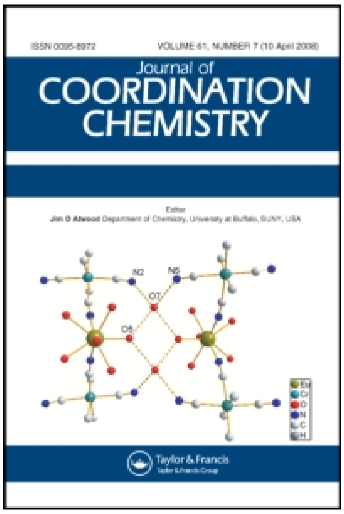

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Novel syntheses of tetrakis(benzylisocyanide)bis (tri-*p*-methoxyphenylphosphine)cobalt(III) tetrafluoroborate

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Co(I), Co(II) and Co(III) complexes containing the same ligands, $[Co(CNCH_2Ph)_3\{P(C_6H_4OMe\text{-}p)_3\}_2]BF_4$ (**1**), $[Co(CNCH_2Ph)_3\{P(C_6H_4OMe\text{-}p)_3\}_2](BF_4)_2$ (**2**) and $[Co(CNCH_2Ph)_4\{P(C_6H_4OMe\text{-}p)_3\}_2](BF_4)_3$ (**3**), have been synthesized and characterized. Syntheses of **3** by disproportionation or competing disproportionation and reduction reactions of the Co(II) complex $[Co(CNCH_2Ph)_5]^{2+}$, and by $AgBF_4$ oxidation of **2**, are both unique reactions for alkylisocyanidetriarylphosphine complexes of Co(II). The physical properties of **1**, **2** and **3** give little indication of their novel synthetic behavior.

Keywords: Alkylisocyanide; Triarylphosphine; Cobalt(III) complex; Disproportionation reaction; Cobalt(II) complex

1. Introduction

Co(II) complexes of alkylisocyanides react differently with triarylphosphines and trialkylphosphines. With the former, the Co(II) complexes undergo reduction/ligand-substitution leading to Co(I) complexes of general formula $[Co(CNR)_3(PR'_3)_2]X$, $X = ClO_4$, BF_4 [1–6]. With the latter, disproportionation/ligand-substitution generally takes place, giving both Co(I) and Co(III) complexes of the type $[Co(CNR)_3(PR'_3)_2]X$ and *trans*-$[Co(CNR)_4(PR'_3)_2]X_3$, respectively, for $PR'_3 = P(C_3H_7\text{-}n)_3$, $P(C_4H_9\text{-}n)_3$, $P(C_6H_{13}\text{-}n)_3$ [5,7,8]. The complex $[Co(CNCMe_3)_4H_2O](ClO_4)_2$, however, yields only $[Co(CNCMe_3)_3(PR'_3)_2]ClO_4$ with these trialkylphosphines and with the ligands $P(CH_2CH_2CN)_3$, $P(CH_2Ph)_3$ and $P(NMe_2)_3$, which also induce reduction/ligand-substitution in other Co(II) alkylisocyanides [4,5]. Disproportionation reactions of Co(II) with trialkylphosphites are well known [9].

Some of the Co(I) complexes resulting from reaction with triarylphosphines can be oxidized to the corresponding Co(II) complexes, $[Co(CNR)_3(PR'_3)_2]X_2$, by reaction with $AgClO_4$ or $AgBF_4$ [10,11]. Pairs of chemically interconvertible Co(I) and Co(II)

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complexes (i.e. Co(I) oxidized with Ag^+, Co(II) reduced with N_2H_4) show reversible cyclic voltammograms in CH_3CN solution [12].

This paper reports an apparent disproportionation of $[Co_2(CNCH_2Ph)_{10}](BF_4)_4 \cdot H_2O$ with $P(C_6H_4OMe\text{-}p)_3$ and oxidation of $[Co(CNCH_2Ph)_3\{P(C_6H_4OMe\text{-}p)_3\}_2](BF_4)_2$ with $AgBF_4$, both reactions leading to the Co(III) complex $[Co(CNCH_2Ph)_4\{P(C_6H_4OMe\text{-}p)_3\}_2](BF_4)_3$ (**3**). This complex has been previously synthesized by ligand-substitution of $[Co(CNCH_2Ph)_4\{OAs(C_6H_4Me\text{-}p)_3\}_2](BF_4)_3$ and $[Co(CNCH_2Ph)_4(OSbPh_3)_2](BF_4)_3$ with $P(C_6H_4OMe\text{-}p)_3$ [13]. These syntheses of **3** are novel in that neither the disproportionation induced by a triarylphosphine ligand nor the Ag^+ oxidation of an alkylisocyanidetriarylphosphine Co(II) complex has been observed before.

