡N stretching.

Studies of Chemical Properties of $BH_2(CN)_2^-$. a. Attempted Reductions of Acetophenone with $NaBH_2(CN)_2$. (i) A mixture of acetophenone (0.48 g, 4.0 mmol) and excess $NaBH_2(CN)_2$ (0.35 g, 4.0 mmol) in 30 mL of methanol, to which was added 2 mL of 1 N HCl (pH ~2), was stirred at room temperature for 24 h. The reaction mixture gave a positive DNPH test for ketone. Another 4 mmol of $NaBH_2(CN)_2$ was added, followed by 3 mL of 1 N HCl (pH ~1), and the solution was refluxed for 24 h. There was no indication of reduction of the ketone (by IR and NMR spectroscopy and DNPH test).

(ii) A mixture of acetophenone (0.48 g, 4.0 mmol) and a large excess of $NaBH_2(CN)_2$ (1.05 g, 12 mmol) in 60 mL of anhydrous methanolic HCl (pH ~1) was stirred at room temperature for 16.5 h, followed by refluxing for 22 h. An additional 20 mL of CH_3OH/HCl was added, and heating of the mixture continued for 3 days. The progress of the reaction was monitored by IR spectral examination and DNPH test of an aliquot of the reaction mixture. The starting ketone was recovered.

(iii) The reaction was repeated in 25 mL of HMPA and 2 mL of concentrated H_2SO_4 at room temperature for 20 h. Some gas was evolved. Spectral examination of the reaction mixture indicated that no reduction took place. The starting material was decomposed.

b. Attempted Reductions of Acetophenone with *n*-$Bu_4NBH_2(CN)_2$. (i) A mixture of acetophenone (0.48 g, 4.0 mmol) and an excess of *n*-$Bu_4NBH_2(CN)_2$ (2.45 g, 8.0 mmol) in 50 mL of methanolic HCl was stirred at room temperature for 16.5 h, followed by refluxing for 3 days with fresh CH_3OH/HCl added. After workup (extracted with $CHCl_3$, washed with H_2O, dried, and solvent removed), the starting material was recovered and no alcohol was formed.

(ii) The reaction was repeated in methanolic HCl at reflux for 68 h, but no reduction of the ketone took place.

c. Attempted Hydrogenolysis of $C_7H_{15}I$ with $NaBH_2(CN)_2$. (i) A mixture of heptyl iodide (0.452 g, 2 mmol) and $NaBH_2(CN)_2$ (0.702 g, 8 mmol) in 15 mL of HMPA was stirred at room temperature for 16 h. After workup (addition of 20 mL of H_2O, extraction with ether, drying), the pale yellow liquid was analyzed by gas chromatography; no heptane was produced.

(ii) The reaction was repeated at 90 °C for 16 h with no success.

Results and Discussion

$NaBH_2(CN)_2$ has been prepared in 71% yield by cyanide displacement of the weak base aniline in the complex aniline–cyanoborane:

$$C_6H_5NH_2\cdot BH_2CN + NaCN \rightarrow NaBH_2(CN)_2 + C_6H_5NH_2$$

The salt, which is very hygroscopic, has been characterized by elemental analysis and ^{11}B NMR and IR spectroscopy. The *n*-$Bu_4NBH_2(CN)_2$ salt was prepared from $NaBH_2(CN)_2$ in a manner analogous to that described for the preparation of the corresponding *n*-Bu_4NBH_3CN[3] from $NaBH_3CN$. It was characterized by elemental analysis and ^{11}B and ^{1}H NMR and IR spectroscopy. The ν_{CN} of 2200 cm^{-1} observed in $H_2B(CN)_2^-$ is shifted to a higher frequency than that of 2179 cm^{-1} found[4] in BH_3CN^- as might be expected. No evidence could be found for isocyano attachment to boron, as an absorption to a lower frequency would be expected (cf. ν_{NC} at 2065 cm^{-1} in $NaBH_3NC$[5]). The formation of the more stable dicyano (rather than a mixed cyano–isocyano hydroborate) is not surprising in view of the preparative conditions (prolonged reflux in THF).

In preliminary studies of the reducing ability of $BH_2(CN)_2^-$ compared to BH_3CN^-, it was found that $BH_2(CN)_2^-$ failed to reduce selected organic functional groups, which H_3BCN^- does readily. Thus, in acidic media, $NaBH_3CN$ reduces

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acetophenone to benzyl alcohol in 93% in yield in 1 h at room temperature[6] whereas, even under much more forcing conditions, acetophenone was not reduced by $NaBH_2(CN)_2$ or n-$Bu_4NBH_2(CN)_2$. Likewise, $NaBH_3CN$ in hexamethylphosphoramide readily reduces alkyl halides to the corresponding alkane[7] but no evidence could be found for the reduction of heptyl iodide under much more forcing conditions.

We conclude that the reducing ability of $BH_2(CN)_2^-$ is appreciably less than that of H_3BCN^-, and experiments to exploit the difference and its reducing ability toward other functional groups are in progress.

Acknowledgment. The experimental assistance of Mark Scheitlin is gratefully acknowledged. We thank the Army Research Office for support of this work.

Note Added in Proof. Alternate preparations of $BH_2(CN)_2^-$ have now been published: $LiBH_2(CN)_2$ (Gyori, B.; Emri, J.; Feher, I. *J. Organomet. Chem.* **1983**, *225*, 17) and $NaBH_2(CN)_2$ (Emri, J.; Gyori, B. *J. Chem. Soc., Chem. Commun.* **1983**, 1303).

