In order to examine the effect of steric hindrance on the coordination of metallic derivatives of **3**, we have prepared their zinc complexes. Their electronic spectra are much more intense than those of other zinc tetraphenylporphyrins.[9] The formation constants for five-coordinated species with pyridine are very weak, between 14 and 40 L·M^{-1}. A 100-fold reduction in equilibrium constants is observed in comparison to that measured with zinc porphyrins hindered on both faces.[10]

The assignment of the ^{13}C NMR spectra of phthalocyanines **5** and **2** was carried out by analogy of these with **4**. The spectrum of **5** exhibits nine resonances for the aromatic part, 11 for glucose groups, and six for the methyl of isopropylidenes.[11] This multiplicity of peaks could represent some possible environmental interactions present in several isomers. The phthalocyanines **5** and **2** could exist as a mixture of positional isomers around the phthalocyanine ring.

Zinc phthalocyanine **5**, in organic solvents, exhibits a spectrum typical of monomeric metalled phthalocyanine, with a Soret band and Q band.[12] The optical spectra of **2** are characteristic of a dimer in water and of a monomer in DMSO.[12]

In conclusion, our preliminary work has established that the new class of tetrapyrrollic macrocycles on which glucopyranoses or glucofuranoses were linked allows an increase in both their steric hindrance in the vicinity of the metal site and their solubility in water. Futher work, directed on studies of other metal complexes, is now in progress.

Acknowledgment. This work was supported by the Institut National de la Santê et de la Recherche Mêdicale.

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[9] UV–visible spectra in $CHCl_3$: λ_{max} nm (ϵ/mmol·L^{-1}). **Zn3a**: 404 (40), 425 (517.3), 557 (17.2), 593 (5.5). **Zn3b**: 405 (49.5), 426 (632), 558 (23.2), 595 (5.9). **Zn3c**: 404 (53.2), 425.5 (720.2), 557 (25), 594 (5.8).

[10] The equilibrium constant for the complexation of zinc basket handle porphyrin [a-$(C_{12})_2$-CT]^{2b} was found to be 3.6 × 10^3 L·M^{-1} in CH_2Cl_2 at 25 °C.

[11] ^{13}C NMR spectra of phthalocyanines in C_6D_6 at 24 °C (δ en ppm). **5**: 167.3, 159.4, 125, 124, 120.8, 120.5, 118, 110, 108 (C phthalyl), 106.2, 105.8, 83.4, 82.9, 81.6, 81.1, 73.3, 72.6, 60.7 (C oses), 112.5, 109.6 (C isopropylidene), 27.25, 27, 26.6, 26.3, 25.8, 25.5 (C methyl), 141, 133 (C pyrrolic). **2**: 170.7, 159.3, 158.5, 125.5, 123.5, 118.8, 113.2, 112.2 (C phthalyl), 113.2, 112.3, 98, 94, 86.7, 84.2, 77.7, 75.7, 73.1, 73, 71, 62.7, 62.5 (C oses), 136.4 (C pyrrolic).

[12] UV–visible spectra: λ_{max} nm (ϵ/mmol·L^{-1}). **5** (acetone): 348 (101.7), 611 (34.8), 677 (176.2). **2** (DMSO): 354 (35.4), 618 (14.3), 685.5 (73).

[Cp*(Me)Cr(μ-Me)]$_2$: An Electron-Deficient Chromium(III) Alkyl with Bridging Methyl Groups and a Cr–Cr Bond

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Electron-deficient organometallic compounds of both main-group and transition elements may exhibit bridging alkyl ligands.[1] In the special case of bridging methyl groups, several different binding modes have been observed (featuring symmetric pyramidal, symmetric planar, or agostic μ-CH_3 groups).[2] As part of our continuing exploration of the reactivity of paramagnetic chromium(III) alkyls,[3] we have now prepared a complex that displays two methyl groups bridging chromium atoms with 13 electron configurations. Herein we report the full characterization of this compound and some theoretical considerations concerning the metal–metal interaction in this d^3–d^3 system.

