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Oligonuclear cyanide complexes from silver containing building blocks

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Dedicated to Professor Sheldon Shore on the occasion of his 70th birthday.

Abstract

The complexes $L_nM–CN–Ag–CN$ with L_nM=Cp(dppe)Fe or $Cp(PPh_3)_2Ru$ are available from $[Ag(CN)_2]^-$ and $[L_nM]^+$ or $L_nM–CN$. They are precursors of $[L_nM–CN–Ag–NC–ML_n]^+$. Likewise the trinuclear complexes $[L_nM'–CN–Ag–NC–M'L_n]^+$ with L_nM'=(TPA)Cu and *cis*-$(bpy)_2FeCN$ have been prepared. Irrespective of the CN attachment in the starting materials the cyanide-bridged compounds always contain M–CN–Ag arrays, i.e. silver–isocyanide coordination, thereby proving the lability of the silver–cyanide linkage. Electrochemical measurements have shown that there is no electronic communication between the outer metal centers in the trinuclear complexes. © 2000 Elsevier Science B.V. All rights reserved.

Keywords: Trinuclear complexes; Cyanide bridging; Electrochemistry; Metal–metal interactions; Structure

1. Introduction

In the previous papers of this series [1–6] and in a recent review [7] we have outlined our interest in oligonuclear metal complexes containing chain-like arrays of metals linked by bridging cyanide ligands. It is not so much the materials properties of these species that attracts us, but their basic foundations: single electron redox steps, long-range electronic interactions, mixed valence, and metal–metal charge transfer, which are mediated by the cyanide bridges. According to this the methods of investigation are electrochemistry, UV–vis/NIR spectroscopy, and structure determinations.

Our synthetic approach has allowed us to vary the nature and oxidation state of the metallic building blocks, the orientation of the bridging ligand (CN versus NC), and the geometry at the interconnected metal units $M(\mu\text{-}CN)_2$ (square–planar, tetrahedral, octahedral, *cis*, *trans*). It was found that long-range metal–metal interactions occur only in *trans*-configured (i.e. linearly arranged) octahedral and square–planar and also in tetrahedral complexes, but not in *cis*-configured complexes (i.e. 'around the corner'). As a rule the bridging cyanides were inert, i.e. did not undergo cyanide/isocyanide isomerisation, in the organometallic or heavy metal-containing compounds.

We have now extended the studies of trinuclear $M(\mu\text{-}CN)M'(\mu\text{-}CN)M$ complexes to those containing naked metal ions M′ (Cu, Ag, Au, Hg) in the center. All these ions prefer linear twofold coordination and should hence be qualified for the transmission of electronic interactions between the outer metallic constituents. This paper deals with di- and trinuclear systems containing silver ions in the center. Our work extends and complements previous work by Connelly et al. [8] on Cu, Ag, and Au centered trinuclear complexes, and there are a few reports on polymeric cyanometal complexes containing AgCN building blocks [9,10].

2. Preparations and structures

2.1. Dinuclear complexes

In order to prepare dinuclear complexes with terminal cyanide ligands as building blocks for higher nuclearity species, silver salts or $[Ag(CN)_2]^-$ were reacted with cyanometal complexes or with organometallic

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Lewis acids. This way the two complexes **1** and **2** were obtained. **1** resulted both from Cp(dppe)Fe-CN and $[Ag(CN)_2]^-$ and from $[Cp(dppe)Fe(MeCN)]^+$ and $[Ag(CN)_2]^-$. Likewise, **2** was obtained from $[Ag(CN)_2]^-$ with both $Cp(PPh_3)_2Ru$–CN and $Cp(PPh_3)_2Ru$–Cl. These reactions had been intended to produce the two possible isomers of the dinuclear complexes, i.e. with M–CN–Ag–CN and M–NC–Ag–CN arrays. The products of the two Fe/Ag reactions originally seemed to verify this intention, appearing as yellow and red crystals of different habit. Spectroscopy, electrochemistry and structure determinations showed, however, that the presumed isomers of **1** are identical, their different colors resulting only from their different crystal systems. Thus it must be concluded that after attachment of an organometallic unit to a Ag–CN unit the cyanide bridge is reoriented from the Ag–CN–M array to the Ag–NC–M array. This conforms to our experience that in purely organometallic species the M–CN–M′ arrays are inert toward cyanide reorientation [1,2] but that Cp(dppe)Fe–NC–M′ arrays with M′ units from classical coordination chemistry may be prone to cyanide rearrangement [11]. The preferred arrangement, i.e. M–CN–Ag in **1** and **2**, is easy to understand: it contains the π-acceptor terminus of CN^- bound to the electron-rich organometallic unit and the σ-donor terminus bound to the pure σ-acceptor Ag^+.

