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A Fully Characterized Complex Ion with Unreduced TCNQ as Fourfold Bridging Ligand: $[(\mu_4\text{-TCNQ})\{fac\text{-Re(CO)}_3(\text{bpy})\}_4]^{4+}$**

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Coordination compounds of 7,7,8,8-tetracyano-*p*-quinodimethane (TCNQ) and of the related tetracyanoethene (TCNE)[1] have found wide interest in materials science as potential organic conductors[2] and molecular magnets.[3] As universal π-acceptor components and archetypical "non-innocent" ligands, these TCNX molecules can exist in several readily accessible oxidation states (TCNX^0, paramagnetic TCNX$^{\bullet-}$ or TCNX^{2-}), they may form stacks with mixed valency, they can be coordinated through the carbon π system, or act as σ donors through the nitrile-N atoms and thus bind (bridge) up to four metal centers. All these features have led to a wide variety of unusual geometrical and electronic structures.[1] Although discrete complexes with tetranucleating TCNQ or TCNE ligands have been reported with manganese,[4] ruthenium,[5] osmium,[6] iron,[7] rhenium,[8] and copper,[9] no structural information has been obtained yet for such species. Based on spectroscopic data, some of these systems were described as tetrametalla π systems with extensive metal/ligand electron delocalization.[4–6] On the other hand, there has been a number of structurally characterized coordination polymers involving silver,[10, 11] dimolybdenum,[12] diruthenium,[13] and dirhodium units;[13] however, these extended systems could not be studied as individual entities in solution, for example, by electrochemical methods.

Using the nitrile-binding and potentially π back-donating organometallic complex fragment $[\text{Re(CO)}_3(\text{bpy})]^+$ (bpy = 2,2′-bipyridine),[14] we have now obtained the tetranuclear complex cation $[(\mu_4\text{-TCNQ})\{fac\text{-Re(CO)}_3(\text{bpy})\}_4]^{4+}$ (**1**),[15] which has unprecedented electronic characteristics and which could be structurally studied as the tetrakis(hexafluorophosphate) salt.[16]

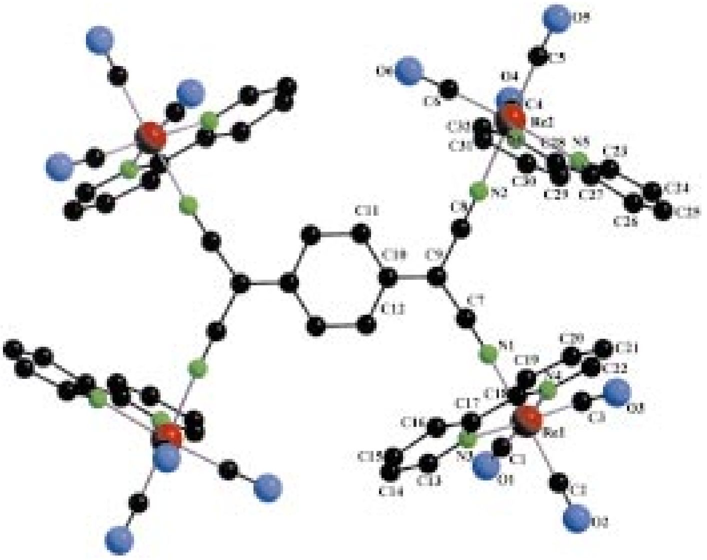

Figure 1. Molecular structure of the tetracation **1** in the crystal of $[(\mu_4\text{-TCNQ})\{fac\text{-Re(CO)}_3(\text{bpy})\}_4](\text{PF}_6)_4$ (**1**-$(\text{PF}_6)_4$). Selected bond lengths [Å] and angles [°]: Re1-N1 2.098(7), Re1-N3 2.175(8), Re1-N4 2.137(10), Re2-N2 2.121(8), Re2-N5 2.216(10), Re2-N6 2.161(10), C7-C9 1.390(14), C8-C9 1.487(13), C9-C10 1.377(14), C10-C11 1.423(14), C11-C12 1.372(16), C10-C12 1.453(15), Re1-Re2 8.18, Re1-Re2A 10.02, Re1-Re1A 12.97, Re2-Re2A 12.89; C9-C7-N1 176.0(10), C9-C8-N2 178.2(11), C7-N1-Re1 170.9(8), C8-N2-Re2 174.2(9).

