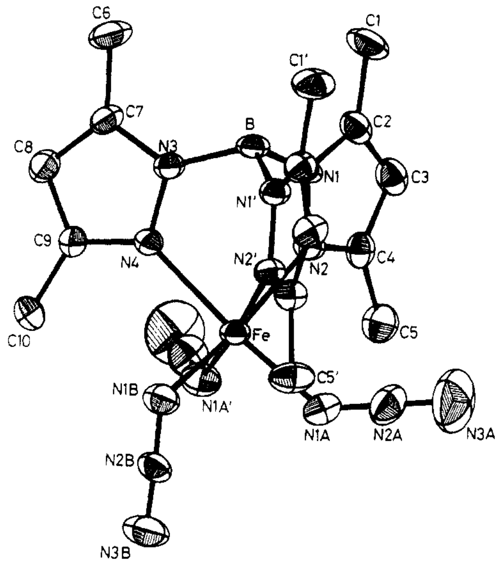

Figure 2. ORTEP drawing of the crystal structure of the anion in **4**.

Table V. Selected Bond Distances (Å) and Angles (deg) for $[(HB(3,5\text{-}Me_2pz)_3)_2Fe(N_3)_3]^-$

	Distances		
Fe–N1A	2.014 (7)	Fe–N1B	2.032 (8)
Fe–N2	2.224 (7)	Fe–N4	2.163 (5)
N1A–N2A	1.159 (10)	N2A–N3A	1.158 (16)
N1B–N2B	1.189 (8)	N2B–N3B	1.124 (9)
N1–N2	1.376 (8)	N1–C2	1.354 (11)
N1–B	1.521 (13)	N3–N4	1.382 (4)
N3–C7	1.354 (7)	N3–B	1.543 (6)
C1–C2	1.492 (10)	C2–C3	1.391 (16)
C3–C4	1.406 (9)	C4–C5	1.499 (14)
C4–N2	1.332 (15)	C6–C7	1.500 (6)
C7–C8	1.386 (6)	C8–C9	1.401 (7)
C9–C10	1.481 (6)	C9–N4	1.346 (7)
	Angles		
N1–N2A–N3A	179.3 (1.6)	N1B–N2B–N3B	177.0 (1.2)
N1A–Fe–N1B	93.0 (3)	Fe–N1A–N2A	135.1 (5)
Fe–N1B–N2B	122.4 (8)	N2–Fe–N1A	92.6 (2)
N2–Fe–N1B	172.0 (3)	N4–Fe–N1A	174.6 (2)
N4–Fe–N1B	90.5 (2)	N2–Fe–N4	83.5 (2)
B–N1–C2	130.5 (6)	B–N1–N2	119.8 (6)
B–N3–C7	130.6 (3)	B–N3–N4	119.4 (4)
N1–B–N3	108.9 (4)		

$FeOFe(HB(pz)_3)]^{2+}$. In fact, Wieghardt et al. reported that an aerobic treatment of $LFeCl_3$ with NaOAc gives $LFeO(OAc)_2FeL$.[3a,3c] However, attempts to synthesize $(HB(pz)_3)FeO(OAc)_2Fe(HB(pz)_3)$ by treating **2** with NaOAc under various reaction conditions were all unsuccessful.

Although **2** and **3** were found to not be useful as a starting material for the synthesis of a binuclear iron complex, they may be suited as reagents for preparing a class of mononuclear iron complexes by substitution of the chloride. This possibility is demonstrated in the reaction of **3** and NaN_3 to give $(Et_4N)[(HB(3,5\text{-}Me_2pz)_3)Fe(N_3)_3]$ (**4**). **4** is also obtained by the direct reaction of **1** and $KHB(3,5\text{-}Me_2pz)_3$ in the presence of NaN_3. A similar mononuclear azido complex $LFe(N_3)_3$ (L = 1,4,7-triazacyclononane and 1,4,7-trimethyl-1,4,7-triazacyclononane) was reported.[3b] The structure of $[(HB(3,5\text{-}Me_2pz)_3)Fe(N_3)_3]$ anion of **4** is shown in Figure 2, and the bond lengths and angles are summarized in Table V. The complex contains a crystallographically imposed miller plane. One azide ion (N1B–N2B–N3B) lies on the Miller plane, whereas the other two (N1A–N2A–N3A and N1A′–N2A′–N3A′) are related by the symmetric operation. The azide ion (N1B–N2B–N3B) positions itself along the axis formed by the boron and iron, whereas the other azide ions are almost perpendicular. The bond distances Fe–N1A and Fe–N1B are close to each other (ca. 2.0 Å) and are shorter than those between the iron and nitrogens from the tris(pyrazolyl)borate ligand (ca. 2.2 Å). The distances between the iron and azide nitrogens are slightly longer than one reported for an azido iron(III) porphyrin complex $(TPP)Fe(N_3)(py)$[9] (TPP = tetraphenylporphyrinato, py = pyridine) (1.925 (7) Å). The Fe–N1A–N2A angle (135.1 (5)°) is higher than that of Fe–N1B–N2B (122.4 (8)°). The latter value is typical for azido transition-metal complexes whereas the former one is not; for instance, the angle found in $(TPP)Fe(N_3)(py)$ is 125.6 (7)°.[9] The spreading of the angle of Fe–N1A–N2A may be due to the steric hindrance of the ligand, since the azide ion (N1A–N2A–N3A or N1A′–N2A′–N3A′) positions itself closer to the methyl groups from the tris(pyrazolyl)borate ligand than the other azide ion (N1B–N2B–N3B).

