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SYNTHESES OF POLYFLUOROPHENYLCOBALT(III) SCHIFF BASE COMPLEXES USING ORGANOTHALLIUM REAGENTS

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Summary

The complexes RCo(acacen) [R = C_6F_5, p-HC_6F_4, or o-HC_6F_4; H_2acacen = *N,N'*-ethylenebis(acetylacetonimine)] and RCo(salen) [R = C_6F_5 or p-HC_6F_4; H_2salen = *N,N'*-ethylenebis(salicylaldimine)] have been prepared by reaction between Co(acacen) or Co(salen) and the appropriate bromobis(polyfluorophenyl)-thallium(III) compounds, and have been isolated as pyridinates. Spectroscopic evidence for formation of C_6F_5Co(salophen) [H_2salophen = *N,N'*-o-phenylenebis-(salicylaldimine)] has also been obtained. The reactivity of the thallium compounds increased in the sequence $Ph_2TlBr \ll (o\text{-}HC_6F_4)_2TlBr < (p\text{-}HC_6F_4)_2TlBr < (C_6F_5)_2TlBr$, and of the cobalt complexes in the sequence Co(salophen) < Co(salen) < Co(acacen). Possible mechanisms are discussed.

Introduction

Bromobis(pentafluorophenyl)thallium(III) [1] oxidises elements [2,3], e.g. Zn, In, Ge, P, Te, and compounds in low oxidation states [4-7], e.g. $(Ph_3P)AuCl$, $(Ph_3P)_3RhCl$, $SnCl_2$, with elimination of thallous bromide and formation of pentafluorophenylorganometallics.

$$E + n(C_6F_5)_2TlBr \rightarrow (C_6F_5)_{2n}E + nTlBr \qquad (1)$$

$$EL_m + n(C_6F_5)_2TlBr \rightarrow (C_6F_5)_{2n}EL_m{}^{**} + nTlBr \qquad (2)$$

We now report preparations of polyfluorophenylcobalt(III) Schiff base complexes by this method. One related reaction, the conversion of $[Co(diars)_2]Br_2$ [diars = o-phenylenebis(dimethylarsine)] into [*trans*-$C_6F_5(Br)Co(diars)_2$]Br by $(C_6F_5)_2TlBr$ has been briefly mentioned in two reviews [5,8], but details have not appeared.

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** Some ligand molecules originally coordinated may be displaced during reaction, e.g. in conversion of $(Ph_3P)_2Ni(CO)_2$ into $(Ph_3P)_2Ni(C_6F_5)_2$ [6].

Results and discussion

The complexes RCo(acacen) [R = C_6F_5, *p*-HC_6F_4, or *o*-HC_6F_4; H_2acacen = *N,N'*-ethylenebis(acetylacetonimine)], RCo(salen) [R = C_6F_5 or *p*-HC_6F_4; H_2salen = *N,N'*-ethylenebis(salicylaldimine)], and C_6F_5Co(salophen) [H_2salophen = *N,N'*-*o*-phenylenebis(salicylaldimine)] have been prepared by reaction of the appropriate cobalt(II) Schiff base complexes and bromobis(polyfluorophenyl)thallium(III) compounds.

$$2\ \mathrm{CoL} + \mathrm{R_2TlBr} \rightarrow 2\ \mathrm{RCoL} + \mathrm{TlBr}\downarrow$$

(L = acacen, salen, or salophen; R as above) (3)

Details of reaction conditions and yields are given in Table 1. All complexes were isolated as pyridinates, the crude products from reactions in toluene being recrystallized from pyridine-containing media. Satisfactory analyses could not be obtained for RCo(acacen)py (R = *p*-HC_6F_4 or *o*-HC_6F_4), but the compounds gave analytically pure pyridine-free derivatives RCo(acacen) on being heated under vacuum. A satisfactory purification could not be devised for C_6F_5Co(salophen)py, and the compound was identified spectroscopically (see below). Attempts to prepare PhCo(acacen) and *o*-HC_6F_4CoL (L = salen or salophen) were unsuccessful (Table 1). Only one polyfluoroarylcobalt(III) Schiff base complex has been previously prepared, viz. C_6F_5Co(salen)py, which was obtained by the Grignard method [9].