Cp(dppe)Fe–CN–Ag–CN
1
$Cp(PPh_3)_2Ru$–CN–Ag–CN
2

1 forms yellow crystals of space group $P2_1/c$ and red crystals of space group $P2_1/n$. In the two monoclinic cells the orientation of the molecules is quite different, but the molecular shapes are virtually identical. The structure determination with the better *R* value is presented here, see Fig. 1. The main feature identifying the orientation of the bridging cyanide ligand is the distinctly nonlinear Ag–N–C array with a bending angle

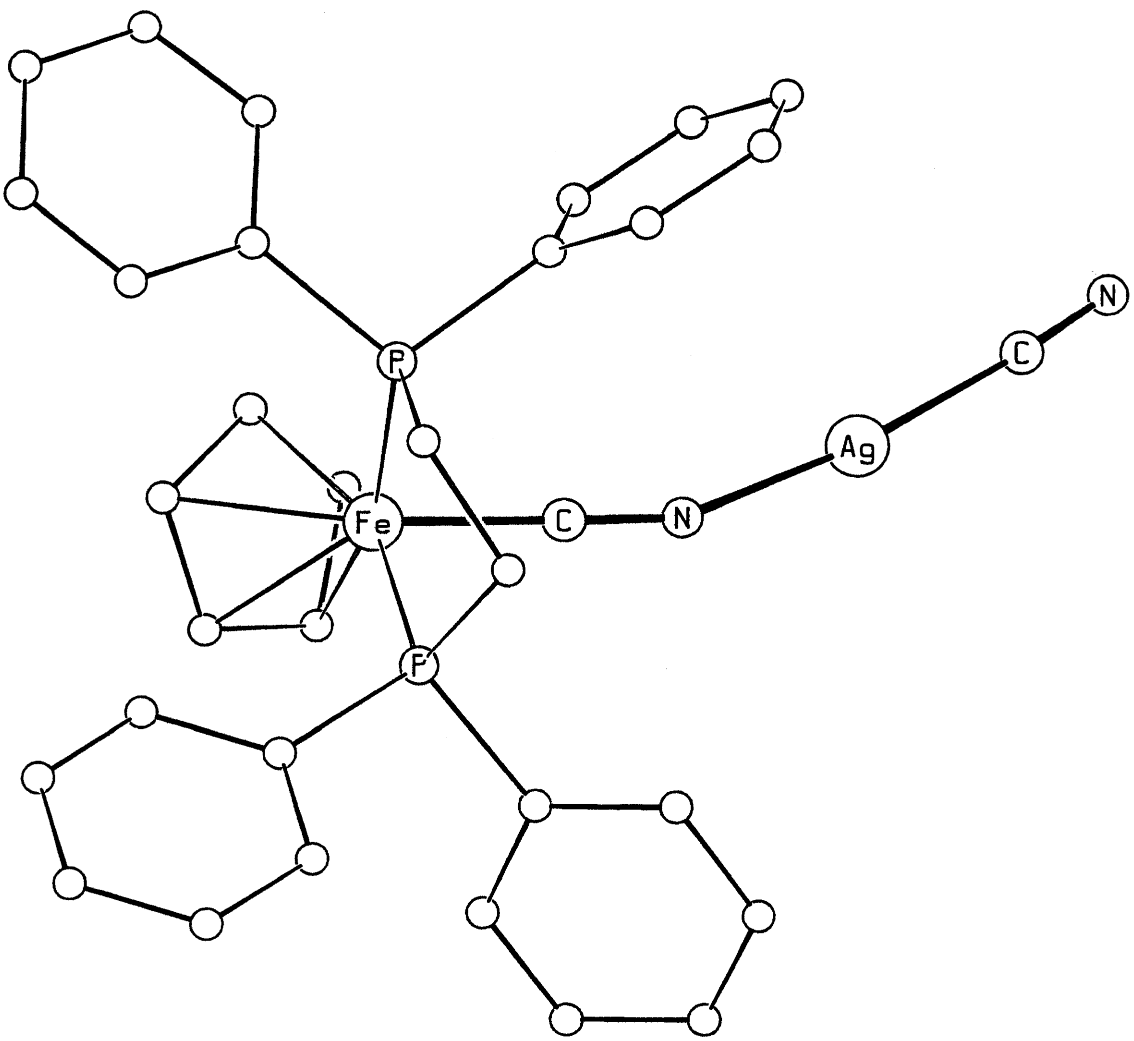

Fig. 1. Molecular structure of complex **1**. Important bond lengths (Å) and angles (°). Fe–C 1.860(3), C–N(bridge) 1.156(4), N–Ag 2.055(3), Ag–C 2.035(3), C–N(terminal) 1.117(4), Fe–C–N 178.0(2), C–N–Ag 150.6(2), N–Ag–C 176.2(1), Ag–C–N 176.7(3).

of 150°. While terminal cyanide is almost always linear, cf. the terminal CN in **1**, bridging cyanide shows the varying degree of backdonation at its C and N termini by a varying degree of bending which, as a rule, is much more pronounced at nitrogen than at carbon [1,4,5,7]. The bending in **1** is exceptionally large, being unprecedented in many structures with Cp(dppe)Fe–CN building blocks [1–6,11]. It leaves no doubt about the orientation of the bridging cyanide. The other structural features of **1** are normal, i.e. the geometrical details of the Cp(dppe)Fe–CN [1–6] or Ag–CN [9,10] fragments. The coordination of silver is close to linear (176°). The Ag–C and Ag–N bond lengths are remarkably similar, but within the usual range. The terminal CN is also close to ideally linear (177°).

2.2. Trinuclear complexes