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Supplementary Material Available: Tables SI–SV, listing crystallographic details, thermal and hydrogen atom parameters, and bond distances and angles for **2** and **4** (12 pages); Table SVI, listing observed and calculated structure amplitudes for **2** and **4** (11 pages). Ordering information is given on any current masthead page.

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^{31}P and ^{195}Pt NMR Data of Several "T-Frame" Diplatinum Complexes That Exhibit Unusual Structure, Bonding, and Reactivity

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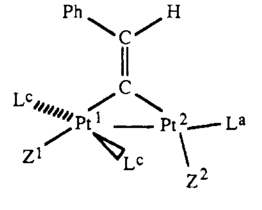
We reported recently the results of an initial, though comprehensive, study of the synthesis, structure, bonding, and reactivity of several new dinuclear complexes, including the diplatinum compounds **1a–g**.[1] The molecular structures of complexes **1a**, **1b**, **1d**, and **1f** have been determined by X-ray diffraction.[1a,2] The more interesting results of this study are as follows. (1) The molecules possess a "T-frame" structure of C_s symmetry in which the principal coordination planes of the two platinum atoms have an orthogonal relative orientation. (2) The Pt–Pt distance decreases from 2.750 (2) Å for **1a** to 2.685 (1) Å for **1f** as the electronegativity of the ancillary set of ligands increases. (3) Theoretical calculations of the electronic structure of the cationic complex **1a** indicate a large negative charge localization (nearly −1.0) on each platinum atom and a net repulsive interaction



$L^a = L^c = PEt_3$

cpd#	Z^1	Z^2
1a	PhC≡C	$P^bEt_3{}^+$
1b	PhC≡C	Cl
1c	PhC≡C	Br
1d	PhC≡C	I
1e	Cl	Cl
1f	Br	Br
1g	I	I

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Table I. ^{31}P and ^{195}Pt NMR Data for Complexes **1a–g** (δ, ppm; J, Hz)^{a,b}

	^{31}P NMR data						^{195}Pt NMR Data		
	L^a			$L^{c,c}$					
compd	δ	$^1J_{PtP}$	$^2J_{PtP}$	δ	$^1J_{PtP}$	$^2J_{PtP}$	$\delta(Pt^1)$	$\delta(Pt^2)$	$^1J_{PtPt}$
1a^c	23.4	4945	547	13.5	2162	47	−2204	−2122	1604
1b	27.2	5672	414	13.7	2215	40	−2166	−1860	902
1c	28.2	5644	399	13.4	2212	53	−2193	−1929	866
1d	31.3	5599	376	12.4	2212	37	−2227	−2021	602
1e	20.7	5481	614	14.8	2311	51	−1895	−1897	724
1f	20.7	5426	603	13.8	2281	47	−2012	−1940	603
1g	25.4	5347	567	14.3	2251	46	−2267	−2030	567

^{a} ^{31}P{H} and ^{195}Pt{H} spectra were recorded by direct observation on IBM NR-200 (Pt or P) and Brucker AM-400 (P only) instruments at normal probe temperatures as $CDCl_3$ solutions; $L^a = PEt_3$ and $L^{c,c} = PEt_3$. ^{b} $\delta(^{31}P)$ values are relative to 85% H_3PO_4 as an external reference. $\delta(^{195}Pt)$ values are relative to a frequency of 21.4 MHz on a spectrometer where TMS resonates at exactly 100 MHz. For example, before correction to this reference, the TMS resonance appears at 200 132 341 Hz and the Pt(1) and Pt(2) resonances of **1a** appear, respectively, at 42 733 369 and 42 737 147 Hz. Relative to this reference, the Pt resonance of a 0.496 M aqueous solution of K_2PtCl_4 appears at +2,893 ppm. ^{c} For P^b, $\delta = 19.2$, $^1J_{PtP} = 2178$ Hz, and $^2J_{PtP} = 12$ Hz.