The PMR spectra of RCo(acacen)py (R = C_6F_5, *p*-HC_6F_4, or *o*-HC_6F_4) and RCo(salen)py (R = C_6F_5 or *p*-HC_6F_4) (see Experimental) established that the Schiff base ligands remained unchanged after reaction with the organothallium derivatives, and the ^{19}F NMR spectra confirmed the presence of the appropriate polyfluorophenyl groups. Chemical shifts for the protons of the quadridentate ligands are similar to those of (Me or Ph)Co(acacen)py [10] or (Me or Ph)Co-

TABLE 1

REACTIONS OF COBALT(II) SCHIFF BASE COMPLEXES WITH BROMOBIS(POLYFLUOROPHENYL)THALLIUM(III) COMPOUNDS

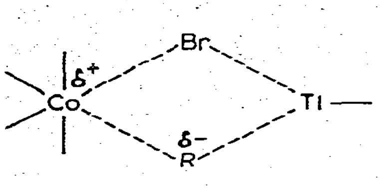
Cobalt complex	R_2TlBr R =	Solvent^{a}	Reaction time(min)	Product	Yield(%)
Co(acacen)	C_6F_5	MePh	15 (<1^{b})	C_6F_5Co(acacen)	84^{c}
	p-HC_6F_4	MePh	15 (4^{b})	*p*-HC_6F_4Co(acacen)	60^{c}
	o-HC_6F_4	MePh	15 (8^{b})	*o*-HC_6F_4Co(acacen)	54^{c}
	Ph	py	240		
Co(salen)	C_6F_5	MePh	15 (<1^{b})	C_6F_5Co(salen)	98^{c}
	p-HC_6F_4	MePh	120		
		MePh/py	25	*p*-HC_6F_4Co(salen)py	21
	o-HC_6F_4	MePh/py	240	*o*-HC_6F_4Co(salen)py	Trace^{d}
Co(salophen)	C_6F_5	MePh	120		
		MePh/py	60	C_6F_5Co(salophen)py	^{e}
	o-HC_6F_4	MePh/py	240		

^{a}At b.p. in each case. ^{b}Extensive precipitation of thallous bromide [see reaction 3] and a colour change (brown → green) were observed in this time. ^{c}As the pyridinates, after crystallization from py/MeOH/H_2O. ^{d}Detected by TLC. ^{e}Not obtained pure (see text).

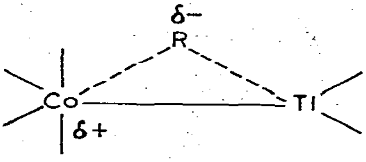
(salen)H_2O [10,11], whilst the fluorine chemical shifts show no unexpected features when compared with data for organometallics with the same fluorocarbon groups [12-14]. The separations between the CH_3—C—O and CH_3—C=N chemical shifts for the present RCo(acacen)py derivatives in perdeuterodimethyl sulphoxide ($\approx$0.2 ppm) are larger than those for (Me or Ph)Co(acacen)py in $CDCl_3$ ($\approx$0.03 ppm) [10], but this is largely due to the solvent difference. Similar variation in the separation with change of solvent has been observed for the free ligand [15]. Of the present compounds, only o-HC_6F_4Co(acacen)py is sufficiently soluble for examination in both $(CD_3)_2SO$ and $CDCl_3$, and a smaller separation was obtained in the latter. Despite the complexity of the infrared spectra of the RCo(acacen or salen)py complexes, comparison with the spectra of Co(acacen or salen) and suitable pyridine complexes [16] enabled bands attributable to the polyfluorophenyl groups to be clearly distinguished for each compound (see Experimental section). In the case of impure C_6F_5Co(salophen)py, ν(CF) frequencies [17] at 1100-950 cm^{-1} and fluorine chemical shifts are almost identical with those of C_6F_5Co(acacen or salen)py.

Qualitative reactivity orders for the organothallium compounds and cobalt complexes in reaction 3, viz. (*a*), $Ph_2TlBr \ll (o\text{-}HC_6F_4)_2TlBr < (p\text{-}HC_6F_4)_2TlBr < (C_6F_5)_2TlBr$ and (*b*), Co(salophen) < Co(salen) < Co(acacen), can be inferred from the data of Table 1. The dependence of eqn. 3 on a high degree of fluorine substitution [sequence *a*] is indicative of a polar mechanism and a transition state in which the polyfluorophenyl groups have considerable carbanionic character. Similar substituent effects have been observed for the hydrogen exchange, via polyfluorophenyl carbanions, of polyfluorobenzenes with methoxide ions in methanol [18]. A free radical mechanism, proposed [19] for oxidation of some cobalt(II) Schiff base complexes with organic halides, would not account for the requirement of multiple fluorine substituents. Moreover, competition from scavenging of polyfluorophenyl radicals by toluene (see e.g. the ready reaction of benzene with $C_6F_5^{\bullet}$ [20]) would be expected, whereas essentially quantitative transfer of C_6F_5 groups from thallium to cobalt was observed on reaction of Co(salen) with $(C_6F_5)_2TlBr$ (Table 1). Sequence *b* cannot be correlated with the oxidation potentials of the cobalt(II) complexes, since the values for Co(salen) and Co(salophen) are approximately equal [21] by contrast with their differing behaviour in reaction 3. However, the order can be correlated with increasing flexibility of the quadridentate ligands, suggesting a mechanism in which twisting of these ligands occurs.

