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Registry No. 1, 136316-89-7; (MeCp)Ru(dppe)Cl, 118211-56-6; $[Et_4N]ReS_4$, 103517-31-3.

Supplementary Material Available: Tables of bond distances and angles, atomic coordinates, and thermal parameters (3 pages); a listing of structure factors (30 pages). Ordering information is given on any current masthead page.

Contribution from the Dipartimento di Chimica
e Chimica Industriale, Sezione di Chimica Inorganica,
University of Pisa, Via Risorgimento 35,
I-56126 Pisa, Italy, Scuola Normale Superiore,
Piazza dei Cavalieri, I-56100 Pisa, Italy,
and Institut für Anorganische Chemie der Universität Tübingen,
Auf der Morgenstelle 18, D-7400 Tübingen, Germany

Disproportionation of the Bis(benzene)chromium Cation Induced by Pyridine. Crystal and Molecular Structure of Two Tetrapyridine Complexes Containing Chromium(II) and Chromium(III)[1]

Giovanni Fochi,*,[†] Joachim Strähle,*,[‡] and Franz Gingl[‡]

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Disporportionation of bis(arene)chromium(I) cations has been known since many years, though the reports have been sparse and sometimes contradictory.[2,3]

Among the bases which promote that reaction, 2,2′-bipyridyl appeared to be particularly indicated,[2c] especially because of its chelating properties, which can stabilize Cr(II) and Cr(III) in the form of hexacoordinated complexes. Usually, this is not the case for the related monodentate base pyridine, which seldom yields stable homoleptic structurally established hexacoordinated complexes.[4] In fact, though $Mo(arene)_2^+$ cations readily undergo hydrolytic disproportionation in the presence of aqueous hydroxides,[2a] their oxidation state does not change on moderate heating in neat pyridine.[5]

Nevertheless, the importance of pyridine in the rich and recently clarified disproportionation chemistry of metal carbonyls, another class of low-valent transition-metal complexes,[6] has prompted us to investigate its effects on the $Cr(C_6H_6)_2^+$ cation. We report here that the bis(η^6-benzene)chromium cation disproportionates in pyridine.

Experimental Section

Chemicals. All operations were carried out under an inert atmosphere. Bis(η^6-benzene)chromium hexafluorophosphate was synthesized in 82% yield from bis(η^6-benzene)chromium[2b] by the same procedure as for the corresponding iodide.[2b] Reagent grade pyridine was refluxed 24 h over CaH_2 prior to distillation. Reagent grade toluene was refluxed 3 h over Na and then distilled.

Analyses. Elemental analyses were carried out under an inert atmosphere by Microanalytisches Labor Pascher, Remagen, Germany.

Disproportionation Reaction. A 0.18 M solution of bis(η^6-benzene)chromium hexafluorophosphate in pyridine was kept at 75 °C during 8 h. Violet crystals of $[Cr(py)_4(PF_6)_2]\cdot 2py$ (**1**) (py = pyridine) separated out in 23% yield and were washed with pyridine, which was collected together with the mother liquor. After concentration in vacuo to five-sixths of the initial volume, the clear solution was cooled overnight to −26 °C. This caused black crystals of bis(η^6-benzene)chromium to precipitate (22%). The mother liquor was then diluted 1:1 with toluene and then filtered. The resulting clear solution was kept overnight at −26 °C, yielding pink leaflets of $Cr(py)_5F_2(PF_6)$ (**2**) (10%). Satisfactory complete elemental analyses were obtained for all the reported products ($CrC_{30}H_{30}N_6P_2F_{12}$ (**1**); $CrC_{25}H_{25}N_5PF_8$ (**2**)). To grow a single crystal, **2** was recrystallized from hot ethanol, yielding *trans*-$[Cr(py)_4F_2]PF_6$ (**3**).