Registry No. $NaBH_2(CN)_2$, 88503-36-0; $C_6H_5NH_2{\cdot}BH_2CN$, 66632-44-8; n-$Bu_4NBH_2(CN)_2$, 91128-60-8; $Et_4NBH_2(CN)_2$, 91128-61-9; $C_7H_{15}I$, 4282-40-0; acetophenone, 98-86-2.

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First Observation of Resolvable Coupling between Manganese-55 and Carbon-13

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During our studies of the electron-transfer reaction[1,2]

$$(RNC)_6Mn^+ + (RNC)_6Mn^{2+} \rightleftarrows (RNC)_6Mn^{2+} + (RNC)_6Mn^+$$

(where R = methyl, ethyl, isopropyl, *tert*-butyl, cyclohexyl, and benzyl) we encountered discrepancies in the literature regarding the spectroscopic and electrochemical properties of these complexes. We, therefore began a thorough characterization of the physical properties of them.[2] We report here $^1J(^{55}Mn-^{13}C)$ for R = methyl, ethyl, isopropyl, *tert*-butyl, and cyclohexyl in $(RNC)_6MnBF_4$. This is the first direct observation of resolvable coupling between ^{55}Mn and ^{13}C.

The observation of the ^{13}C NMR signal of a carbon atom bonded directly to a metal atom with a large quadrupole moment is usually difficult because the electric field gradients of the quadrupolar nucleus provide an efficient relaxation mechanism for it and thus a broadened ^{13}C signal.[3,4] Due to the low natural abundance of ^{13}C, this broadened signal is often unobservable. Several researchers have performed NMR experiments at lower temperatures in order to cause a shorter T_1 for the quadrupolar nucleus and thus "thermally decouple" the ^{13}C spin from the quadrupolar nucleus.[5,6] This gives sharpened ^{13}C signals, but the spin-spin coupling information is lost. For the case of symmetric complexes such as the octahedral $(RNC)_6MnBF_4$ series,[7] the electric field gradients are averaged out to nearly zero. This removes the major relaxation mechanism, and better resolved spectra result. The reason $^{55}Mn-^{13}C$ coupling constants are previously unreported is primarily the lack of diamagnetic, symmetric, organometallic complexes of manganese. A ^{55}Mn NMR study on $Mn(CO)_6^+$ has been reported, but the authors did not report any resolvable coupling of ^{13}C to ^{55}Mn.[8] The only previously reported coupling constant of a nucleus bonded to ^{55}Mn is for ^{17}O in MnO_4^-.[9,10] A further problem, at least for the isocyanide series, is that the coupling is not observable as satellites in the ^{55}Mn spectrum. This occurs because the center of the satellites is isotopically shifted 36 Hz upfield, which causes the downfield satellite to become hidden under the large singlet. The ^{55}Mn spectrum thus appears as a large peak with a smaller peak slightly upfield. For wider lines, the small peak is a shoulder on the larger one.

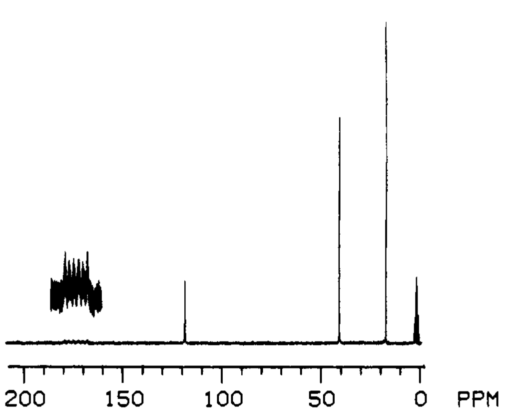

Figure 1. ^{13}C NMR spectrum for saturated $(CH_3CH_2NC)_6MnBF_4$ in CD_3CN at 50 °C. NMR experimental conditions are found in the footnotes to Table I. The septet at 1.2 ppm and the peak at 118 ppm are due to CD_3CN. The peaks at 16 and 40 ppm are due to CH_3 and CH_2, respectively. The insert above the 175 ppm region is the multiplet enlarged 10 times to show the fine structure due to the $^{55}Mn-^{13}C$ coupling. All shifts were measured relative to CD_3CN, which was taken to be 1.2 ppm downfield from Me_4Si.

The ^{13}C NMR spectra of $(RNC)_6MnBF_4$ (R = methyl, ethyl, isopropyl, *tert*-butyl, cyclohexyl, and benzyl) were collected at room temperature in CD_3CN, and six peaks were resolved for the isocyano carbon in all cases except for the benzyl isocyanide complex, which showed a broad absorption with no resolvable splitting. The resolution of the splitting became poorer as the R group increased in size from methyl to cyclohexyl. An additional spectrum of the ethyl isocyanide complex obtained at 50 °C gave better resolution, and the total width of the multiplet was equal to that of the spectrum taken at room temperature.

Figure 1 shows the ^{13}C NMR spectrum of $(CH_3CH_2NC)_6MnBF_4$ in CD_3CN at 50 °C. The insert shows the isocyano carbon region of the spectrum enlarged 10 times to exhibit the fine structure. Since ^{55}Mn has a spin of $^5/_2$, six lines are observed in the ^{13}C NMR spectrum for the carbon bonded to the manganese. They have different intensities and widths but the same area. It has been shown,[12] for the NMR spectrum of a nucleus of spin $^1/_2$ bonded to a nucleus of spin $^5/_2$, that six lines of equal intensity will be observed only if the spin-

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