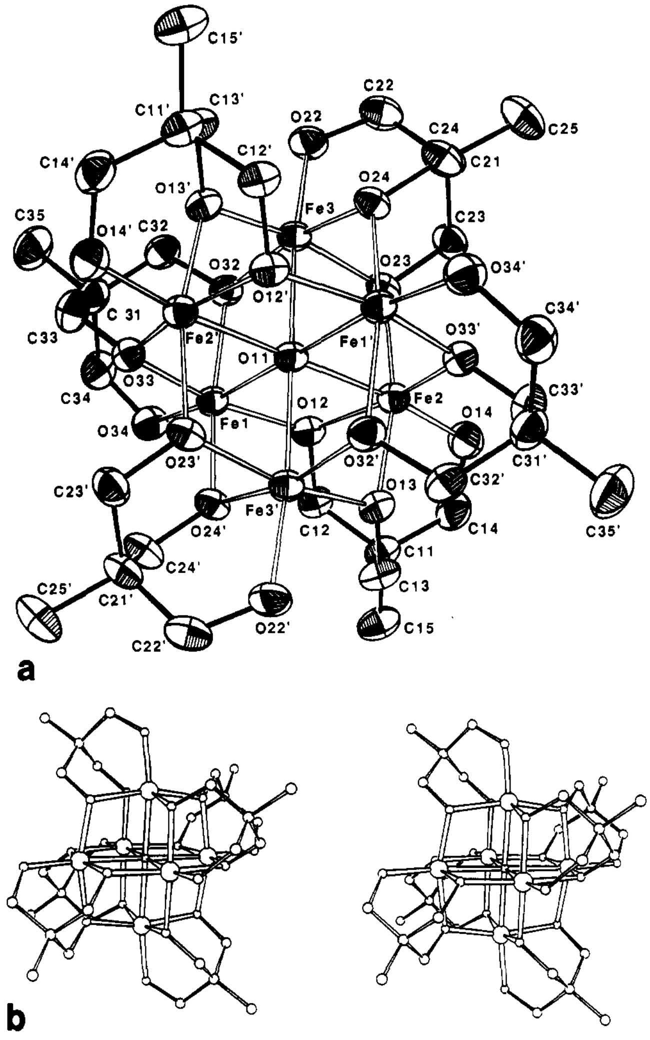

Figure 1. (a) ORTEP drawing of $O[Fe(OCH_2)_3CCH_3]_6^{2-}$ with numbering scheme and vibrational ellipsoides at the 50% probability level. (b) Stereoview of the compound.

Hence it must be taken into account that the crystal isolated does not represent the major solute component.

In the centrosymmetric $O[Fe(OCH_2)_3CCH_3]_6^{2-}$ complex, a central O(-II) is octahedrally surrounded by six Fe(III) atoms. This OFe_6 aggregate is encapsulated by a hydrophobic shell of six fully deprotonated, facially coordinated $CH_3C(CH_2O)_3^{3-}$ entities, and the Fe(III) atoms are located each in the center of a distorted octahedron of six oxygen atoms, forming a Fe_6O_{19} unit as shown in Figure 1. The 19 oxygen atoms are of three types—central O, bridging RO, and terminal RO. The structure of this Fe_6O_{19} core is closely related to the polyoxoanions $M_6O_{19}^{2-}$ of the early transition metals (Table V).[15] Considering the similar ionic radii of Fe(III), Mo(VI) and W(VI),[16] this analogy is quite understandable. However, the lower charge of Fe(III) requires the presence of alkoxo-groups instead of O(-II) as bridging and terminal ligands. The long bond distance between the Fe atoms and the central oxygen atom (Table III) is remarkable and does not fit in the common range of Fe-O bond lengths. However, elongated Fe-O bonds are formed, when the coordination number of the central oxygen atom increases from 2 to 4 as demonstrated in the sequence μ_2-O, 1.79 Å (in $Fe_2O(O_2CR)_2L_2$);[7a] μ_3-O, 1.92 Å (in $Fe_3O(O_2CR)_6L_3$);[17] and μ_4-O, 2.03 Å (in $Fe_{16}CoO_{10}(OH)_{10}(O_2CPh)_{20}$.[5b] Hence, the observed average value of 2.25 Å for CN = 6 seemingly extends this series.