There are no unusual interionic contacts in the crystal, especially no indications for coordination polymer formation. Figure 1 illustrates that the TCNQ molecule acts as an esentially planar $\mu_4,\eta^1{:}\eta^1{:}\eta^1{:}\eta^1$-bridging ligand towards four cations $[fac\text{-Re(CO)}_3(\text{bpy})]^+$. In comparison to TCNQ, TCNQ$^{\bullet-}$ and the coordination polymers referred to above,[13] the bond parameters of the bridging molecule in the cation **1** point to relatively little metal-to-ligand π-electron delocalization, as illustrated by the bond length of 1.377(14) Å for the exocyclic bond (C9–C10). This bond should lengthen towards a C–C single bond value on significant electron acquisition by TCNQ.[1] The binding of $[fac\text{-Re(CO)}_3(\text{bpy})]^+$ by the nitrile groups of TCNQ occurs in two different conformations, either

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with the bpy coligand inversion-symmetrically situated above or below the C-N-Re axis (at Re1) or with the bpy molecule twisted away from the center of the tetracation (at Re2). While the structural data already suggest a relatively small extent of π back-donation from the organorhenium centers towards highly π-accepting TCNQ, the electrochemical results are even more striking (Table 1).

Table 1. Reduction potentials $E^{[a]}$ of TCNQ and its tetranuclear complexes $[(\mu_4\text{-TCNQ})\{ML_n\}_4]^k$.

$\{ML_n\}$	k	E_{red1}	E_{red2}	K_c[b]	Medium[c]	Ref.
$\{Re(CO)_3(bpy)\}$	4+	+0.45[d]	+0.09[d]	$10^{6.1}$	CH_2Cl_2	this work
$\{Os(P\textit{i}Pr_3)_2(CO)(H)Cl\}$	0	−0.20	−0.94	$10^{12.5}$	CH_2Cl_2	[6b]
– (free TCNQ)	0	−0.29	−0.88	$10^{10.0}$	CH_2Cl_2	[6b]
– (free TCNQ)	0	−0.25	−0.97	$10^{12.2}$	CH_3CN	[5b]
– (free TCNQ)	0	−0.19[e]	−0.78[e]	$10^{10.0}$	DMF	[4]
$\{Ru(NH_3)_5\}$	8+	−0.59	−0.84	$10^{4.2}$	CH_3CN	[5b]
$\{Mn(CO)_2(C_5Me_5)\}$	0	−0.58[e]	−0.80[e,f]	$\approx 10^{3.2}$	DMF	[4]
$\{Fe(dppe)(C_5H_5)\}$[g]	4+	−1.15[e]	−1.30[e]	$10^{2.5}$	CH_2Cl_2	[7]

[a] Potentials in V versus Fc^+/Fc^0. [b] Comproportionation constant for the one-electron-reduced intermediate: $K_c = 10^{(E_{red1}-E_{red2})/59\,mV}$. [c] 0.1M Bu_4NPF_6 as electrolyte. [d] Converted from measurements vs. cobaltocenium/cobaltocene (−1.35 V vs. Fc^+/Fc^0). [e] Converted from measurements vs. SCE (−0.50 V vs. Fc^+/Fc^0). [f] Peak potential for irreversible reduction. [g] dppe = 1,2-bis(diphenylphosphanyl)ethane.

In contrast to the other discrete complexes $[(\mu_4\text{-TCNQ})\{ML_n\}_4]^k$ studied so far,[4–7] the cation **1** is much easier to reduce (by 0.74 V!) than the free TCNQ ligand (Table 1). Such behavior would be expected for metal complexes of conventional π-acceptor ligands such as bipyridines and related molecules.[14, 17] However, the extremely strong π-accepting capacity of TCNQ usually results in a situation with negative shifts of the reduction potentials (Table 1) where metal-to-ligand electron transfer through π back-donation overcompensates the effect from the σ polarization exerted by the Lewis acidic metal centers, leading to an anionic TCNQ ligand.[1, 4–7] In other words, the "normal" effect[17] of a large positive shift of the ligand reduction potential upon metal coordination would be the exception for ligands such as TCNQ[4, 18] and it has been observed here for the first time.

Table 1 illustrates the variability of the comproportionation constant K_c of the paramagnetic intermediate in the typical two-step reduction sequence for TCNQ and its tetranuclear complexes. In this respect, the tetrarhenium compound adopts an intermediate position between strongly metal–ligand π-coupled systems (small K_c) and free TCNQ (large K_c). Oxidation of the complex cation occurs irreversibly at a very high potential of +1.45 V versus ferrocenium/ferrocene (Fc^+/Fc^0).