There are two mechanisms which appear to account for the general features of the reactions, viz. (*i*) formation of a thallium—bromine—cobalt bridge, followed by electron transfer from cobalt to thallium via bromine, and then transfer of the polyfluorophenyl groups from thallium (now formally Tl^{II}) to cobalt (now Co^{III}), or (*ii*) nucleophilic attack of cobalt on thallium (cobalt—thallium bonds are known, e.g. in $Tl[Co(CO)_4]_3$ [22]), followed by electron and fluorocarbon group transfer. Transition states (A and B), in which the fluorocarbon groups have considerable carbanionic character and the quadridentate ligands are twisted from their normal planar configurations, can be envisaged for these mechanisms. Related mechanisms, involving thallium—bromine—metal bridging [6] or thallium—metal bonding [4,5], have been proposed for reaction 2, and there is no evidence available to decide between them. Operation of either of



(A)



(B)

the foregoing processes gives a thallium(II) species, which either can disproportionate [23] (eqn. 4a),

$$2\ RTlBr \rightarrow R_2TlBr + TlBr\downarrow \tag{4a}$$

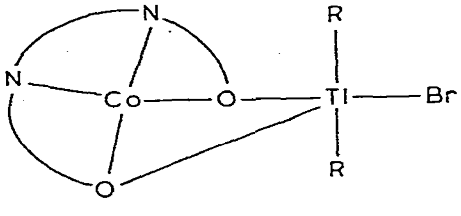
$$CoL + RTlBr \rightarrow RCoL + TlBr\downarrow \tag{4b}$$

or react with further cobalt(II) complex (eqn. 4b).

Enhanced reactivity on addition of pyridine (Table 1) arises because CoL(py) complexes are more readily oxidised than the corresponding CoL derivatives [21] and the polar solvent presumably would stabilise the polar transition states A and B.

Neither mechanism is entirely satisfactory. Firstly formation of a cobalt—halogen bond in mechanism (*i*) might be expected to lead to significant yields of the corresponding cobalt(III) Schiff base halide complexes [19], but this was not observed [see in particular, the quantitative formation of C_6F_5Co(salen) (Table 1)]. Possibly the insolubility of thallous bromide dictates the outcome of the reaction. In the case of the second mechanism, nucleophilic attack by a square planar cobalt(II) complex is unusual*, but has recently been proposed in the group exchange between organocobalt(III) and cobalt(II) Schiff base complexes [25].

A further mechanistic possibility, viz. that electron and group exchange follow chelation of the cobalt(II) complex through oxygen to thallium (C), is attractive, because complexes of this type are well-known [26-28], e.g. Co(salen)-$InCl_3$ [28]. However, twisting of the quadridentate ligands, suggested by sequence *b*, would not occur.



(C)

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* Possibly because the sterically most accessible orbital d_{z^2} does not contain a lone pair in the ground state configuration [24].

As a preparative route to polyfluorophenylcobalt(III) Schiff base complexes, reaction 3 is simple and convenient, but appears restricted to derivatives of flexible quadridentate ligands. Syntheses of the organothallium reactants are straightforward [1,29], and these compounds can be stored for prolonged periods without decomposition.

Experimental

General

Cobalt(II) Schiff base complexes [30,31], bromobis(polyfluorophenyl)-thallium(III) complexes [1,29] and diphenylthallium(III) bromide [32] were obtained by established methods. Toluene was dried with sodium wire. Pyridine was purified by refluxing over potassium hydroxide and distillation under nitrogen, and was stored over molecular sieves.

NMR spectra were obtained with Varian A56/60 and Bruker WH90 instruments. Proton resonances are in ppm downfield from internal tetramethylsilane, and were obtained using $(CD_3)_2SO$ as solvent. Fluorine resonances are in ppm upfield from internal $CFCl_3$, and the solvents are given below. Infrared spectra (2000-650 cm^{-1}) of complexes as Nujol and hexachlorobutadiene mulls were recorded with a Perkin—Elmer 521 spectrophotometer. Frequencies clearly attributable to the fluorocarbon groups are given in italics. Mass spectra were obtained with a Hitachi—Perkin—Elmer RMU-6E instrument. Only intense peaks at or above *m/e* 150 are listed. Most organocobalt complexes were insufficiently volatile to give mass spectra.