The formation of a polynuclear complex, starting with a Fe:L ratio of 1:6, is surprising and demonstrates the high nucleophility of alkoxo groups. Consequently, the formation of mononuclear complexes requires alkoxides with lower basicity. For perfluoropinacole (p*K* = 5.95), the formation of the mononuclear tris chelate in water has been reported,[18] and recent investigations revealed mononuclear Fe(III) complexes with substituted cyclohexanetriols (p*K* = 8.14)[19] in aqueous solution.[20]

Acknowledgment. We thank Dr. I. Erni, Dr. N. Oswald, and Dr. V. Gramlich for helpful advice. This work was supported by the ETHZ, Kredite für Unterricht and Forschung.

Supplementary Material Available: Tables SI-SV, listing crystallographic data, anisotropic displacement parameters of non-hydrogen atoms, positional parameters of hydrogen atoms, and bond distances and angles and a stereoview of the unit cell (13 pages); a table of calculated and observed structure factors (16 pages). Ordering information is given on any current masthead page.

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Chelate Binding Preferences in $CpML_4$ Complexes: Preparation and Structure of *trans*-(η^5-C_5H_5)$Mo[(C_6F_5)_2PCH_2CH_2P(C_6F_5)_2](CO)Cl$

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Introduction

Complexes of the general formula $CpML_n$ are ubiquitous to organometallic chemistry and constitute an important class of "coordinatively compact" systems intermediate between metallocene (Cp_2ML_x) and classic L_nM coordination compounds. $CpML_4$ complexes in particular may be considered to be electronically seven-coordinate as well as sterically pseudo-five-coordinate, and have been the subject of numerous theoretical,[1] structural,[1-3] and dynamic[4] studies. Although $CpML_4$ coordination

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Table I. Summary of Crystallographic Data for *trans*-$(C_5H_5)Mo(dfppe)(Cl)(CO)$

$C_{32}H_9F_{20}ClMoOP_2$	fw = 982.7
a = 17.561 (4) Å	space group: $A2/a$
b = 15.264 (3) Å	T = 293 K
c = 26.456 (5) Å	V = 6575 (2) Å^3
β = 112.01 (2)°	Z = 8
ρ_{calc} = 1.99 g/cm^3	μ = 0.707 mm^{-1}
$R(F_o)$ = 3.20%	$R_w(F_o)$ = 3.97%
trans coeff = 0.534–0.589	λ = 0.71073 Å

Table II. Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Coefficients (Å^2 $\times$ 10^3)