Just like for the dinuclear complexes **1** and **2**, it was attempted to obtain trinuclear complexes with two different orientations of the cyanide bridges. For this purpose either silver salts were reacted with cyanometal complexes or $[Ag(CN)_2]^-$ was treated with metal complex species able to be attached at the CN nitrogen. Again it was found that irrespective of the reaction procedure only one product was obtained, and this product invariably had both cyanide bridges attached to silver with their nitrogen termini.

Cp(dppe)Fe units were again introduced as Cp(dppe)Fe–CN (with $AgNO_3$ or $Ag(BF_4)_2$) or [Cp(dppe)Fe(MeCN)]Br (with $KAg(CN)_2$), $Cp(PPh_3)$-Ru units were offered as $Cp(PPh_3)_2Ru–CN$ or $Cp(PPh_3)_2RuCl$. The resulting cationic complexes **3** and **4** were isolated as SbF_6 or BF_4 salts.

$[Cp(dppe)Fe–CN–Ag–NC–Fe(dppe)Cp]^+$

3

$[Cp(PPh_3)_2Ru–CN–Ag–NC–Ru(PPh_3)_2Cp]^+$

4

With the hope that different metals or different ligand environments would render the trinuclear complexes more inert toward CN rearrangement, the Ag/Cu and Ag/Fe combinations represented by **5** and **6** were realized. Yet the different preparations of **5** again yielded only one product. $[(TPA)Cu(MeCN)]^{2+}$ and $[(TPA)Cu–CN]^+$ were brought in as (TPA)Cu units (TPA = tris (2–pyridylmethyl)amine). Their reactions with $[Ag(CN)_2]^-$ and $AgBF_4$, respectively, both yielded **5** which was isolated as the perchlorate. Complex **6** was obtained from *cis*-$(bpy)_2Fe(CN)_2$ and $AgBF_4$ as the BF_4 salt. One reason for preparing **6** was also the intention to use it as a building block for higher-nuclearity complexes with cyanide bridges.