2. Experimental

2.1. *Reagents*

Benzylisocyanide ($CNCH_2Ph$), tri-*p*-methoxyphenylphosphine [$P(C_6H_4OMe\text{-}p)_3$], and anhydrous silver(I) tetrafluoroborate ($AgBF_4$) were obtained from Sigma-Aldrich S.A. Anhydrous diethylether was filtered through an alumina column immediately before use. $[Co_2(CNCH_2Ph)_{10}](BF_4)_4 \cdot H_2O$ was prepared as reported previously [14]. For cyclic voltammetry, 1 mM solutions of the compounds were freshly prepared in chromatographic-grade acetonitrile containing 0.05 M tetrabutylammonium hexafluorophosphate (TBAH) as supporting electrolyte.

2.2. *Instrumentation*

IR spectra were recorded on a Perkin Elmer 2000 FTIR instrument. Solution electronic spectra were recorded on a Shimadzu UV-2501 PC spectrophotometer over the range 1100–200 nm. Magnetic susceptibility was measured at room temperature using a Johnson Matthey Alfa balance. The effective magnetic moment was calculated assuming Curie law behavior; diamagnetic corrections were taken from the literature [8,15,16]. C, H and N analyses were performed using Carlo Erba CHN-O/S 1106 and Vario EL CHNOS instruments. Cyclic voltammetry measurements were performed using a Metrohm 757 VA Computerace system with a three-electrode compartment cell. The working and auxiliary electrodes were both made of glassy carbon. The reference electrode was a double junction Ag–AgCl system. Voltammograms were recorded in the potential range -1.2 to $+1.2$ V *vs* Ag–AgCl, at a $0.1\,V\,s^{-1}$ scan rate. Nitrogen gas was bubbled through each solution for 500 s before the run.

2.3. $[Co(CNCH_2Ph)_3\{P(C_6H_4OMe\text{-}p)_3\}_2]BF_4$ *(1) and* $[Co(CNCH_2Ph)_4\{P(C_6H_4OMe\text{-}p)_3\}_2](BF_4)_3$ *(3)*

$[Co_2(CNCH_2Ph)_{10}](BF_4)_4 \cdot H_2O$ (500 mg, 0.604 mmol) was dissolved in CH_2Cl_2 (1.5 cm^3), filtered through cotton with a CH_2Cl_2 (0.5 cm^3) rinse, and chilled in ice. Then $P(C_6H_4OMe\text{-}p)_3$ (745 mg, 2.11 mmol; 1 : 3.5 Co : P mol ratio) dissolved in CH_2Cl_2 (3.0 cm^3) was added dropwise while the chilled Co(II) solution was stirred. The yellow–brown solution became dark red upon the addition. Diethylether was immediately added dropwise at room temperature, 5.0 cm^3 for initial precipitation, 10.0 cm^3 total. The reaction mixture was chilled in ice (50 min), and the bright red–orange, microcrystalline product was filtered off and washed twice with diethylether. The crude product (164 mg) was recrystallized from CH_2Cl_2 (6.5 cm^3) and diethylether (12.0 cm^3). Yield: 120 mg (13%), m.p. 180–184°C (dec.). Anal. Calcd. for $C_{74}H_{70}B_3F_{12}CoO_6P_3 \cdot 1.0CH_2Cl_2$(%): C, 57.10; H, 4.60; N, 3.55. Found: C, 57.00; H, 4.66; N, 3.55. Overnight refrigeration of the original filtrate afforded a substantial crop (478 mg) of golden yellow microcrystals. This crude product was recrystallized from CH_2Cl_2 (1.5 cm^3) and diethylether (4.0 cm^3). Yield: 240 mg (31%), m.p. 152–155°C (dec.). Anal. Calcd. for $C_{66}H_{63}BF_4CoO_9P_3 \cdot 0.1CH_2Cl_2$(%): C, 65.59; H, 5.26; N, 3.47. Found: C, 65.41; H, 5.29; N, 3.46.