	x	y	z	U(eq)[a]
Mo(1)	1884 (1)	5270 (1)	7472 (1)	36 (1)
P(1)	1078 (1)	4205 (1)	6824 (1)	35 (1)
P(2)	1322 (1)	4644 (1)	8106 (1)	36 (1)
Cl(1)	2639 (1)	3853 (1)	7787 (1)	51 (1)
O(1)	140 (3)	6020 (3)	7134 (2)	69 (2)
F(1)	68 (2)	2792 (2)	6054 (1)	62 (1)
F(2)	421 (3)	1811 (3)	5338 (2)	92 (2)
F(3)	1913 (3)	1927 (3)	5262 (2)	99 (2)
F(4)	3067 (3)	3025 (3)	5931 (2)	91 (2)
F(5)	2734 (2)	4016 (3)	6641 (1)	68 (2)
F(6)	−868 (2)	4130 (2)	6526 (1)	64 (1)
F(7)	−2026 (2)	4955 (3)	5723 (1)	77 (2)
F(8)	−1624 (2)	5995 (3)	5027 (1)	78 (2)
F(9)	−29 (2)	6166 (2)	5140 (1)	76 (2)
F(10)	1147 (2)	5293 (2)	5929 (1)	60 (1)
F(11)	202 (2)	3975 (2)	8679 (1)	61 (2)
F(12)	−710 (2)	4860 (3)	9110 (2)	86 (2)
F(13)	−688 (2)	6641 (3)	9124 (2)	85 (2)
F(14)	264 (3)	7518 (2)	8700 (2)	90 (2)
F(15)	1206 (2)	6654 (2)	8283 (2)	73 (2)
F(16)	1577 (2)	2611 (2)	8404 (1)	69 (2)
F(17)	2643 (3)	1896 (3)	9307 (2)	104 (2)
F(18)	3644 (2)	2914 (3)	10122 (2)	108 (2)
F(19)	3566 (2)	4687 (3)	10015 (2)	102 (2)
F(20)	2505 (2)	5422 (2)	9101 (1)	70 (2)
C(1)	759 (4)	5744 (4)	7247 (2)	48 (2)
C(2)	695 (3)	3427 (3)	7216 (2)	39 (2)
C(3)	522 (3)	3867 (3)	7687 (2)	41 (2)
C(4)	2969 (5)	6196 (5)	7942 (3)	77 (3)
C(5)	2281 (5)	6699 (4)	7632 (3)	75 (4)
C(6)	2122 (4)	6519 (4)	7075 (3)	65 (3)
C(7)	2697 (4)	5928 (4)	7048 (3)	58 (3)
C(8)	3222 (4)	5722 (5)	7576 (3)	75 (3)
C(12)	832 (4)	2852 (4)	6049 (2)	50 (2)
C(13)	992 (4)	2337 (4)	5677 (2)	61 (3)
C(14)	1748 (5)	2392 (4)	5639 (3)	67 (3)
C(15)	2339 (4)	2953 (4)	5973 (3)	63 (3)
C(16)	2144 (4)	3462 (4)	6340 (2)	50 (2)
C(11)	1395 (3)	3440 (3)	6393 (2)	41 (2)
C(22)	−626 (3)	4611 (3)	6189 (2)	45 (2)
C(23)	−1240 (3)	5043 (4)	5774 (2)	54 (2)
C(24)	−1038 (3)	5561 (4)	5425 (2)	55 (2)
C(25)	−234 (4)	5647 (4)	5483 (2)	54 (2)
C(26)	355 (3)	5202 (4)	5891 (2)	45 (2)
C(21)	188 (3)	4667 (3)	6260 (2)	37 (2)
C(32)	227 (3)	4862 (3)	8666 (2)	47 (2)
C(33)	−242 (3)	5303 (5)	8896 (2)	56 (3)
C(34)	−234 (3)	6197 (5)	8903 (2)	59 (3)
C(35)	255 (4)	6633 (4)	8690 (3)	61 (3)
C(36)	719 (3)	6177 (4)	8460 (2)	52 (3)
C(31)	719 (3)	5271 (4)	8433 (2)	42 (2)
C(42)	2048 (3)	3154 (4)	8791 (2)	50 (2)
C(43)	2599 (4)	2775 (5)	9254 (3)	65 (3)
C(44)	3106 (4)	3276 (6)	9665 (3)	74 (3)
C(45)	3071 (3)	4172 (6)	9616 (2)	67 (3)
C(46)	2520 (3)	4544 (4)	9145 (2)	53 (3)
C(41)	1991 (3)	4057 (3)	8724 (2)	40 (2)

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

geometries based on a Cp-capped trigonal bipyramid are conceptually reasonable, all structurally characterized examples are best approximated by a Cp-capped square-pyramidal or "four-legged piano-stool" geometry with L–M–L angles ranging from 72 to 87° (cis) and from 101 to 143° (trans or diagonal). For unsymmetrically substituted complexes $CpML_2(X)(Y)$ where the possibility of cis and trans isomers is present, virtually all examples of chelate complexes $CpM(L\frown L)(X)(Y)$ are reported to have cis stereochemistry.[3,4b,c] This is not surprising since the natural bite angles of $L(B)_nL$ (B = bridging link, n = 1,2) chelates fall within the range of *cis*-L–M–L bond angles commonly observed. Probable exceptions to this generalization are $[(\eta^6\text{-}C_6H_5R)Mo(PMe_3)(dmpe)R']^+$ (R = H, Me; R′ = H, Me, Et)[5] and the dihydrides $(\eta^6\text{-}C_6H_6)Mo(dmpe)H_2$[6] and $[CpRu(R_2PCH_2CH_2PR_2)(H)_2]^+$ (R = Me, Ph),[7] which exhibit triplet hydride resonances consistent with a trans piano-stool geometry. However, since NMR cannot readily distinguish between a static symmetrical ground state and a sufficiently low energy site exchange process,[4] these assignments are ambiguous in the absence of definitive structural data.[8,9]