Table I. Crystallographic Data for **1** and **3**

	1
$C_{30}H_{30}N_6CrF_{12}P_2$	fw = 816.5
a = 11.375 (1) Å	space group $I422$ (No. 97)
c = 14.084 (1) Å	ρ_{calc} = 1.488 g cm^{-3}
V = 1822.1 Å^3	μ = 4.812 cm^{-1}
Z = 2	$R(F_o)$ = 0.049
T = 18 °C	$R_w(F_o)$ = 0.045
λ = 7.1069 Å	
	3
$C_{20}H_{20}N_4CrF_8P$	fw = 551.4
a = 12.210 (6) Å	space group $C2/c$ (No. 15)
b = 14.177 (3) Å	λ = 1.5418 Å
c = 13.994 (5) Å	ρ_{calc} = 1.591 g cm^{-3}
β = 107.55 (3)°	μ = 55.861 cm^{-1}
V = 2301.5 Å^3	$R(F_o)$ = 0.064
Z = 4	$R_w(F_o)$ = 0.065
T = 18 °C	

Table II. Selected Bond Lengths (Å) and Angles (deg) for **1** and **3**

	Bond Lengths for **1**		
Cr–N1	2.113 (3)	P–F3	1.566 (6)
Cr···F1	2.449 (5)	N1–C1	1.329 (5)
P–F1	1.613 (6)	C1–C2	1.375 (9)
P–F2	1.565 (3)	C2–C3	1.356 (7)
	Bond Angles for **1**		
F1···Cr···F1′	180.0	C2–C3–C2′	116.7 (7)
N1–Cr–N1′	180.0	F1–P–F2	88.8 (2)
N1–Cr–N1″	90.0	F1–P–F3	180.0
Cr–N1–C1	121.2 (2)	F2–P–F2′	177.6 (3)
C1–N1–C1′	117.7 (5)	F2–P–F2″	90.0 (3)
N1–C1–C2	121.8 (5)	F2–P–F3	91.2 (2)
C1–C2–C3	121.0 (6)		
	Bond Lengths for **3**		
Cr–F1	1.853 (2)	P–F21	1.490 (7)
Cr–N1	2.085 (2)	P–F22	1.508 (4)
Cr–N2	2.095 (2)	P–F23	1.560 (3)
N1–C11	1.328 (4)	P–F24	1.477 (7)
N1–C15	1.345 (4)	F1′···H11	2.395 (2)
N2–C21	1.325 (5)	F1···H15	2.389 (2)
N2–C25	1.338 (4)	F1′···H21	2.324 (2)
C–C	1.352 (5)–1.393 (5)	F1···H25	2.395 (2)
	Bond Angles for **3**		
F1–Cr–F1′	179.2 (1)	Cr–N1–C11	121.9 (2)
F1–Cr–N1	89.88 (9)	Cr–N1–C15	120.5 (3)
F1–Cr–N1′	89.58 (9)	C11–N1–C15	117.5 (3)
F1–Cr–N2	91.0 (1)	Cr–N2–C21	120.5 (2)
F1–Cr–N2′	89.6 (1)	Cr–N2–C25	120.7 (2)
N1–Cr–N1′	91.9 (1)	C21–N2–C25	118.6 (3)
N1–Cr–N2	91.34 (9)	F21–P–F22	89.4 (3)
N1–Cr–N2′	176.7 (2)	F21–P–F23	90.2 (2)
N2–Cr–N2′	85.4 (1)	F21–P–F24	180.0

X-ray Data Collection. A crystal of **1** with the approximate dimensions (mm) 0.4 × 0.35 × 0.35 was mounted under N_2 in a glass capillary for intensity data collection. The structure was solved using direct methods.[7] Refinement of the $[Cr(py)_4]^{2+}$ ion and the PF_6^- counterion

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[†] University of Pisa and Scuola Normale Superiore.

[‡] Universität Tübingen.