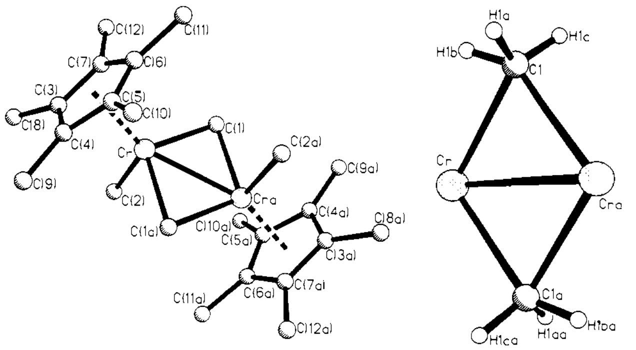
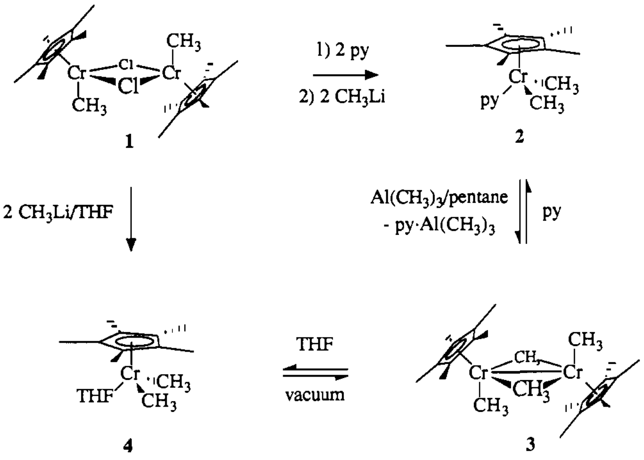


Figure 1. The molecular structure of **3**. Selected bond distances: Cr–Cra, 2.606 (2) Å; Cr–C1, 2.206 (4) Å; Cra–C1, 2.170 (5) Å; Cr–C2, 2.096 (5) Å; Cr–H1b, 2.124 Å; C1–H1a, 0.965 Å; C1–H1b, 0.874 Å; C1–H1c, 0.939 Å. Interatomic angles: Cr–C1–Cra, 73.1 (1)°; C1–Cr–C1a, 106.9 (1)°; Cr–C1–H1b, 73.1°; H1a–C1–H1b, 89.9°; H1a–C1–H1c, 114.3°; H1b–C1–H1c, 104.7°.

Scheme I



Consecutive addition of 2.0 equiv each of pyridine and methyllithium to dimeric [Cp*(Me)Cr(μ-Cl)]$_2$ (**1**) afforded the half sandwich complex Cp*Cr(Py)Me$_2$ (**2**) in 73% yield.[4] Reaction of this compound with 1.0 equiv of trimethylaluminum in pentane followed by rapid workup (evaporation of solvent and recrystallization from pentane at −35 °C) yielded brown crystals of [Cp*(Me)Cr(μ-Me)]$_2$ (**3**) (see Scheme I). Once **3** had been identified, a more direct synthesis was attempted. Addition of 2.0 equiv of methyllithium to a THF solution of **1** resulted in a rapid color change from purple to green. However, upon evaporation of the THF, the solid residue turned brown, and extraction with pentane yielded **3** in 65% isolated yield.[5] **3** was air sensitive,

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[4] (a) **2**: ^{1}H NMR (C_6D_6) 25.9 (Cp*), 23.3, −50.5, −59.6 ppm; IR (Nujol) 1599 (s), 1500 (s), 1480 (s) 1383 (s), 1376 (w), 1211 (m), 1062 (m), 757 (s), 697 (s), 640 (m) cm^{-1}. Anal. Calcd for $C_{17}H_{26}CrN$: C, 68.89; H, 8.84; N, 4.73. Found: C, 68.71; H, 8.76; N, 4.56. The crystal structure of **2** has been determined by X-ray diffraction and will be reported in a full paper. (b) Related complexes and their NMR spectra have been reported by Grohmann et al.: Grohmann, A.; Köhler, F. H.; Müller, G.; Zeh, H. *Chem. Ber.* **1989**, *122*, 897.

[5] **3**: ^{1}H NMR (C_6D_6) 88.6 (br, 3 H), 75.8 (br, 3 H), 0.56 (15 H) ppm; IR (Nujol) 2188 (w), 1182 (s), 1108 (m), 1022 (s), 804 (m), 721 (m), 616 (m) cm^{-1}.