We have been interested in developing the coordination chemistry of (fluoroalkyl)phosphine chelate Mo(II) systems of the general formula $CpMo(P\frown P)(L)X$ as a logical extension to our studies of electron-poor Mo(0) complexes incorporating sterically-demanding fluoroalkylphosphine ligands.[10] As part of this work we report here the preparation and crystal structure of *trans*-CpMo(dfppe)(CO)Cl (dfppe = $(C_6F_5)_2PCH_2CH_2P(C_6F_5)_2$). The unusual trans stereochemical preference for this molecule is in direct contrast to the established cis conformation of the nonfluorinated parent molecule, CpMo(dppe)(CO)Cl,[3a,b] and thus affords a useful comparison between these two prototypical piano-stool geometries.

Experimental Section and Results

General Data. All manipulations were conducted under an atmosphere of nitrogen by using Schlenk, high-vacuum-line, and/or glovebox techniques. The synthesis of compound **1** follows the method of Curtis.[11] Reactions were carried out under a reduced ambient pressure of approximately 590 Torr (approximately 7200 ft. elevation). Dry, oxygen-free solvents were vacuum distilled prior to use. Elemental analyses were performed by Desert Analytics. Infrared spectra were recorded on a Mattson Cygnus 100 FTIR instrument. ^1H and ^{31}P NMR spectra were

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Table III. Selected Bond Distances (Å) and Angles (deg) for *trans*-(η^5-C_5H_5)Mo(dfppe)(CO)Cl[a]

Bond Distances			
Mo(1)–P(1)	2.402 (1)	Mo(1)–P(2)	2.437 (2)
Mo(1)–Cl(1)	2.510 (2)	Mo(1)–C(1)	1.975 (7)
Mo(1)–C(4)	2.324 (7)	Mo(1)–C(5)	2.281 (7)
Mo(1)–C(6)	2.288 (7)	Mo(1)–C(7)	2.347 (8)
Mo(1)–C(8)	2.363 (7)	P(1)–C(2)	1.861 (6)
P(1)–C(11)	1.857 (6)	P(1)–C(21)	1.848 (4)
P(2)–C(3)	1.854 (5)	P(2)–C(31)	1.862 (6)
P(2)–C(41)	1.849 (5)	O(1)–C(1)	1.098 (8)
C(2)–C(3)	1.541 (8)	C(4)–C(5)	1.407 (10)
C(5)–C(6)	1.419 (11)	C(6)–C(7)	1.376 (10)
C(7)–C(8)	1.392 (9)	C-F range	1.328 (10)–1.363 (7) (average = 1.344)

Bond Angles			
P(1)–Mo(1)–P(2)	86.8 (1)	P(1)–Mo(1)–Cl(1)	75.6 (1)
P(2)–Mo(1)–Cl(1)	74.5 (1)	P(1)–Mo(1)–C(1)	77.1 (2)
P(2)–Mo(1)–C(1)	74.6 (2)	Cl(1)–Mo(1)–C(1)	139.5 (2)
P(1)–Mo(1)–CNT	134.2	P(2)–Mo(1)–CNT	139.0
Cl(1)–Mo(1)–CNT	110.5	C(1)–Mo(1)–CNT	110.0
Mo(1)–C(1)–O(1)	178.1 (5)	Mo(1)–P(1)–C(2)	105.8 (1)
Mo(1)–P(1)–C(11)	129.2 (2)	Mo(1)–P(1)–C(21)	114.3 (2)
Mo(1)–P(2)–C(3)	104.5 (2)	Mo(1)–P(2)–C(31)	124.4 (2)
Mo(1)–P(2)–C(41)	120.8 (2)		

[a] CNT = centroid of toluene ring.

obtained with a JEOL JNM-FX270 spectrometer at 269.7 and 109.1 MHz, respectively. ^{31}P spectra were referenced to a 85% H_3PO_4 external standard. $CpMo(CO)_3Cl$[12] and $(C_6F_5)_2PCH_2CH_2P(C_6F_5)_2$[10a] were prepared by following literature procedures.