2.4. *$[Co(CNCH_2Ph)_3\{P(C_6H_4OMe$-p$)_3\}_2](BF_4)_2$ (2)*

Complex **1** (200 mg, 0.129 mmol) was dissolved in CH_2Cl_2 (2.0 cm^3 total) and filtered through cotton. $AgBF_4$(s) (28 mg, 0.14 mmol; 1 : 1.1 Co : Ag mol ratio) was added at room temperature, and continuously triturated with a glass stirring rod for 5 min. The solution immediately took on a rust-red colour, while the $AgBF_4$ rapidly blackened and a silver mirror formed on the bottom of the flask. The heterogeneous reaction mixture was then decanted and filtered twice through cotton with a CH_2Cl_2 (1.0 cm^3) rinse each time. Diethylether was added in small aliquots to the clear, blood-red solution; 2.5 cm^3 for initial precipitation, 5.0 cm^3 total. The reaction mixture was chilled in ice (45 min) and the yellow–brown microcrystalline product was filtered and washed twice with diethylether. Crude product (156 mg) was recrystallized from CH_2Cl_2 (1.5 cm^3) and diethylether (2.0 cm^3). Yield: 127 mg (59%), m.p. 188–192°C (dec.). Anal. Calcd. for $C_{66}H_{63}B_2F_8CoN_3O_6P_2 \cdot 1.0CH_2Cl_2$(%): C, 58.59; H, 4.77; N, 3.06. Found: C, 58.42; H, 4.82; N, 3.12.

2.5. *Alternative synthesis of $[Co(CNCH_2Ph)_4\{P(C_6H_4OMe$-p$)_3\}_2](BF_4)_3$ (3)*

Complex **2** (150 mg, 0.115 mmol) was dissolved in CH_2Cl_2 (3.0 cm^3) and filtered through cotton. $AgBF_4$ (125 mg, 0.64 mmol) was added at room temperature and triturated with a glass stirring rod. After 5 min, a black film could be observed in the flask, and excess $AgBF_4$(s) slowly darkened to a gray sludge. After 15 min the heterogeneous mixture was decanted and filtered through cotton with a CH_2Cl_2 (1.0 cm^3) rinse. Diethylether was then added in small aliquots to the clear, dark orange solution; 2.5 cm^3 for initial precipitation, 5.0 cm^3 total. The reaction mixture was chilled in ice (30 min) and the bright red–orange, microcrystalline product was filtered and washed twice with diethylether. Yield: 81.2 mg (61%), m.p. 178–184°C (dec.). Anal. Calcd. for $C_{74}H_{70}B_3F_{12}CoO_6P_3 \cdot 0.7CH_2Cl_2$(%): C, 57.80; H, 4.64; N, 3.61. Found: C, 57.72; H, 4.79; N, 3.66.

3. Results and discussion

Tetrakis(benzylisocyanide)bis(tri-*p*-methoxyphenylphosphine)cobalt(III) tetrafluoroborate, $[Co(CNCH_2Ph)_4\{P(C_6H_4OMe\text{-}p)_3\}_2](BF_4)_3$ (**3**), and tris(benzylisocyanide) bis(tri-*p*-methoxyphenylphosphine)cobalt(I) tetrafluoroborate, $[Co(CNCH_2Ph)_3\{P(C_6H_4OMe\text{-}p)_3\}_2]BF_4$ (**1**), have been synthesized, in unequal yields, by reaction of

$P(C_6H_4OMe\text{-}p)_3$ (in 3.5 : 1 P : Co mol ratio) with $[Co_2(CNCH_2Ph)_{10}](BF_4)_4 \cdot H_2O$

$$2[Co(CNCH_2Ph)_5](BF_4)_2 \xrightarrow{7PR_3,\,CH_2Cl_2} [Co(CNCH_2Ph)_4\{P(C_6H_4OMe\text{-}p)_3\}_2](BF_4)_3$$
$$+ [Co(CNCH_2Ph)_3\{P(C_6H_4OMe\text{-}p)_3\}_2]BF_4$$