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and solutions of it in noncoordinating solvents decomposed at room temperature with a half-life of ca. 5 h. The decomposition proceeded cleanly and produced methane and a new chromium complex. The full characterization of this compound, to which we tentatively assign the structure $[Cp^*(Me)Cr]_2(\mu\text{-}CH_2)$, as well as other reactions of **3** will be the subject of a future report. Dissolution of crystalline **3** in THF, however, produced brilliant green, stable solutions of the THF adduct **4** (^1H NMR in THF-d_8, 21.8 ppm (Cp*), methyl groups not observed[4b]) during the course of several minutes. It is informative to compare the rather low affinity of the highly electron deficient Cp^*CrMe_2 for THF with the stronger binding of the same ligand in the isolable cationic complex $[Cp^*Cr(THF)_2Me]^+BPh_4^-$.[3d]

The crystal structure of **3** was determined by X-ray diffraction at −80 °C, and the result is shown in Figure 1.[6] **3** is a dimer and possesses a crystallographic inversion center. Each chromium atom is bonded to one pentamethylcyclopentadienyl ligand, one terminal methyl group, and two bridging methyl ligands, thus completing the familiar pseudooctahedral coordination environment of a three-legged piano stool. The Cr–Cr distance of 2.606 (2) Å is the shortest such distance we have yet found in this series of edge-sharing Cr^{III} bioctahedra and is certainly consistent with metal–metal bonding (see below).[7] The hydrogen atoms of the bridging methyl groups were located in a difference map, and their positions were refined. We are not certain whether the Cr–(μ-CH_3) bonds have some agostic character. The Cr–H1b distance (2.124 Å) is shorter than the Cr–C1 distance (2.206 Å), and the Cr–C1–H1b angle is fairly acute (73.1°). In addition, the infrared spectrum of **3** shows a weak absorption at 2188 cm^{-1}. However, the latter value is outside of the commonly accepted range of C–H vibrations involved in agostic interactions (i.e., 2700–2300 cm^{-1}),[8] and the distances and angles involving hydrogen atom positions derived from an X-ray diffraction study must of course be viewed with some caution. Due to the paramagnetic nature of **3**, the NMR experiments commonly used to resolve this ambiguity are not applicable in this case.

Many binuclear chromium(III) complexes with two bridging ligands have been structurally characterized, and their magnetism has been studied. All are thought to exhibit repulsive interactions between the metal atoms (Cr–Cr > 3.0 Å).[9] To address the extent of metal–metal bonding in **3**, we have performed EHMO calculations on $[Cp(Me)Cr(\mu\text{-}Me)]_2$. The short Cr–Cr distance of **3** (compared to 3.287 Å in $[Cp(Me)Cr(\mu\text{-}Cl)]_2$ or even 2.837 Å and 2.793 Å in $[CpCr(\mu\text{-}Cl)]_3(\mu_3\text{-}CH))$[3a] does not by itself imply the presence of a metal–metal bond. It is more appropriately viewed as the consequence of the three-center/two-electron bonding associated with the bridging methyl groups. As may be seen from the theoretical analysis of methyl (or hydrogen) bridged dimers containing boron[10] or aluminum[11] and also osmium trimers,[12] metal–metal interactions are implicit in such bonding. Figure 2 shows how combination of the valence orbitals of two Cp(Me)Cr fragments and the sp^3 hybrid orbitals of the two bridging groups gives rise to partial Cr–Cr σ and π bonds due to core levels. Replacement of a two-electron methyl bridge with a four-electron ligand like chloride generates additional bridge–metal bonding combinations, which are, however, metal–metal antibonding in character.[11c,12] This may be illustrated by listing the changes in Cr–Cr overlap populations due to core orbitals only, as the methyl groups are replaced by chloride bridges (i.e., $[Cp_2Me_2Cr_2(\mu\text{-}Me)_2]^{6+}$, 0.186; $[Cp_2Me_2Cr_2(\mu\text{-}Me)(\mu\text{-}Cl)]^{6+}$, 0.115; $[Cp_2Me_2Cr_2(\mu\text{-}Cl)_2]^{6+}$, 0.043).[13] We also note the similarity in trend between the above-mentioned chromium complexes and the series $Me_2Al(\mu\text{-}Me)_2AlMe_2$ (Al–Al, 2.606 Å),[14] $Me_2Al(\mu\text{-}Me)(\mu\text{-}NPh_2)AlMe_2$ (Al–Al, 2.72 Å),[15] and $Me_2Al(\mu\text{-}Cl)_2AlMe_2$ (Al–Al, 3.22 Å).[16]