$[(TPA)Cu–CN–Ag–NC–Cu(TPA)]^{3+}$

5

$[CN(bpy)Fe–CN–Ag–NC–Fe(bpy)CN]^+$

6

In those cases where an inert cyanometal reagent was attached to a silver salt, e.g. when preparing **3** and **4** from $AgBF_4$, there could be little doubt about the orientation of the resulting cyanide bridge. In case of the reagents (TPA)Cu–CN or $(bpy)_2Fe(CN)_2$ the possibility of cyanide reorientation after formation of the trinuclear complexes cannot be completely ruled out. For **5** its blue color which corresponds to that of $[(TPA)Cu(MeCN)]^{2+}$ and not to that of $[(TPA)Cu–CN]^+$ (blue–green) seems to indicate a Cu–NC–Ag arrangement, as do the electrochemical data (see below). Attempts to resolve this ambiguity by a structure determination were not successful.

2.3. Attempts at higher nuclearity complexes

Complexes **1**, **2** and **6** contain terminal cyanide ligands and thus offer themselves for the construction of higher nuclearity systems. Attempts to use them for this purpose, however, were not met with success yet. They either resulted in untractable mixtures, e.g. when treating two equivalents of **6** with one equivalent of $FeCl_3$, or they led to redistribution reactions producing simple and known complexes.

Treating **1** with $[Cp(dppe)Fe(MeCN)]^+$ produced the dinuclear complex **7** [1]. Likewise treatment of **1** with $Cp(PPh_3)_2RuCl$ resulted in **8** [1]. And attempts to produce a pentanuclear complex from [1] and $AgBF_4$ yielded trinuclear [3] instead. All these results underline the lability of the silver–cyanide combination, and each corresponds to the elimination of AgCN from the reagent [1]. This lability must be high: it has prevented even the formation of the stable trinuclear complex **3** from the reaction between **1** and $[Cp(dppe)Fe(MeCN)]^+$.

$[Cp(dppe)Fe–CN–M']^+$

7: $M' = Fe(dppe)Cp$ **8**: $M' = Ru(PPh_3)_2Cp$

3. Spectra and redox properties

The diamagnetic complexes described here show their constituents in their NMR spectra (see Section 4) which, however, yield no information concerning the orientation of the cyanide bridges. In contrast, the latter can be extracted with some confidence from both the ν(CN) bands in the IR spectra and the redox potentials as obtained as $E_{1/2}$ values from the cyclic voltammograms, see Table 1.

The interplay of σ-donation, π-acceptance and kinematic effects for the ν(CN) data of cyanide-bridged complexes containing Fe(dppe)Cp and $Ru(PPh_3)_2Cp$ units has been discussed in detail by us [1,4–6]. When the metal attached at the nitrogen of Cp(dppe)Fe–CN or $Cp(PPh_3)_2Ru–CN$ is not a very strong σ-acceptor,

Table 1
IR and CV data

Complex	ν(CN) [a]	$E_{1/2}$ [b]
Cp(dppe)Fe–CN	2063	0.48
$Cp(PPh_3)_2Ru–CN$	2072	0.79
$[(TPA)Cu–CN]ClO_4$	2143	−0.33 [c]
cis-$(bpy)_2Fe(CN)_2$	2079, 2069	0.46
$K[Ag(CN)_2]$	2140	
1	2134, 2060	0.55
2	2131, 2050	1.02
3	2072	0.72
4	2077	1.05
5	2179	−0.34 [c]
6	2110, 2077	0.61, 0.80

[a] In KBr, cm^{-1}.

[b] In CH_2Cl_2, V versus Ag/AgCl, scan rate 100 mV s^{-1}.