Preparations of organocobalt(III) Schiff base complexes

N,N'-Ethylenebis(acetylacetoniminato)(pentafluorophenyl)pyridinecobalt-(III) (nc). Hot degassed solutions of *N,N'*-ethylenebis(acetylacetoniminato)cobalt-(II) (0.20 mmol) and bromobis(pentafluorophenyl)thallium(III) (0.10 mmol) in toluene (20 ml) were mixed and heated under reflux in an atmosphere of nitrogen for 15 min. The reaction mixture was filtered through cellulose powder, the filtrate was evaporated to dryness, and the resulting green powder was recrystallized from pyridine/methanol/water giving gold-brown crystals of the required compound, m.p. 193-194°C. (Found: C, 52.2; H, 4.2; F, 17.7; N, 7.6. $C_{23}H_{23}CoF_5N_3O_2$ calcd.: C, 52.4; H, 4.4; F, 18.0; N, 8.0%.) Infrared absorption: 1620s, 1596vs(br), 1497vs(br), 1463m(sh), 1433vs(br), 1415s(sh), 1347w, 1337w, 1276s, 1226s, 1202m, *1062vs*, 1038m, 1020m, 1010m, *970vs*, 958s, 948w, 758vs, 749vs, 720m and 694vs cm^{-1}. PMR spectrum: 1.77 [s, 6H, $CH_3C{=}N$], 1.97 [s, 6H, $CH_3C{-}O$], 3.35 [br, 4H, CH_2], 4.65 [s, 2H, CH], and 7.28-8.44 ppm [br, 5H, aromatic]. ^{19}F NMR spectrum [in C_5D_5N]: 123.0 [m, 2F, F2, 6], 162.8 [m, 1F, F4], and 166.0 ppm [m, 2F, F3, 5].

The following three compounds were prepared in a similar way.

N,N'-Ethylenebis(acetylacetoniminato)pyridine(2,3,5,6-tetrafluorophenyl)-cobalt(III) (nc). Gold-brown crystals, m.p. 245-247°C. (Found (after 1 h at 90°C under vacuum): C, 50.0; H, 4.3; F, 17.6; N, 6.5. $C_{18}H_{19}CoF_4N_2O_2$ (pyridine free complex) calcd.: C, 50.2; H, 4.4; F, 17.7; N, 6.5%.) Satisfactory analyses could not be obtained for the pyridinate. Infrared absorption: 1619s, 1596vs(br), 1501vs(br), 1443s, 1415s, 1351s, 1340m, 1281vs, 1222s, *1216s*, *1196vs*, *1166vs*, 1120m, 1090w, 1071s, 1041s, 1010vs, 960w, 948s, *899vs*, *841s*, 796s, 748vs and 694vs cm^{-1}. PMR spectrum: 1.78 [s, 6H, $CH_3C{=}N$], 1.93 [s, 6H, $CH_3C{-}O$], 3.35 [br, 4H, CH_2], 4.65 [s, 2H, CH], and 7.25-8.46 ppm [br, 6H, aromatic]. ^{19}F NMR spectrum [in C_5D_5N]: 124.1 [m, 2F, F2, 6] and 142.7 ppm [m, 2F, F3, 5]. Mass spectrum: *m/e* 430 [$C_6HF_4Co(acacen)^+$], 298 [$(C_6HF_4)_2{}^+$], 281 [$Co(acacen)^+$], 248 [$C_{11}H_2F_6{}^+$], 170 [$C_6H_9CoON^+$] and 150 [$C_6H_2F_4{}^+$].