between Pt^1 and Pt^2. (4) Complex **1a** participates in atom-transfer reactions under photolysis, such as the photocatalytic formation of acetone and hydrogen from 2-propanol or the formation of Pt_2I (**1d**) and Pt_2I_2 (**1g**) complexes from **1a** upon photoreaction with methyl iodide. (5) The Pt_2Cl (**1b**) complex emits a red-orange luminescence at 696 nm in an alcohol matrix at 77 K upon excitation at 410 nm. We have also confirmed that **1a** reacts with diphenyl acetylene under photolysis to give a μ-η^1,η^3-butadienediyl ligand through a unique C–C coupling reaction, and that complexes **1d** and **1e–g** also luminesce in a low-temperature matrix upon photoexcitation.[3]

We now report an analysis of the ^{31}P and ^{195}Pt NMR spectra of these complexes. These data have considerable diagnostic value for determining the formation of diplatinum complexes of this structural type and may permit a better understanding of the unusual electronic structure of these molecules.

The relevant ^{31}P and ^{195}Pt NMR data are presented in Table I along with a description of the experimental conditions.[4] Spectral data for compounds **1a**, **1b**, **1d**, and **1e**, were analyzed by using PANIC (parameter adjustment in NMR by iteration calculation) for all isotopomers to ensure proper spectral interpretation and to verify the nominal effect of second-order coupling. This analysis also indicates that $^3J_{PP}$ coupling is small and can be neglected, presumably because the Pt^2–L^a and the two Pt^1–L^c bond vectors lie in orthogonal planes.

Important aspects of the ^{31}P NMR data are as follows: (1) While the values of $^1J_{PtP}$ coupling between the platinum atoms and the L^b or $L^{c,c}$ ligands have typical values of 2162–2311 Hz,[5] $^1J_{PtP}$ coupling to the unique L^a ligands is unusually large (from 4945 to 5672 Hz) and correlates well with the shorter Pt–P^a distances observed in compounds **1a**, **1b**, **1d**, and **1f**. In these complexes, Pt^2–P^a distances range from 2.173 (4) to 2.228 (8) Å, while all other Pt–P distances fall within the range of 2.305 (6)–2.368 (6) Å. A sensitivity of $^1J_{PtP}$ values to Pt–P distance has been noted in other systems,[6] and $^1J_{PtP}$ coupling constants larger than 5000 Hz are known for Pt(II) complexes.[7] Dinuclear Pt(I) compounds of the type $Pt_2(\mu\text{-}CO)(PPh_3)_3X_2$, where X is Cl or Br, as reported by Goodfellow et al., have structures very similar to **1** and exhibit $^1J_{PtP}$ coupling to a related P^a nucleus of 5350 or 5760 Hz.[8] Theoretical calculations indicate some degree of mixed-valence character to the Pt–Pt bond [of distance 2.654 (1) Å] in these complexes. A $Pt^1 \rightarrow Pt^2$ formal Lewis acid–base interaction has also been proposed to rationalize the presence of a Pt–Pt bond in compounds **1**.[2] (2) The strong bonding between Pt^2 and P^a and the nearly trans orientation of Pt^1 and P^a about Pt^2 leads to large $^2J_{PtP}$ coupling between Pt^1 and P^a of 376–614 Hz. The nearly cis orientations of Pt^1 and P^b or Pt^2 and $P^{c,c}$ about their respective central platinum atoms give more typical smaller values for $^2J_{PtP}$ coupling of 12–53 Hz.[5]