Tris(benzylisocyanide)bis(tri-*p*-methoxyphenylphosphine)cobalt(II)tetrafluoroborate, $[Co(CNCH_2Ph)_3\{P(C_6H_4OMe\text{-}p)_3\}_2](BF_4)_2$ (**2**), has been synthesized by oxidation of **1** with $AgBF_4$,

$$[Co(CNCH_2Ph)_3\{P(C_6H_4OMe\text{-}p)_3\}_2]BF_4$$
$$\xrightarrow{AgBF_4} [Co(CNCH_2Ph)_3\{P(C_6H_4OMe\text{-}p)_3\}_2](BF_4)_2$$

and **3** has also been synthesized by oxidation of **2** with $AgBF_4$.

$$4[Co(CNCH_2Ph)_3\{P(C_6H_4OMe\text{-}p)_3\}_2](BF_4)_2$$
$$\xrightarrow{AgBF_4} 3[Co(CNCH_2Ph)_4\{P(C_6H_4OMe\text{-}p)_3\}_2](BF_4)_3$$

The physical properties of the complexes include the following.

*$[Co(CNCH_2Ph)_3\{P(C_6H_4OMe\text{-}p)_3\}_2]BF_4$ (**1**).* IR (cm^{-1}): $\nu(N{\equiv}C)$ 2058 vs, ~2087 m(sh), 2143 m (Nujol); 2085 vs, ~2136 vw(sh) (CH_2Cl_2); 2087 vs, ~2137 vw(sh) (CH_3NO_2); 2088 vs, ~2137 vw(sh) (CF_3CH_2OH). Electronic spectra: λ_{max} (ε, M^{-1} cm^{-1}): ~355 sh (~2800), 299 (36,000), ~283 sh, ~276 sh, 248 (78,000) nm (CH_2Cl_2); ~345 sh (~1300), 296 (34,000), ~283 sh, ~274 sh, 243 (76,000) nm (CH_3CN); ~350 sh (~1300), 295 (33,000), ~282 sh, ~273 sh, 246 (73,000) nm (CF_3CH_2OH). Cyclic voltammogram: $E_{ox} = 179$ mV, $E_{red} = 83.5$ mV, $E_{1/2} = 131$ mV *vs* Ag–AgCl, $\Delta E = 96$ mV.

*$[Co(CNCH_2Ph)_3\{P(C_6H_4OMe\text{-}p)_3\}_2](BF_4)_2$ (**2**).* IR (cm^{-1}): $\nu(N{\equiv}C)$ 2222 m, 2194 vs, ~2155 vw(sh) (Nujol); 2192 m, ~2108 vw(sh), 2088 s (CH_2Cl_2); ~2220 vw(sh), 2196 vs, ~2094 m (CH_3NO_2); 2244 w, 2193 vs (CF_3CH_2OH). Electronic spectra: λ_{max} (ε, M^{-1} cm^{-1}): ~910 br (350), 472 (4350), 392 (14,400), 353 (10,800), ~305 sh (11,800), 251 (76,200) nm (CH_2Cl_2); ~910 br (410), 476 (3960), 390 (10,100), 358 (7700), 247 (72,100) nm (CH_3CN); ~915 br (310), 477 (3500), 395 (14,100), 353 (9700), 308 (10,200), 251 (80,100), 214 (72,100) nm (CF_3CH_2OH). Magnetic susceptibility: $\chi_g = (1.02 \pm 0.09) \times 10^{-6}$ (cgs), $\mu_{eff} = 2.24 \pm 0.06$ BM. Cyclic voltammogram: $E_{red} = 83.3$ mV, $E_{ox} = 167$ mV, $E_{1/2} = 125$ mV *vs* Ag–AgCl, $\Delta E = 84$ mV.

*$[Co(CNCH_2Ph)_4\{P(C_6H_4OMe\text{-}p)_3\}_2](BF_4)_3$ (**3**).* Data concerning $\nu(N{\equiv}C)$ IR, solution electronic spectra and magnetic susceptibility have been reported [13]. Cyclic voltammogram: $E_{red} = 107$ mV, $E_{ox} = 173$ mV, $E_{1/2} = 140$ mV *vs* Ag–AgCl, $\Delta E = 66$ mV.