***trans*-(η^5-C_5H_5)Mo[$(C_6F_5)_2PCH_2CH_2P(C_6F_5)_2$](CO)Cl (1).** A solution of $CpMo(CO)_3Cl$ (1.57 g, 5.60 mmol) in 65 mL of warm (ca. 50 °C) toluene was added dropwise to a refluxing solution of dfppe (3.98 g, 5.25 mmol) in 80 mL of toluene over a period of 5 h. After the addition was complete, the reaction mixture was refluxed an additional 1.5 h and then cooled and filtered to remove a brown solid. Toluene was removed from the filtrate and the residue was triturated with diethyl ether. Filtration, washing several times with ether, and drying under vacuum afforded 3.22 g (62%) of a blue microcrystalline product. Anal. Calcd for $C_{32}H_9F_{20}ClMoOP_2$: C, 39.11; H, 0.92. Found: C, 39.04; H, 0.78. IR (Nujol, cm^{-1}): 1880 vs, 1641 m, 1522 s, 1293 m, 1094 s, 977 s. ^1H NMR (benzene-d_6, 23 °C): δ 5.34 (s, 5 H; C_5H_5), 3.15 (m, 2 H; PCH_2), 2.70 (m, 2 H; PCH_2). ^{31}P NMR (toluene-d_8, 25 °C): δ 29.2 (s).

Crystal Structure Determination. A suitable crystal (blue prism, 0.19 × 0.31 × 0.42 mm) was grown by slow diffusion of hexane into a saturated toluene solution of 1 at ambient temperature and mounted in a sealed glass capillary under nitrogen. Crystallographic data were collected at 293 K on a Nicolet R3m/V diffractometer with a molybdenum tube (λ(Mo Kα) = 0.71073 Å) and a graphite monochromator. Lattice parameters were determined from 31 strong reflections with $15 \leq 2\theta \leq 25°$. Crystal data are summarized in Table I. A total of 4571 unique reflections were collected ($R_{int} = 0.022$) between $4 \leq 2\theta \leq 46°$ with indices $+h,+k,\pm l$ by using an Ω scan mode. An empirical φ-scan absorption correction was applied by using 12 reflections with $5 \leq 2\theta \leq 38°$. The structure was solved by using Patterson techniques. Full-matrix least-squares refinement of anisotropic non-hydrogen atoms with ring and ligand backbone hydrogens riding in idealized positions gave an R value of $0.032(F_o)$ ($R_w(F_o) = 0.040$) for 3111 reflections with $I > 3\sigma(I)$. Atomic parameters are given in Table II, and selected bond distances and angles are given in Table III. All computations used the SHELXTL PLUS (Version 3.4) program library (Nicolet Corp., Madison, WI).

Discussion

CpMo(dfppe)(CO)Cl exhibits a single carbonyl band in the infrared at 1880 cm^{-1}, slightly higher in energy than values reported for the more electron-rich systems $CpMo(P(OMe)_2Ph)_2(CO)Cl$ (1845 cm^{-1}), CpMo(dppe)(CO)Cl (1845 cm^{-1}),[11] and $CpMo(PPh_2Me)_2(CO)Cl$ (1810 cm^{-1}).[13] In contrast to *cis*-CpMo(dppe)(CO)Cl, which exhibits mutually coupled phosphorus resonances ($^2J_{PP}$ = 38 Hz) at 67.9 and 93.3 ppm at 20 °C in the ^{31}P NMR consistent with a static cis stereochemistry, only a single uncoupled ^{31}P resonance at 29.2 ppm is observed for 1 at temperatures down to −75 °C. Since fluxional *cis*-CpM($L\frown L$)(X)(Y) chelate complexes are well-known[4b,c] and *cis*-$CpMo(PR_3)_2(CO)Cl$ complexes in particular are known to be highly fluxional,[13] this result does not rigorously exclude a dynamic cis ground state structure for 1. The crystal structure of 1 was carried out to settle this issue.