Vibrational spectroscopic data are in agreement with the interpretation of the structural and the electrochemical results: At 2241 cm^{-1} in nujol, the single nitrile stretching band of **1**-$(PF_6)_4$ is shifted to *higher* energies than in free TCNQ (2228 cm^{-1}), in contrast to what is usually observed.[1, 4–9] The δ(CH) bending mode of coordinated TCNQ is found at 840 cm^{-1}, which has usually been interpreted as an indication of "partial reduction" of the TCNQ ligand.[13]

The absorption spectrum of **1**-$(PF_6)_4$ in CH_2Cl_2 shows an intense metal-to-ligand charge transfer (MLCT) band at 680 nm. This value is distinctly lower than the charge transfer (MLCT or LMCT)[18] maxima observed for tetranuclear TCNQ complexes of manganese (1418 nm in toluene),[4b] ruthenium (935 nm in acetonitrile),[5b] osmium (1170 nm in 1,2-dichloroethane),[6b] or iron (1008 nm in CH_2Cl_2).[7] Even poorly soluble $[(\mu_4\text{-TCNQ})\{Re(CO)_4Cl\}_4]$ has an absorption maximum at 990 nm in toluene.[8] The intra-ligand $\pi \rightarrow \pi^*$ transition is shifted from 395 nm in free TCNQ to 382 nm in **1**.

One-electron reduction to $[(\mu_4\text{-TCNQ})\{\textit{fac}\text{-}Re(CO)_3(bpy)\}_4]^{3+}$ produces new absorption bands at 1020, 905, 425(sh), and 405 nm (Figure 2). The first three bands signify a slightly perturbed $TCNQ^{\bullet-}$ chromophore (free $TCNQ^{\bullet-}$ has 842, 761, and 420 nm[19]); the band at 405 nm is tentatively attributed to a hypsochromically shifted MLCT transition.[5b] The ESR signal of the paramagnetic trication in glassy frozen CH_2Cl_2 at 3.4 K or 110 K exhibits two g components at $g_1 = 2.014$ and $g_{2,3} = 2.006$. This small g anisotropy despite the presence of four rhenium atoms with their large spin–orbit coupling contributions supports the notion of TCNQ-localized spin.[5b, 6b]

This assignment is further supported by results from IR spectroelectrochemistry in dichloromethane.[15] Whereas the metal carbonyl stretching bands move only by about 10 cm^{-1} or less on successive electron acquisition by **1**, the nitrile stretching bands are shifted from 2235 cm^{-1} via 2223 and 2182 cm^{-1} for the trication to 2210 and 2149 cm^{-1} for the doubly reduced species.

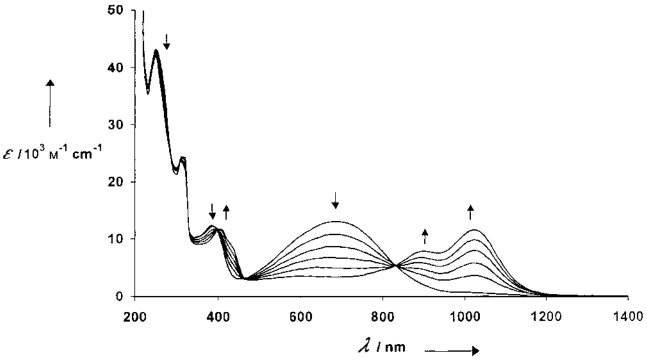

Figure 2. Spectral response of **1**-$(PF_6)_4$ on one-electron reduction in CH_2Cl_2/0.1M Bu_4NPF_6.

In summary, we have not only obtained the first structure analysis of a discrete tetranuclear complex of TCNQ, **1** is also the first such well-characterized TCNQ species which exhibits the "normal" response of metal coordination to a π-acceptor ligand. As a consequence, the fourfold coordination of rhenium(I) causes a shift of +0.74 V for the potential of a clearly TCNQ-based reduction—a remarkable result considering the already very facile reduction of free TCNQ and the capacity of rhenium(I) species for π back-donation. Further study of the stabilized reduced states will be our next objective. Since bis-chelating α-diimines of the bpy type are available[20] and the *fac*-$Re(CO)_3$ group is well suited to act as innocent anchoring corner in molecular rectangles[20] or inorganic clusters,[21] the discrete ion presented here can serve as a starting point for more extended systems.