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0020-1669/91/1330-4669$02.50/0

Table III. Atomic Coordinates of Significant Atoms

atom	*x*	*y*	*z*
		Compound **1**	
Cr	0.000	0.000	0.000
N1	0.1313 (3)	0.1313	0.000
C1	0.2120 (5)	0.1362 (5)	0.0682 (4)
C2	0.2968 (6)	0.2223 (7)	0.0700 (5)
C3	0.3039 (5)	0.039	0.000
P	0.000	0.000	0.2884 (2)
F1	0.000	0.000	0.1739 (4)
F2	0.0176 (6)	0.1364 (3)	0.2861 (2)
F3	0.000	0.000	0.3996 (4)
		Compound **3**	
Cr	0.000	0.10841 (7)	0.250
F1	0.0741 (2)	0.1075 (2)	0.1520 (2)
N1	0.1137 (3)	0.0061 (3)	0.3304 (3)
N2	0.1070 (3)	0.2170 (3)	0.3273 (3)
C11	0.0905 (4)	−0.0458 (3)	0.4011 (4)
C12	0.1682 (5)	−0.1074 (4)	0.4626 (4)
C13	0.2760 (5)	−0.1152 (4)	0.4506 (4)
C14	0.3010 (5)	−0.0637 (4)	0.3783 (4)
C15	0.2183 (4)	−0.0034 (4)	0.3183 (4)
C21	0.0766 (5)	0.2691 (4)	0.3940 (4)
C22	0.1376 (6)	0.3465 (4)	0.4399 (5)
C23	0.2354 (6)	0.3691 (4)	0.4187 (5)
C24	0.2706 (5)	0.3150 (4)	0.3509 (5)
C25	0.2032 (4)	0.2393 (4)	0.3053 (4)
P	0.000	0.6338 (2)	0.250
F21	0.000	0.7389 (7)	0.250
F22	0.1259 (4)	0.6348 (7)	0.3085 (4)
F23	0.0323 (3)	0.6335 (4)	0.1500 (3)
F24	0.000	0.5296 (7)	0.250

with isotropic temperature factors gave *R* values of $R = 0.104$ and $R_w = 0.097$. Anisotropic thermal parameters were introduced for all non-H atoms; H atoms in calculated positions were included in the structure factor calculation. The refinement was based on 528 unique reflections with $I > 3\sigma(I)$ for 64 refined parameters. A crystal of **3** with approximate dimensions (mm) 0.7 × 0.4 × 0.3, mounted on a glass fiber, was used for the data collection. The structure was solved using SHELXS-86 direct methods[7] and completed by subsequent difference electron density maps. The refinement with anisotropic thermal parameters converged to $R = 0.077$ ($R_w = 0.084$). After that the hydrogen atoms were placed in idealized positions and constrained to a riding motion. Final *R* values were based on 2006 unique reflections with $I > 3\sigma(I)$ for 157 refined parameters. Crystals of **1** are tetragonal, while crystals of **3** are monoclinic. Cell dimensions were determined by least-squares refinement of 25 accurately centered reflections, in the θ range of $10.3° < \theta < 16.4°$ and $19.8° < \theta < 35.4°$ for **1** and **3**, respectively. Crystallographic data are summarized in Tables I–III. Intensity data were collected on Enraf-Nonius CAD4 automatic four-circle diffractometers with monochromatized Mo Kα (**1**) or Cu Kα (**3**) radiation. During the data collection three standard reflections were monitored periodically. In the case of **1**, a linear decrease of 16% in intensity was observed; the intensities were corrected accordingly. In the reduction of the data, Lorentz and polarization corrections were applied to both data sets. Further corrections were made for absorption[8] and secondary extinction.[9]

Results and Discussion

Bis(η^6-benzene)chromium hexafluorophosphate disproportionates in pyridine at moderately high temperature (75 °C). Three main products could be isolated thanks to their different solubilities, but the overall yield accounts only for 55% of the mass balance, a few fractions of the reaction mixture being obtained as intractable solids. From neat pyridine, violet crystals of a new compound analyzing as $Cr(py)_6(PF_6)_2$ (**1**) (23%; py = pyridine) separated out during the reaction; black crystals of $Cr(C_6H_6)_2$ (22%) were then isolated on cooling to −26 °C. By addition of toluene to the mother liquor, another new compound was collected, which analyzed as $Cr(py)_5F_2(PF_6)$ (**2**) (10%).