[c] In CH_3CN.

the shifts of ν(CN) are small and normally toward higher wavenumbers. If one applies this to complexes **1**–**4** one finds one IR band each in the vicinity of that of Cp(dppe)Fe–CN or $Cp(PPh_3)_2Ru–CN$, respectively. In contrast, $[Ag(CN)_2]^-$ shows its ν(CN) band about 100 cm^{-1} higher, i.e. in a well-separated region, and both complexes **1** and **2** have their second ν(CN) band close to this. It is therefore easy to conclude that **1** and **2** contain one Ag–CN and one Ag–NC linkage each, and that **3** and **4** contain only Ag–NC linkages. The conclusions are less unambiguous for **5** and **6**, the reason being that $[(TPA)Cu–CN]^+$ and $(bpy)Fe(CN)_2$ have ν(CN) bands close to that of $[Ag(CN)_2]^-$ in the complexes. However, considering the rule that upon bridging a slight increase in the band position occurs one finds that this can be stated in both cases (2143 → 2179 cm^{-1} for **5**, 2079 → 2110 cm^{-1} for **6**. Hence there is a consistent set of structural assignments and IR effects for all complexes.

The conclusions to be drawn from the redox potentials are the same. When Cp(dppe)Fe or $Cp(PPh_3)_2Ru$ are attached to another metal in the Fe–CN–M′ or Ru–CN–M′ array their redox potential is raised in comparison to that of Cp(dppe)Fe–CN or $Cp(PPh_3)_2Ru–CN$, while in a Fe–NC–M′ or Ru–NC–M′ array it is lowered [1,5]. In complexes **1**–**4** it is raised by the typical amounts. We have little experience with (TPA)Cu(II) or $(bpy)_2Fe(CN)$ in this respect, but in complex **6** the increase of the redox potential as referred to the reference compound $(bpy)_2Fe(CN)_2$ has the same order of magnitude as that in complexes **1**–**4**. In contrast, for complex **5** the redox potential does not differ from that of $[(TPA)Cu–CN]^+$ again leaving the possibility that **5** contains a Cu–NC–Ag arrangement. Thus for **5** the conclusions from the color (see above) and the electrochemical data are in disagreement with those from the IR data. Except for this case all data for the di- and trinuclear complexes conform to the presence of M–CN–Ag arrays.

One major expectation, which initiated the work described here, was not fulfilled. With the exception of **6** none of the trinuclear complexes has yielded two separate redox waves in the cyclic voltammograms. Thus there is no indication of electronic communication between the external metal units by electrochemical means, and it was not possible to prepare mixed-valent species. The same observation was made by Connelly et al. for related trinuclear complexes with central $Ag(NC)_2$ units [8]. This means that a linear array is not a sufficient condition for electronic communication across two cyanide bridges, but that additional properties of the central connecting unit are important which have yet to be qualified. One of these seems to be the position of the central metal in the periodic table, as Connelly has found communication across $Au(NC)_2$ [8], and we have found it across linear $L_2Pt(NC)_2$ [5]. We plan to address this question by comparative studies of systems with Ni versus Pt, Cu versus Au, and Zn versus Hg as central metals.

4. Experimental

The general experimental and measuring techniques are given in Ref. [5]. The complexes Cp(dppe)Fe–CN [12], [Cp(dppe)Fe(MeCN)]Br [13], $Cp(PPh_3)_2Ru–CN$ [12], $Cp(PPh_3)_2RuCl$ [14], $[(TPA)Cu–CN]ClO_4$ [15], $[(TPA)Cu(MeCN)](ClO_4)_2$ [16] and cis–$(bpy)_2Fe(CN)_2$ [17] were prepared as described.

4.1. Complex **1(a)**

Cp(dppe)Fe–CN (0.545 g, 1.00 mmol) in 20 ml of methanol was treated with $KAg[CN]_2$ (0.20 g, 1.00 mmol) in 5 ml of methanol and then with 1 ml of acetic acid with stirring. After 24 h of stirring the yellow precipitate was filtered off and recrystallized from dichloromethane by layering with petroleum ether. Yield 0.32 g (47%) of **1** as yellow crystals, m.p. 210°C (dec.).