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[15] A mixture containing $[\text{Re(CO)}_3(\text{bpy})\text{Cl}]$ (300 mg, 0.650 mmol) and AgPF_6 (176 mg, 0.696 mmol) in dichloromethane (20 mL) and methanol (5 mL) was stirred for 18 h at room temperature. After filtration over celite and removal of the solvents the yellow residue was redissolved in CH_2Cl_2 (20 mL) and treated with a solution of TCNQ (33 mg, 0.163 mmol) in CH_2Cl_2 (10 mL). To the blue solution formed after 8 h (IR monitoring) *n*-pentane was added (10 mL). Cooling to $-30\,^\circ\text{C}$ produced a dark blue precipitate which was collected, redissolved in dichloromethane, and reprecipitated with *n*-pentane. Drying under vacuum yielded **1**-$(\text{PF}_6)_4$ (150 mg; 37%). Elemental analysis calcd (%) for $\text{C}_{64}\text{H}_{36}\text{F}_{24}\text{N}_{12}\text{O}_{12}\text{P}_4\text{Re}_4$: C 30.87, H 1.46, N 6.75; found: C 30.65, H 1.46, N 6.40; ^{1}H NMR (CD_2Cl_2): $\delta = 7.32$ (s, 4H; TCNQ-H), 7.71 (dd, 8H; $\text{H}^{5,5'}$(bpy)), 8.26 (dd, 8H; $\text{H}^{4,4'}$(bpy)), 8.43 (d, 8H; $\text{H}^{3,3'}$(bpy)), 8.99 (d, 8H; $\text{H}^{6,6'}$(bpy)); IR (nujol): $\tilde{\nu} = 2241$ (ν(CN)), 2031, 1967, 1950 (ν(CO)), 840 cm^{-1} (δ(CH)); IR spectroelectrochemistry (CH_2Cl_2/0.1 M Bu_4NPF_6): tetracation: $\tilde{\nu} = 2235$ (ν(CN)), 2039, 1956, 1945 cm^{-1} (ν(CO)); trication: $\tilde{\nu} = 2223$, 2182 (ν(CN)), 2038, 1945, 1940sh cm^{-1} (ν(CO)); dication: 2210(br), 2149 (ν(CN)), 2036, 1934(br) cm^{-1} (ν(CO)); UV/Vis (CH_2Cl_2): $\lambda_{\max}$ (ε) = 680 (13100), 382 (12400), 321 (26700) nm ($\text{M}^{-1}\,\text{cm}^{-1}$); UV/Vis spectroelectrochemistry (CH_2Cl_2/0.1 M Bu_4NPF_6): trication: $\lambda_{\max}$ (ε) = 1020 (12100), 905 (9000), 425(sh), 405 (11800), 305 (23600) nm ($\text{M}^{-1}\,\text{cm}^{-1}$).

[16] Single crystals were obtained from a saturated solution in CD_2Cl_2. $\text{C}_{64}\text{H}_{36}\text{F}_{24}\text{N}_{12}\text{O}_{12}\text{P}_4\text{Re}_4$: Dark blue rods, $0.8 \times 0.1 \times 0.05$ mm, monoclinic, space group $P2_1/n$, $a = 10.3137(4)$, $b = 22.0360(5)$, $c = 20.6476(6)$ Å, $\beta = 97.2520(1)^\circ$, $V = 4655.1(2)$ Å^3, $T = 293$ K, $Z = 2$, $\rho_{\text{calcd}} = 1.776\ \text{g}\,\text{cm}^{-3}$, $\mu(\text{Mo}_{\text{K}\alpha}) = 5.356\ \text{cm}^{-1}$, 17485 reflections measured, 6048 observed with $I > 2\sigma(I)$. $R_1 = 0.0603$, $wR_2 = 0.1564$, GOF = 1.026, min/max electron density $-0.938/0.949\ \text{e}\,\text{Å}^{-3}$. Crystallographic data (excluding structure factors) for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-160093. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB21EZ, UK (fax: (+44) 1223-336-033; e-mail: deposit@ccdc.cam.ac.uk).

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Targeting Molecular Recognition: Exploring the Dual Role of Functional Pseudoprolines in the Design of SH3 Ligands[**]

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Protein–protein interactions that are mediated by the binding of proline-rich sequences are involved in a large variety of cellular processes, for example, signal transduction, motility, membrane trafficking, and cell division.[1–5] Such proline-rich ligands adopt a left-handed polyproline II (PPII) helical conformation and bind to a highly conserved patch of aromatic amino acids. These residues are positioned to accommodate the unique geometric properties of a PPII helix on the recognition surface of, for example, SH3 domains, and to participate in van der Waals contacts and specific hydrogen bonds between carbonyl oxygen atoms of the ligand backbone and functionalities on the aromatic residues of the protein.[6] SH3 domains are enticing conceptual targets for pharmacological intervention in a number of pathologies, for example, AIDS, cancer, and inflammatory diseases, since they occur in several critical, intracellular signaling proteins.[5b, 7] The essential feature of most SH3 binding ligands is the consensus sequence Pro-Xaa-Xaa-Pro (where Xaa represents various amino acids).[8] Synthetic[5, 9] and phage-displayed[7, 10] combinatorial peptide libraries have been generated to understand the ligand properties in more detail, and these studies have yielded compounds that in general bind to SH3 domains with equal or slightly lower affinity than natural Pro-

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