Important aspects of the ^{195}Pt NMR data are as follows. (1) The observed platinum chemical shift values of −1860 to −2267 ppm occur at very low frequency and are very similar to the chemical shift values observed for platinum nuclei in PtL_2 complexes, where L is P(i-Pr)$_3$, P(t-Bu)$_3$, or PCy_3.[9] These values are consistent with the computed high degree of localization of negative charge on the platinum atoms of **1a**; however, the magnitude of the paramagnetic term of the nuclear screening constant of these compounds has not been determined. The relatively small energy difference between the HOMO and LUMO of **1a** (of 0.36 eV) might contribute to the significance of this paramagnetic effect.[1a] (2) For the Pt_2X or Pt_2X_2 series, the δ(Pt) values for both Pt^1 and Pt^2 shift to lower field as X is changed from I to Br to Cl. This shift of δ(Pt) to lower field with an increase in the electronegativity of halide ligands has been observed in other complexes.[10] As expected, in the Pt_2X compounds, the shift of δ(Pt) is greater for Pt^2 (161 ppm) than it is for Pt^1 (61 ppm). In the Pt_2X_2 compounds, the corresponding δ(Pt) shifts for Pt^1 and Pt^2 are, respectively, 372 and 133 ppm. In **1a**, the chemical shift of Pt^2 appears at the highest field for this series of compounds due presumably to the strong σ-donor strength of the P^bEt_3 ligand relative to that of a halide ligand. Also, the resonance for the pseudopentacoordinate Pt^1 metal center lies consistently upfield of that of the pseudotetracoordinate Pt^2 nucleus, except for compound **1e** where the two ^{195}Pt chemical shifts are essentially the same. (3) Values of $^1J_{PtPt}$ range from 567 to 1604 Hz and could possibly be interpreted as either $^1J_{PtPt}$ or $^2J_{PtPt}$ coupling.[11] While these values do not strictly correlate with either Pt–Pt distance or the electronegativity of the Z substituents, within each Pt_2X or Pt_2X_2 series of compounds, the value of $^1J_{PtPt}$ increases with an increase in the electronegativity of X. This trend parallels the prediction of a reduction in the Pt–Pt repulsive interaction as the electronegativity of the ancillary ligands is increased.[1a] In addition, a Pt(II) dinuclear complex, $[Pt_2H_2Cl(\mu\text{-}dppm)_2]PF_6$, which also presumably has a "T-frame" structure and a formal $Pt \rightarrow Pt$ donor–acceptor bond similar to that of **1a**, has a recorded a $^1J_{PtPt}$ coupling constant of 1320 Hz and δ(Pt) values of −199

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(3) Unpublished results.

(4) For a discussion of the choice of reference for ^{195}Pt NMR spectra, see: Kidd, R. G.; Goodfellow, R. J. In *NMR and the Periodic Table*; Harris, R. K., Mann, B. E., Eds.; Academic Press: London, 1978; Chapter 8.

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and −525 ppm.[12] The metal–metal interactions in these related molecules might be similar.

For this class of dinuclear compound, the ^{195}Pt NMR data is most diagnostic of this structural type. However, the rather large $^{1}J_{\mathrm{PtP}}$ and $^{2}J_{\mathrm{PtP}}$ couplings to P^{a} are also diagnostically important parameters that are readily evident in the ^{31}P NMR spectra.

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Crystal Structure of Pyrazinium Chlorochromate

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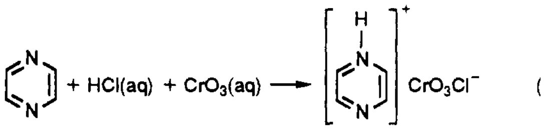
The preparation, reactions, and structures of high-valent chromium(VI) complexes continue to attract the attention of chemists who are interested in their ability to function as effective and selective oxidants. In previous studies, we and others have found chromium(VI) heterocyclic complexes to be effective and selective oxidants with a number of primary and secondary alcohols.[1-5] In two recent reports, a bond length correlation for $(\mathrm{Cr}_n\mathrm{O}_{3n+1})^{2-}$ anions was presented[6] and the existence of some short C–H···O contacts between the dichromate ion and a cation were observed.[7]

In our attempts to study effective and selective organic oxidants, pyrazinium chlorochromate was prepared via the reaction[4]


(1)

A stable nonhygroscopic solid, with a shelf life of at least 3 months when stored in the dark,[4] pyrazinium chlorochromate was found to be an efficient but mild oxidant for a number of alcohols.[4] Spectroscopic and solubility characteristics were supportive[4] of an ionic structure. This note reports the results of a single-crystal X-ray structural analysis. A further exploration of short C–H···O interactions in chromate salts with organic cations will be given.

Experimental Section

The pyrazinium chlorochromate crystals were prepared according to the literature method.[4] An irregularly shaped orange crystal, 0.30 × 0.60 × 0.15 mm, was mounted on a Syntex P2$_1$ diffractometer with graphite monochromated Mo Kα radiation (λ = 0.71069 Å), upgraded to Nicolet P3F specifications. Cell constants were defined by a least-squares refinement from 24 reflections in the range 31° < 2θ < 40°.[8] A total of 749 reflections were measured by ω scans (3 ≤ 2θ ≤ 70°, ((sin θ)/λ)$_{\max}$ = 0.807 Å, 0 ≤ h ≤ 18,0 ≤ k ≤ 9,0 ≤ l ≤ 7), with 749 unique and 712 observed ($F \geq 3\sigma(F)$) reflections. Empirical (ψ-scan) absorption corrections assuming an ellipsoidal crystal and Lorentz and polarization corrections were made.