4.2. Complex **1** **(b)**

[Cp(dppe)Fe(MeCN)]Br (0.640 g, 1.00 mmol) in 20 ml of methanol was treated with $K[Ag(CN)_2]$ (0.20 g, 1.00 mmol) in 5 ml of methanol with stirring. Workup as before after 24 h of stirring yielded 0.35 g (52%) of **1** as red crystals which turn yellow at 140°C and melt at 210°C with decomposition. ^1H-NMR ($CDCl_3$): 7.8–7.1 (*m*, 20H, Ph), 4.26 (*s*, 5H, Cp), 2.5–2.3 (*m*, 4H, dppe). Anal. Found: C, 57.59; H, 4.73; N, 3.69. $C_{33}H_{29}AgFeN_2P_2$ (679.4). Calc.: C, 58.29; H, 4.30; N, 4.12%.

4.3. Complex **2** (a)

$Cp(PPh_3)_2Ru–CN$ (360 mg, 0.50 mmol) suspended in 30 ml of methanol was treated with $K[Ag(CN)_2]$ (100 mg, 0.50 mmol) in 10 ml of methanol and then 1 ml of acetic acid with stirring. Workup as before after 3 d of stirring yielded 200 mg (24%) of **2** as yellow crystals, m.p. 182°C.

4.4. Complex **2** (b)

$Cp(PPh_3)_2RuCl$ (360 mg, 0.50 mmol) in 30 ml of methanol was treated with $K[Ag(CN)_2]$ (100 mg, 0.50 mmol) with stirring. Workup as before after 3 d of stirring yielded 180 mg (21%) of **2** as yellow powder. ^{1}H-NMR ($CDCl_3$): 7.4–7.1 (*m*, 30H, Ph), 4.40 (*s*, 5H, Cp). Anal. Found: C, 60.26; H, 4.12; N, 3.18. $C_{43}H_{35}AgN_2P_2Ru$ (850.0). Calc.: C, 60.71; H, 4.15; N, 3.29%.

4.5. Complex **3** (a)

A solution of 140 mg (0.26 mmol) of Cp(dppe)Fe–CN in 20 ml of methanol was treated with 25 mg (0.13 mmol) of $AgBF_4$ in 5 ml of methanol. After stirring for 12 h the yellow precipitate was collected and recrystallized from methanol at 0°C, yielding 50 mg (30%) of **3** BF_4 as yellow microcrystals, m.p. 145°C.

4.6. Complex **3** (b)

[Cp(dppe)Fe(MeCN)]Br (145 mg, 0.20 mmol) in 20 ml of methanol was treated with $K[Ag(CN)_2]$ (20 mg, 0.10 mmol) in 5 ml of methanol and $NaBF_4$ (20 mg, 0.2 mmol). Workup as before yielded 78 mg (49%) of **3** BF_4 as orange crystals. ^{1}H-NMR ($CDCl_3$): 7.7–7.1 (*m*, 40H, Ph), 4.32 (*s*, 10H, Cp), 2.55–2.35 (*m*, 8H, dppe). Anal. Found: C, 59.74; H, 4.55; N, 2.26. $C_{64}H_{58}AgBF_4Fe_2N_2P_4$ (1285.4). Calc.: C, 59.80; H, 4.55; N, 2.18%.

4.7. Complex **4**(a)

$Cp(PPh_3)_2Ru–CN$ (200 mg, 0.30 mmol) and $AgBF_4$ (28 mg, 0.15 mmol) in 30 ml of methanol was stirred for 1 d. The precipitate was collected and redissolved in 5 ml of dichloromethane. Layering with petroleum ether yielded, after several days, 100 mg (40%) of (**4**) BF_4 as yellow crystals, m.p. 215°C (dec.).