3.1. *Synthesis of the complexes*

The reaction of $[Co_2(CNCH_2Ph)_{10}](BF_4)_4 \cdot H_2O$, as $[Co(CNCH_2Ph)_5]^{2+}$ in solution, with excess $P(C_6H_4OMe\text{-}p)_3$ is either a disproportionation or competing reduction and disproportionation reaction. This reaction was performed several times, and yields of the Co(III) product (**3**) were always substantially lower than 50%, while yields of the Co(I) product (**1**) sometimes exceeded 50% even after recrystallization. If yields of **3** decrease, yields of **1** increase. This is unlike reactions using trialkylphosphines, where yields of the Co(III) products often approached 50%, but the Co(I) product was seldom recovered [5,7,8]. With trialkylphosphine ligands, the Co(III) complexes are stable and very slightly soluble in CH_2Cl_2, whereas the Co(I) complexes are very soluble and relatively unstable. However, complex **3** is relatively unstable and appreciably soluble in CH_2Cl_2 [13], while **1** is stable. Although it is possible that equal amounts of **3** and **1** are formed in disproportionation and **3** partially reduces to **1** before isolation, the absence of any Co(II) product supports a competing reduction and disproportionation reaction route. In previous reactions of alkylisocyanide Co(II) complexes with triarylphosphines, Co(II) species were sometimes observed, but not Co(III) species [4–6,10,11,17,18].

Oxidation of **1** to **2** with $AgBF_4$ is analogous to previously reported oxidations of Co(I) complexes to five-coordinate Co(II) species [11,19], except that slight amounts of **3** may be formed before all of **1** has been oxidized to **2**. Best results are obtained by stopping the reaction when only **1** and **2** are present, as any **1** is easily removed by recrystallization.

Oxidation of **2** to **3** with excess $AgBF_4$ is unexpected because a fourth benzylisocyanide ligand must be abstracted from a second cobalt moiety. The mechanism for this reaction is currently unknown. Excess $AgBF_4$ should not "drive" the reaction, because $AgBF_4(s)$ is not soluble in CH_2Cl_2, although increasing the amount seems to favor the reaction, possibly by providing more surface area. A Co(III) species was not observed in the syntheses of other Co(II) complexes with Ag^+ [11]. This oxidation and the disproportionation or disproportionation and reduction reaction, therefore, are novel for the $CNCH_2Ph\text{-}P(C_6H_4OMe\text{-}p)_3$ combination of ligands.

3.2. *Physical properties of the complexes*

Physical properties of complexes **1**, **2** and **3** are analogous to those reported for other five-coordinate complexes of Co(I) and Co(II), and six-coordinate complexes of Co(III) with alkylisocyanide and triarylphosphine ligands [4–6,10–13], except for subtle differences. The $\nu(N{\equiv}C)$ IR pattern for **1** in Nujol is analogous to spectra of several Co(I) complexes [4–6], but the solution spectra are similar only to $[Co(CNCH_2Ph)_3(PPh_3)_2]BF_4$ [12]. The $\nu(N{\equiv}C)$ IR pattern for **2** in Nujol is analogous only to $[Co(CNCH_2Ph)_3(PPh_3)_2](BF_4)_2$ [11], whereas the pattern in CH_3NO_2, showing slight reduction to Co(I), is shared with other Co(II) complexes [11,12]. Complex **2** in CH_2Cl_2, showing extensive reduction, is analogous to $[Co(CNCH_2Ph)_3(PPh_3)_2](BF_4)_2$, but similar to other Co(II) complexes that exhibit moderate reduction [12]. Complex **2** in CF_3CH_2OH is unique, however, in showing apparent oxidation to Co(III), albeit slight. Complex **3** exhibits moderate to extensive reduction in all three solvents, analogous to other $[Co(CNR)_4(PR'_3)_2]X_3$ complexes [13].

Electronic spectra for **1**, **2**, and **3** are analogous to those reported for similar Co(I), Co(II) and Co(III) species [4–6,10–13]. Complex **2** and especially **3** have limited stability in solution. The magnetic susceptibility of **2** is within the range previously reported for $[Co(CNR)_3(PR'_3)_2]X_2$ complexes [11], and the effective magnetic moment (2.24 BM) is within the range normally observed for low-spin Co(II) complexes, i.e. 1.8–2.7 BM [15]. Complexes **1** and **3** are diamagnetic, as expected.