Because the number of pyridine molecules in both **1** and **2** exceeded 4, i.e. the usual highest number of such ligands in a number of complexes,[10] single-crystal X-ray analyses were carried out on both **1** and a violet product, **3**, *trans*-$[Cr(py)_4F_2]PF_6$, derived from **2** by recrystallization from hot ethanol.[11]

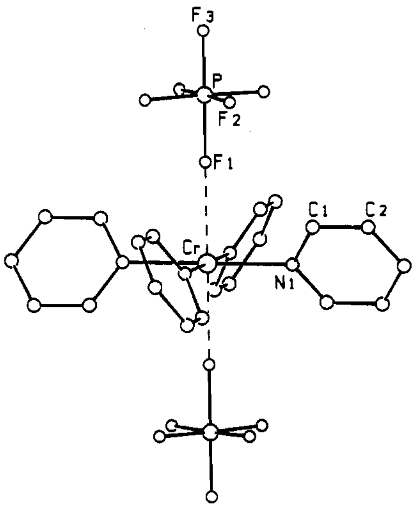

Figure 1. Molecular structure of $[Cr(py)_4(PF_6)_2]$ in **1** (the two noncoordinated pyridine molecules omitted).

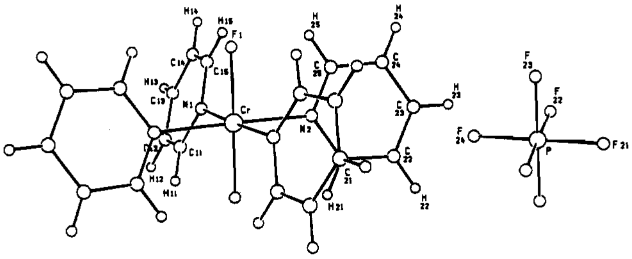

Figure 2. Structure of **3**. The cation and the anion as a whole have C_2 symmetry, with Cr, F24, P, and F21 on the 2-fold axis.

The structures of **1** and **3** are shown in Figures 1 and 2, respectively. Selected bond lengths and angles of both complexes are given in Table II, and the atomic coordinates of the significant atoms are listed in Table III.

At first, we stress that both complexes are octahedral, with an equatorial set of four pyridine ligands; the two additional pyridine moieties in the composition of **1** are clathrated; thus, they do not enter the coordination sphere of Cr(II), contrary to a generally accepted view.[12,13]

In both **1** and **3**, the two ligands different from pyridine are in trans geometry. As to $[Cr(py)_4F_2]^+$, this is the first crystallographic report on such cation, although the trans array of the fluoro ligands was hitherto accepted on the basis of spectral properties.[14,15]

In **1** the axial positions are occupied by two fluorine atoms belonging to the PF_6^- counterions; it is remarkable that the Cr···F1 distance (2.449 (5) Å) is only slightly longer than the Cr–F(axial) bond length in CrF_2 (2.43 Å),[17] thus envisaging each Cr–PF_6 group in **1** as a tight ion pair. The coordination geometry of chromium

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(11) **3** contains one pyridine molecule less than **2**. By analogy with the structure of **1** and other complexes[10] and on the basis of the stability toward ethanol of the chromium(III)–pyridine bond in complexes where not more than four pyridine ligands are present,[12] we believe that a lattice pyridine molecule had been lost from **2** on recrystallization.

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may probably best be described as a square-planar chromium(II) ion-paired to fluorine (the cationic moiety having D_4 and the anions C_{4v} symmetry, respectively) or alternatively as an octahedron elongated due to Jahn–Teller distorsion of the d^4 system. This interaction is likely to be responsible for the small lengthening of the bond between the bridging fluorine atom and phosphorus (about 0.05 Å with respect to the other P–F bonds) [this could suggest that **1** is a precursor to **2** in a Cr(II) $\rightarrow$ Cr(III) oxidation process, where Cr(I) could be the oxidant[2c]].