4.8. Complex **4** (b)

$Cp(PPh_3)_2RuCl$ (145 mg, 0.20 mmol) in 15 ml of dichloromethane was treated with $K[Ag(CN)_2]$ (10 mg, 0.05 mmol) in 5 ml of methanol and then $NaBF_4$ (10 mg, 0.1 mmol). After stirring for 12 h workup as before yielded 72 mg (40%) of (**4**) BF_4 as yellow crystals. ^{1}H-NMR ($CDCl_3$): 7.3–6.9 (*m*, 60H, Ph), 4.45 (*s*, 10H, Cp). Anal. Found: C, 61,76; H, 4.30; N, 1.67. $C_{84}H_{70}AgBF_4N_2P_4Ru_2$ (1628.2). Calc.: C, 61.97; H, 4.33; N, 1.72%.

4.9. Complex **5**(a)

A solution of $[(TPA)Cu–CN]ClO_4$ (100 mg, 0.21 mmol) in 15 ml of methanol was treated with a solution of $AgBF_4$ (20 mg, 0.10 mmol) in 5 ml of methanol with stirring. The resulting blue precipitate was collected and redissolved in acetonitrile. Layering with diethyl ether yielded, after several days, 60 mg (52%) of (**5**) $(ClO_4)_3$ as blue crystals, m.p. 210°C (dec.).

4.10. Complex **5**(b)

A solution of $K[Ag(CN)_2]$ (100 mg, 0.50 mmol) in 20 ml of methanol was treated with a solution of $[(TPA)Cu(MeCN)](ClO_4)_2$ (596 mg, 1.00 mmol) in 50 ml of methanol. After stirring for 30 min. workup as before yielded 360 mg (61%) of **5** $(ClO_4)_3$. Anal. Found: C, 39.22; H, 3.40; N, 11.08. $C_{38}H_{36}AgCl_3Cu_2N_{10}O_{12}$ (1166.1). Calc.: C, 39.14; H, 3.11; N, 12.01%.

4.11. Complex **6**

$AgBF_4$ (30 mg, 0.15 mmol) and *cis*-$(bpy)_2Fe(CN)_2\cdot 3H_2O$ (146 mg, 0.30 mmol) in 30 ml of methanol were stirred for 1 d. The mixture was evaporated to dryness, the residue picked up in 20 ml of dichloromethane and filtered. After several days, layering with petroleum ether yielded, 40 mg (30%) of **6** BF_4 as dark red crystals, m.p. 140°C. Anal. Found: C, 50.85; H, 3.12; N, 16.48. $C_{44}H_{32}AgBF_4Fe_2N_{12}$ (1035.2). Calc.: C, 51.05; H, 3.12; N, 16.24%.

4.12. Crystal structure determination

Crystals of **1** were obtained directly from the isolated compound. The data set was obtained with an Enraf-Nonius CAD4 diffractometer using Mo–K_α radiation and the $\omega/2\theta$ scan technique at $2\theta = 4–52°$. An absorption correction based on psi scans was applied. The structure was solved by direct methods and refined anisotropically using the SHELX program suite [18]. Hydrogen atoms were included with a common isotropic temperature factor and a fixed C–H distance

Table 2
Crystallographic data for **1**

Crystal size (mm)	$0.7 \times 0.7 \times 0.9$
Space group	$P2_1/c$
a (Å)	8.967(2)
b (Å)	21.008(4)
c (Å)	16.078(3)
α (°)	90
β (°)	104.61(3)
γ (°)	90
Z	4
V (Å^3)	2931(1)
D_{calc} (g cm^{-3})	1.54
μ (mm^{-1})	1.30
Number of reflections collected	5943
Number of independent reflections [$I > 2\sigma(I)$]	5735
Variables	352
R_1 (independent reflections)	0.029
wR_2 (all reflections)	0.084
Residual elemental densities	$+0.5$
Largest difference peak and hole (e Å^{-3})	-0.4

of 0.96 Å. The R values are defined as $R_1 = \Sigma\,|F_o - F_c|/\Sigma\,F_o$ and $wR_2 = [\Sigma\,[w(F_o^2 - F_c^2)^2/\Sigma\,[w(F_o^2)^2]]^{1/2}$. Drawings were produced with SCHAKAL [19]. Table 2 lists the crystallographic details.

5. Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Center, CCDC No. 142235 for compound **1**. Copies of these data may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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