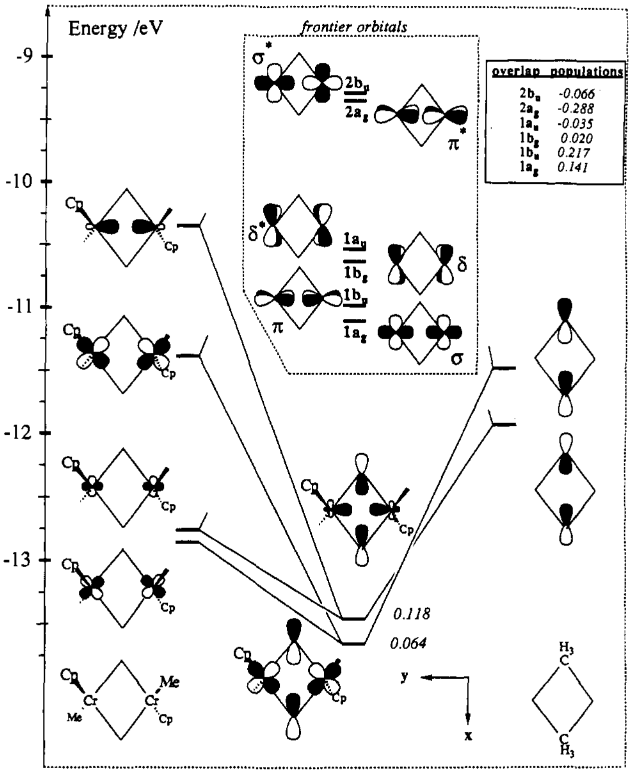

Figure 2. Partial interaction diagram for **3**. Numbers in italics are overlap populations; symmetry labels were assigned with the assumption of C_{2h} symmetry. The frontier d orbitals (inner box) are on the same energy scale as the other levels. The overlap populations for these orbital were calculated for doubly occupied levels.

The effect of the chromium d orbitals (spread over 1.8 eV and filled with six electrons; see Figure 2) on Cr–Cr bonding depends strongly on the electron configuration chosen. A low-spin configuration ($1a_g^2 1b_u^2 1b_g^2$) would be strongly bonding (overlap population: 0.378) while the high-spin state ($1a_g^1 1b_u^1 1b_g^1 1a_u^1 2a_g^1 2b_u^1$) would be slightly antibonding (overlap population: −0.006). The extended Hückel method[17] does not take into account electron–electron interactions and can thus not be expected to give a reliable prediction for the ground state of **3**. However, due to the large energy gap between the $1a_u$ (δ^*) and $2a_g$ (π^*) levels, only the four lowest d-orbital-based MOs should be populated. Thus our calculations point to a singlet or triplet ground state or a mixture thereof. These configurations retain a large portion of the d-block overlap population, namely, the contribution from the $1a_g$ (σ) and $1b_u$ (π) levels.

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(6) Brown cubes from pentane; monoclinic $P2_1/c$; a = 8.062 (4) Å, b = 14.865 (5) Å, c = 10.065 (3) Å, β = 106.76 (3)°; Z = 4, R = 0.058, R_w = 0.084.

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Experimental support for the above conclusions was obtained by measurements of the magnetic susceptibility of **3** in the temperature interval 65–300 K. The effective magnetic moment of **3** increased with temperature, consistent with strong antiferromagnetic coupling.[18] At room temperature, μ_{eff} reached a value of only 2.1 μ_B, which may be compared to the μ_{eff} of 3.5 μ_B for **1** at the same temperature.[3e] The close approach of the two metal atoms in **3** apparently leads to a significant splitting of the d orbitals and enforces spin pairing. We conclude that **3** exhibits an exceptional case of metal–metal bonding between two octahedral Cr(III) ions.

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Supplementary Material Available: X-ray structure determination summary and tables of atomic coordinates and equivalent isotropic displacement coefficients, bond lengths and angles, anisotropic displacement coefficients, and hydrogen atom coordinates and isotropic displacement coefficients for **3** (6 pages). Ordering information is given on any current masthead page.

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Uses of Metallacyclobutenes in Heterocyclic Synthesis. Synthesis and Structural Characterization of 1,2-Dihydrophosphetes

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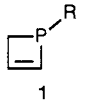
In contrast to the wealth of chemistry reported for many small-ring heterocyclic compounds, there is a surprising dearth of information on the 1,2-dihydrophosphete ring system (structure **1**). Though a transition-metal complex of a 1,2-dihydrophosphete was recently reported,[2] in no case has a simple 1,2-dihydrophosphete been isolated; existing reports are of the corresponding 1-oxides (structure **2**).[3-5]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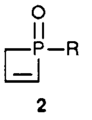
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As part of a continuing study of the synthesis of carbocyclic and heterocyclic compounds through the intermediacy of metallacyclobutenes, we have discovered an apparently general synthetic route to these 1,2-dihydrophosphete heterocycles. A variety of substituents may be introduced, and the heterocycles may be readily isolated in good yield as the unoxidized dihydrophosphetes (structure **1**).