A comparison of the orientation of the planes of the pyridine ligands in **1** and **3**, respectively, reveals that while **1** contains a four-bladed propeller, thus lowering the steric repulsions between the *o*-H's,[4b] an unusual overall disposition is found in **3**, similar to that observed in *trans*-PPh_3F_2.[18] The hypothesis of F···H attractive interactions[19] could explain the observed less tilted orientation of the pyridine rings in **3** with respect to **1** (the dihedral angle between the $Cr-N_4$ and individual py planes is 58.1 (1)° in **1**, while it is 69.3 (1) and 73.2 (1)° for py1 and py2, respectively, in **3**). C–H···F hydrogen bonds could be postulated in **3**, the H···F distances resembling the H···O contacts found in some bent C–H···O hydrogen bonds.[21]

Registry No. **1**, 136342-74-0; **2**, 136342-72-8; **3**, 136342-71-7; bis(η^6-benzene)chromium hexafluorophosphate, 51160-14-6.

Supplementary Material Available: For **1** and **3**, text describing the structure determination procedure and parameters and tables listing positional and thermal parameters, bond distances and angles, and least-squares planes (15 pages); tables listing observed and calculated structure factors (13 pages). Ordering information is given on any current masthead page.

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[19] The F···H distances (Table II) are shorter than the sum of the van der Waals radii of fluorine and hydrogen.[20]

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Contribution from Ames Laboratory and the Department
of Chemistry, Iowa State University, Ames, Iowa 50011

Rational Design of Linear Trinuclear Metal Complexes

L. Keith Woo* and Mannar R. Maurya

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Introduction

A great deal of interest in multimetallic complexes derives from the special chemical and physical properties resulting from the mutual interaction of two or more metal centers.[1] Of particular interest are one-dimensional chains of metal complexes.[2] Materials of this type have exhibited unusual magnetic, optical, and conduction properties.[3] Despite the great interest in this area, the development of widely applicable, rational methods for synthesis of desired metal chain complexes remains elusive.

As part of a program to prepare metal-containing polymers, we have synthesized multichelating ligand systems. We recently reported the synthesis of a new binucleating ligand, *cis*-5,15-bis[*o*-(β-alanylamido)phenyl]-2,8,12,18-tetraethyl-3,7,13,17-tetramethylporphyrin, $[H_2(DPE)](NH_2)_2$,[4] and showed that the porphyrin core of this ligand can be metalated to produce mononuclear building blocks.[5] We now report that these complexes can be readily used to prepare linear trinuclear metal complexes.

Experimental Section

All solvents and metal salts used in the syntheses were of analytical grade. Synthesis and characterization of $[H_2(DPE)](NH_2)_2$ and its Zn(II), Cu(II), and Ni(II) complexes were accomplished by literature procedures.[5] Elemental analysis was performed by Galbraith Laboratories, Inc., Knoxville, TN. Visible spectra were recorded at room temperature on a HP 8452A diode array spectrophotometer using chloroform as solvent. Infrared spectra were recorded as Nujol mulls on an IBM IR-98 Fourier transform infrared spectrophotometer. ^1H NMR spectra were recorded on a Nicolet NIC 300 spectrometer using CD_2Cl_2 as solvent. Chemical shifts (in ppm) are reported relative to CD_2Cl_2 (5.32 ppm). EPR spectra were recorded in frozen $CHCl_3$ solution on an IBM ER-200 EPR spectrometer. Molecular weights were determined in $CHCl_3$ solutions with a Knauer vapor pressure osmometer.