Cyclic voltammograms for complexes **1** and **2** are analogous to data reported for nine other pairs of $[Co(CNR)_3(PR'_3)_2]^+$ and $[Co(CNR)_3(PR'_3)_2]^{2+}$ complexes, in both $E_{1/2}$ and ΔE values [12], and are reversible. $E_{1/2}$ values for **1** and **2** are identical within experimental error (± 10 mV). The cyclic voltammogram for complex **3** is essentially the Co(I)–Co(II) cycle, consistent with **3** being extensively reduced in solution. No evidence for a Co(II)–Co(III) cycle was observed in any of the cyclic voltammograms. The apparent inconsistency of isolating complex **3** in chemical synthesis but not observing it electrochemically is probably due to concentration and reaction time effects. In dilute solution ($\sim$1 mM) near the electrode surface in the short time of the cycle, the five-coordinate Co(II) complex cannot readily abstract the extra $CNCH_2Ph$ ligand from a second moiety needed to stabilize Co(III). In concentrated solution ($\sim$40 mM) exposed to $AgBF_4$(s) for a longer time, the necessary exchange of the $CNCH_2Ph$ ligand obviously can take place.

The physical properties of complexes **1**, **2** and **3** (with the exception of the CF_3CH_2OH solution $\nu(N{\equiv}C)$ IR pattern for **2**) show no significant differences from the Co(I), Co(II) and Co(III) complexes previously investigated that would explain, or even anticipate, the two novel syntheses of complex **3**.

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References

[1] R.B. King, M.S. Saran, *Inorg. Chem.*, **11**, 2112 (1972).
[2] J.W. Dart, M.K. Lloyd, R. Mason, J.A. McCleverty, J. Williams, *J. Chem. Soc., Dalton Trans.*, 1747 (1973).
[3] E. Bordignon, U. Croatto, U. Mazzi, A.A. Orio, *Inorg. Chem.*, **13**, 935 (1974).
[4] C.A.L. Becker, A. Anisi, G. Myer, J.D. Wright, *Inorg. Chim. Acta*, **111**, 11 (1986).
[5] C.A.L. Becker, S.A. Al-Qallaf, J.C. Cooper, *Inorg. Chim. Acta*, **188**, 99 (1991).
[6] C.A.L. Becker, K.R. Barqawi, *J. Coord. Chem.*, **34**, 273 (1995).
[7] C.A.L. Becker, M.A.S. Biswas, J.C. Cooper, *Inorg. Chim. Acta*, **188**, 191 (1991).
[8] C.A.L. Becker, M.A.S. Biswas, *J. Coord. Chem.*, **29**, 277 (1993).
[9] S.M. Socol, J.G. Verkade, *Inorg. Chem.*, **25**, 2658 (1986) and references cited therein.
[10] C.A.L. Becker, *Synth. React. Inorg. Met.-Org. Chem.*, **27**, 1527 (1997).
[11] C.A.L. Becker, *J. Coord. Chem.*, **50**, 89 (2000).
[12] J. Ahmad, F. Bothata-Nareetsile, C.A.L. Becker, *Can. J. Chem.*, **81**, 1635 (2003).
[13] C.A.L. Becker, E.E. Mmatli, *J. Coord. Chem.*, **57**, 329 (2004).
[14] C.A.L. Becker, *Inorg. Chim. Acta*, **203**, 175 (1993).
[15] B.N. Figgis, J. Lewis, In *Modern Coordination Chemistry*, J. Lewis, R.G. Wilkins (Eds), Chapter 6, Interscience, New York (1960).
[16] C.A.L. Becker, *J. Coord. Chem.*, **28**, 147 (1993).
[17] C.A.L. Becker, *Synth. React. Inorg. Met.-Org. Chem.*, **22**, 99 (1992).
[18] C.A.L. Becker, *Synth. React. Inorg. Met.-Org. Chem.*, **28**, 1145 (1998).
[19] C.A.L. Becker, N. Samarakkody, *Synth. React. Inorg. Met.-Org. Chem.*, **29**, 1111 (1999).