Table I. Crystallographic Data for Pyrazinium Chlorochromate

formula: $\mathrm{C_4H_4N_2HCrO_3Cl}$
a = 11.615 (3) Å
b = 5.658 (1) Å
c = 5.657 (2) Å
V = 371.7 Å^3
Z = 2
fw = 216.3
space group: *Pma*2 (No. 28)
T = 22 °C
λ = 0.71069 Å
ρ_{calcd} = 1.93 g cm^{-3}
μ = 18.2 cm^{-1}
transm coeff = 0.724–0.940
$R(F_o)$ = 0.022 (= 0.024, for all reflcns)
$R_w(F_o)$ = 0.030 (= 0.030, for all reflcns)

Table II. Non-Hydrogen Atomic Coordinates (×10^4) and Isotropic Thermal Parameters (10^3 × Å^2) for $\mathrm{C_4H_4N_2HCrO_3Cl}$

atom	x	y	z	U_{iso}[a]
Cr	7500	−2157 (1)	0	21 (1)
Cl	7500	−1798 (2)	−3856 (2)	34 (1)
O(1)	6345 (2)	−876 (4)	919 (4)	36 (1)
O(2)	7500	−4918 (4)	661 (5)	34 (1)
N(1)	5000	5000	8031 (7)	25 (1)
N(2)	5000	5000	3188 (7)	29 (1)
C(1)	5536 (2)	3316 (4)	6800 (4)	28 (1)
C(2)	5549 (2)	3290 (4)	4347 (4)	29 (1)

[a] Equivalent isotropic U defined as one-third of the trace of the orthogonalized U_{ij} tensor.

Table III. Non-Hydrogen Interatomic Distances (Å) and Angles (deg) for $\mathrm{C_4H_4N_2HCrO_3Cl}$

Distances			
Cr–O(1)	1.611 (2)	C(1)–C(2)	1.387 (4)
Cr–O(2)	1.606 (2)	N(1)–N(2c)[c]	2.918
Cr–Cl	2.191 (1)	C(2)–O(1)	3.189
N(1)–C(1)	1.335 (3)	C(2)–O(1d)[d]	3.235
N(2)–C(2)	1.332 (3)	C(2)–O(2e)[e]	3.242
Angles			
O(1)–Cr–O(2)	111.2 (1)	C(1)–N(1)–C(1b)[b]	117.0 (4)
O(1)–Cr–Cl	106.2 (1)	C(2)–N(2)–C(2b)[b]	121.0 (4)
O(2)–Cr–Cl	108.8 (1)	N(1)–C(1)–C(2)	112.3 (2)
O(1)–Cr–O(1a)[a]	112.8 (1)	N(2)–C(2)–C(1)	118.7 (2)

[a] Denotes atom whose coordinates were transformed by 1.5 − x, y, z.
[b] Denotes atom whose coordinates were transformed by 1 − x, 1 − y, z.
[c] Denotes atom whose coordinates were transformed by x, y, 1 + z.
[d] Denotes atom whose coordinates were transformed by 1 − x, −y, z.
[e] Denotes atom whose coordinates were transformed by x, 1 + y, z.

Structure solution programs were obtained from the SHELXTL package.[9] Scattering factors were obtained from ref 10. Systematic absences ($h0l$, h odd) indicate *Pma*2, *Pmam*, and *P*2$_1$*am* as possible space groups. Since the centrosymmetric alternative (*Pmam*) requires the chromium atom to be at a site of C_{2v} or C_{2h} symmetry, an impossible situation since the anion has no 2-fold axis, only the two noncentrosymmetric alternatives were considered. All atom positions, including H atoms, were identified from direct methods and subsequent electron density difference maps when the space group *Pma*2 was tried. Final refinement of 56 least-squares parameters gave the $R(F_o)$ values listed in Table I. The left-hand alternative for the coordinate system was rejected on the basis of a higher $R_w(F_o)$ = 0.033 for the 3σ data set. Excursions in the final electron density difference map ranged from −0.3 to +0.3 e/Å^3 (near Cl). The two possible alternatives of interchanging carbon with nitrogen atoms in the ring were rejected on the basis of higher $R_w(F_o)$; $R_w(F_o)$ = 0.053 and $R_w(F_o)$ = 0.052, respectively, for the

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