N,N'-Ethylenebis(acetylacetoniminato)pyridine(2,3,4,5-tetrafluorophenyl)-cobalt(III) (nc). Pink crystals, m.p. 173-175°C. (Found (after 3 h at 100°C under vacuum): C, 50.0; H, 4.4; F, 17.2; N, 6.5%.) Satisfactory analyses could not be obtained for the pyridinate. Infrared absorption: 1612s, 1590vs(br), 1501vs(br), *1460s*, 1430s, 1360m, *1324s*, 1281s, 1231m, *1211vs*, 1180m, 1153w, 1130m, *1090vs*, *1061vs*, 1040m, 1011m, *987s*, 957m, 890w, *861s*, 806w, 772vs, 760vs, and 698s cm^{-1}. PMR spectrum: 1.78 [s, 6H, $CH_3C{=}N$], 1.96 [s, 6H, $CH_3C{-}O$], 3.34 [br, 4H, CH_2], 4.65 [s, 2H, CH], and 7.27-8.45 ppm [br, 6H, aromatic]. In $CDCl_3$, the first four resonances were at 1.90, 2.00, 3.28, and 4.66 ppm respectively. ^{19}F NMR spectrum: 129.0 [m, 1F, F2], 144.5 [m, 1F, F5], 161.1 and 165.1 ppm [each m, total integration 2F, F3, 4]. Mass spectrum: as for the previous compound, except for intensity differences and addition of *m/e* 159. When a solution of the compound in methanol/water/pyridine was allowed to stand exposed to air for 2 days, crystals of 2,2',3,3',4,4',5,5'-octafluorobiphenyl were deposited, m.p. 80-81°C, lit. [33], m.p. 79-80°C (Found: mol. wt. (mass spectrometry), 298. $C_{12}H_2F_8$ calcd.: mol. wt. 298.)

N,N'-Ethylenebis(salicylaldiminato)(pentafluorophenyl)pyridinecobalt(III). Gold-brown crystals, m.p. > 250°C. (Found: C, 56.4; H, 3.4; F, 16.0; N, 7.4. $C_{27}H_{19}CoF_5N_3O_2$ calcd.: C, 56.75; H, 3.35; F, 16.6; N, 7.35%.) Infrared absorption: 1626vs, 1595vs, 1532s, *1501vs*, 1463m(sh), 1433vs(br), 1415s(sh), 1345s, 1308s, 1242w, 1217m, 1201s, 1148s, 1129s, *1061vs*, 1040m, 1030m, 1015m, *967vs*, 905s, 854m, 796m, 761vs, 746s, and 694s cm^{-1} (in agreement with, but more extensive than the reported spectrum [9]). PMR spectrum: 3.74 [br, 4H, CH_2], 8.02 [s, 2H, $>CH{=}N$], and 6.33-8.57 ppm [br, 13H, aromatic]. ^{19}F NMR spectrum [in $(CD_3)_2SO$]: 122.1 [m, 2F, F2, 6], 162.0 [m, 1F, F4], and 165.9 ppm [m, 2F, F3, 5].

N,N'-Ethylenebis(salicylaldiminato)pyridine(2,3,5,6-tetrafluorophenyl)-cobalt(III) (nc). Hot degassed solutions of bromobis(2,3,5,6-tetrafluorophenyl)-thallium(III) (0.10 mmol) in toluene (20 ml) and *N,N'*-ethylenebis(salicylaldiminato)cobalt(II) (0.20 mmol) in pyridine (20 ml) were mixed and heated under reflux in an atmosphere of nitrogen for 1 h. The reaction mixture was filtered through cellulose powder, and the solvent was evaporated under vacuum yielding an orange powder, which was recrystallized from pyridine/methanol/water giving yellow crystals of the required compound, m.p. > 250°C. (Found: C, 58.5; H, 3.6; F, 13.8; N, 7.3. $C_{27}H_{20}CoF_4N_3O_2$ calcd.: C, 58.6; H, 3.6; F, 13.7; N, 7.6%.) Infrared absorption: 1626vs, 1600vs, 1532s, 1450vs(br), 1390w, 1344s, 1312s, 1215m, 1195s, *1187s*, *1156s*, 1145s, 1125s, 1061m, 1038m, 960m, 900s, 850m, 827m, 798m, 760vs, 745s, *708vs*, and 698s cm^{-1}. PMR spectrum: 3.74 [br, 4H, CH_2], 8.00 [s, 2H, $>CH{=}N$], and 6.24-8.65 ppm [br, 14H, aromatic]. ^{19}F NMR spectrum [in $(CD_3)_2SO$]: 123.5 [m, 2F, F2, 6] and 142.9 ppm [m, 2F, F3, 5].

Pentafluorophenyl[N,N'-o-phenylenebis(salicylaldiminato)]pyridinecobalt-(III) (nc). The complex was obtained impure from a preparation similar to that of the preceding compound. Infrared absorption (Nujol mull only): 1608vs, 1578s, 1520s, 1502m, 1458s, 1329m, 1248w, 1202w, 1190s, 1148s, 1129m, *1065s*, 1025w, 1010w, *961vs*, 923m, 840w, 801w, 750s, 745s, and 693s cm^{-1}. ^{19}F NMR spectrum [in $(CD_3)_2SO$]: 123.4 [m, 2F, F2, 6], 162.2 [m, 1F, F4], and 166.0 ppm [m, 2F, F3, 5].

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