$[\{[Ni^{II}(DPE)](NH_2)_2\}_2Zn](ClO_4)_2$. A methanolic solution (4 mL) of $Zn(ClO_4)_2$ (22.0 mg, 0.06 mmol) was added to $[Ni^{II}(DPE)](NH_2)_2$ (100.0 mg, 0.12 mmol) dissolved in 10 mL of $CHCl_3/CH_3OH$ (9:1). The resulting mixture was stirred at room temperature for 4 h and then heated at reflux for 24 h. After removal of the solvent under reduced pressure, the crude solid was dissolved in 5 mL of CH_3OH, and the solution was treated with 16.3 mg (0.12 mmol) of $NaClO_4$ in 2 mL of CH_3OH. The mixture was stirred at room temperature for 4 h and was subsequently evaporated to dryness. Water (15 mL) was added to the resulting crude $[\{[Ni(DPE)](NH_2)_2\}_2Zn](ClO_4)_2$, and the suspension was extracted with CH_2Cl_2 (3 × 40 mL). The combined organic layers were dried over anhydrous $MgSO_4$ and filtered. Removal of the solvent under reduced pressure yielded 86 mg (73%) of pure purple product. UV/vis ($CHCl_3$): 408 (Soret), 532, 568 nm. ^1H NMR (CD_2Cl_2): 9.38 (s, 4 H, meso H), 8.25 (d, 4 H, aryl), 8.01 (d, 4 H, aryl), 7.64 (t, 4 H, aryl), 7.45 (t, 4 H, aryl), 6.89 (s, b, 4 H, NH), 3.62 (q, 16 H, CH_2CH_3), 3.47 (s, b, 8 H, NH_2), 2.19 (s, 24 H, CH_3), 2.08 (t, 8 H, CH_2CH_2), 1.55 (t, 24 H, CH_2CH_3), 1.30 (t, 8 H, CH_2CH_2). IR (mull): $\nu(ClO_4^-) = 1107, 623$ cm^{-1}. Anal. Calcd for $C_{100}H_{112}N_{16}O_{12}Cl_2Ni_2Zn$: C, 60.55; H, 5.65; N, 11.30. Found: C, 60.92; H, 6.04; N, 10.99. Molar conductance (acetone): 190.6 mhos mol^{-1} cm^2.

$\{[Zn^{II}(DPE)](NH_2)_2\}_2CuCl_2$. A methanolic solution (5 mL) of $CuCl_2$ (10.0 mg, 0.06 mmol) was added to a solution of $[Zn^{II}(DPE)](NH_2)_2$ (100.0 mg, 0.12 mmol) dissolved in 10 mL of $CHCl_3/CH_3OH$ (9:1). After the mixture was refluxed for 24 h, the solvent was removed under reduced pressure. Water (15 mL) was added to the residue, the suspension was filtered through a Celite pad, and the solid was washed with water. After the Celite pad was dried in vacuo, the product was washed from Celite with $CHCl_3$. The solution was concentrated to ca. 5 mL, and 5 mL of hexane was added. After cooling to −10 °C for several days, the solution was filtered to yield 63 mg (53%) of brown-red microcrystals. UV/vis ($CHCl_3$): 410 (Soret), 532, 572 nm. EPR ($CHCl_3$, 110 K): $g_\parallel = 2.21$, $g_\perp = 2.07$, $a_{Cu} = 197.5$ G, $a_N = 16.4$ G. Anal. Calcd for $C_{100}H_{112}N_{16}O_4Cl_2Zn_2Cu\cdot{}^1/_2CHCl_3$: C, 62.64; H, 5.88; N, 11.63; Cl, 6.44. Found: C, 62.53; H, 6.01; N, 11.21; Cl, 7.38. Mol wt: calcd, 1865; found, 1742.

$\{[Ni^{II}(DPE)](NH_2)_2\}_2CuCl_2$. This purple-red compound was prepared in the same fashion as described for $\{[Zn(DPE)](NH_2)_2\}_2CuCl_2$ using 100.0 mg (0.12 mmol) of $[Ni(DPE)](NH_2)_2$. Yield: 47 mg (41%). UV/vis ($CHCl_3$): 408 (Soret), 532, 568 nm. Anal. Calcd for $C_{100}H_{112}N_{16}O_4Cl_2Ni_2Cu\cdot CHCl_3$: C, 61.46; H, 5.73; N, 11.36. Found: C, 61.49; H, 6.37; N, 11.01. Mol wt: calcd, 1852; found, 1881.

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