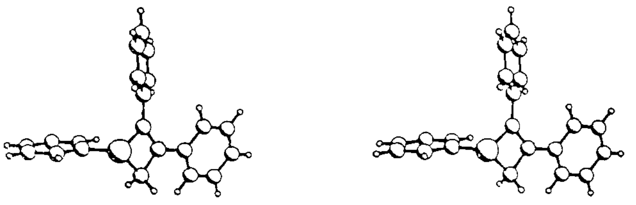
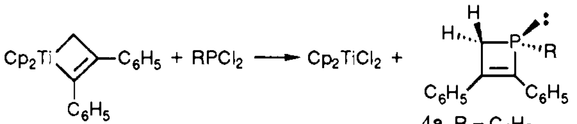


Figure 1. Stereoscopic view of 1,2-dihydrophosphete **4a**. Bond lengths[12] (Å): P(1)–C(2), 1.821 (3); P(1)–C(4), 1.886 (4); C(2)–C(3), 1.366 (5); C(3)–C(4), 1.517 (5); P(1)–C(11), 1.842 (3); C(2)–C(21), 1.477 (4); C(3)–C(31), 1.480 (4). Bond angles (deg): C(4)–P(1)–C(11), 104.7 (1); C(2)–P(1)–C(4), 74.0 (2); C(2)–P(1)–C(11), 104.3 (1); P(1)–C(2)–C(3), 95.5 (2); P(1)–C(2)–C(21), 128.6 (2); C(3)–C(2)–C(21), 135.8 (3); C(2)–C(3)–C(4), 101.3 (3); C(2)–C(3)–C(31), 134.3 (3); C(4)–C(3)–C(31), 124.3 (3); P(1)–C(4)–C(3), 88.0 (2).

When the diphenyltitanacyclobutene **3**[6] is treated with 1 equiv of phenyldichlorophosphine in benzene solution at room temperature, titanocene dichloride immediately precipitates in nearly quantitative yield as a red microcrystalline solid.[7] Filtration under nitrogen of the reaction mixture through a short column of silica, eluting with deoxygenated diethyl ether, followed by evaporation of solvents in vacuo, affords the dihydrophosphete **4a** as a white solid in 66% yield.[8]



3

4a, R = C_6H_5
b, R = CH_3CH_2
c, R = $(CH_3)_3C$
d, R = CH_3CH_2O

The dihydrophosphete **4a** (and its congeners; vide infra) is most readily characterized by its ^{1}H NMR spectrum. The two protons of the methylene group are stereochemically inequivalent and, at moderate field (360 MHz), are sufficiently well resolved to give rise to a simple first-order pattern of two doublets of doublets (J_{H-H} = 14.4 Hz, J_{P-H} = 9.6, 4.2 Hz).

Single crystals of **4a** were obtained from a cooled toluene/pentane solution. X-ray crystallographic analysis of **4a** proceeded routinely.[9] A stereoscopic view of the structure of **4a** is provided in Figure 1. The phosphacyclobutene ring is roughly planar, with an average deviation from planarity of 0.05 Å. The C–P–C bond angle of 74.0° is identical with that reported by Mathey[2] for the tungsten pentacarbonyl complex of a related 1,2-dihydrophosphete, as is the sp^3 C–sp^2 C bond length of 1.517 Å. The C=C double bond is somewhat longer than in the Mathey compound, at 1.366 Å (compared to 1.331 Å), while the sp^3 C–P bond is somewhat shorter, at 1.886 Å (compared to 1.902 Å). Other relevant bond lengths and angles are presented in the caption to Figure 1. In short, **4a** displays a ground-state structure quite consistent with simple formulation as the 1,2-dihydrophosphete. Though partial contribution of the ring-opened phosphabutadiene structure has been invoked to account for the structure of the tungsten pentacarbonyl complex of a 1,2-dihydrophosphete,[2] it does not appear necessary to invoke participation of the phosphabutadiene structure

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(8) Full characterization of **4a–d**, as well as experimental details for the preparation of **4a**, are provided in the supplementary material.

(9) Crystal data: $C_{21}H_{17}P$, M = 300.343, monoclinic, space group $P2_1/n$, a = 9.265 (4) Å, b = 6.863 (3) Å, c = 26.082 (11) Å, β = 93.826 (11)°, V = 1655 Å^3, Z = 4, D_{calcd} = 1.2 g cm^{-3}. Atoms were located by direct methods. Least-squares refinement converged to an R value of 0.050 for 1471 reflections [$I > 3\sigma(I)$]. Crystallographic data are provided in the supplementary material.