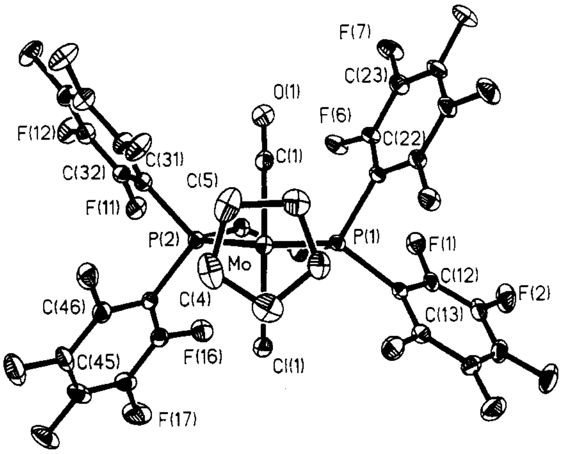

Figure 1. ORTEP view of *trans*-(η^5-C_5H_5)Mo(dfppe)(CO)Cl with the atom-labeling scheme.

Table IV. Structural Comparisons for *trans*-$CpM(PR_3)_2(X)(Y)$ Complexes

compound	P–M–P, deg	X–M–Y, deg	PR_3 cone angle,[a] deg
$CpMo[P(OMe)_3]_2(CC(CN)_2)Cl$[2m]	143.4	114.6	107
$[CpMo[P(OMe)_3]_2(CCH_2{}^tBu)H^+]$[2k]	121.9	106	107
$[CpMo(PMe_3)_2(CO)_2{}^+]$[2n]	132.3	107	118
$CpMo(PPh_3)_2(NCO)(CO)$[2d]	135.2	112.2	145
$CpRe(PPh_3)_2(H)_2$[2j]	108.6	138	145
$CpRe(dmpe)(H)_2$[8]	84.6		107
CpMo(dfppe)(CO)Cl	86.8	139.5	151

[a] Values taken from ref 15.

As shown in Figure 1, CpMo(dfppe)(CO)Cl adopts a pseudo-trans chelate coordination mode with approximate mirror symmetry relating the phosphorus centers and bisecting the cyclopentadienyl group. The closest intermolecular contacts are between fluorine atoms F(3) and F(20) (2.807 Å) and between F(4) and F(14) (2.829 Å). The carbonyl group lies beneath a Cp C–C bond, a feature common to this class of molecules.[2,14] The Cp ring carbons are planar to within 0.001 Å with a 2.7° ring tilt toward the carbonyl ligand. Mo–C(ring) bond distances average 2.321 (7) Å, within the normal observed range of 2.30–2.37 Å. The Mo–Cl bond length, 2.510 (2) Å, is likewise typical for seven-coordinate Mo(II) complexes.

The Mo–P bonds of 1, 2.402 (1) and 2.437 (2) Å, differ by approximately 18σ for no obvious reason since they are essentially mirror related; solid-state chelate backbone and C_6F_5 conformational effects are most likely responsible for this observed difference. The average Mo–P distance of 2.420 Å is intermediate between longer bond distances reported for other arylphosphine $CpML_4$ complexes such as CpMo(dppe)(CO)Cl (2.496 (4), 2.439 (5) Å)[3b] and $CpMo(PPh_3)_2(CO)(NCO)$ (2.500 (3), 2.497 (2) Å)[2d] and the shorter Mo–P(phosphite) distances reported for *trans*-(η^5-C_5H_4Me)$Mo(CO)_2[P(OMe)_2]I$ (2.388 (8) Å)[2e] and *cis*-$CpMo[P(OMe)_3]_2(CO_2CF_3)(CF_3CO_2H)$ (2.403 (3), 2.413 (3) Å).[2k] Although the inductive effect of C_6F_5 substituents on phosphine acceptor ability is believed to surpass that of alkoxy groups,[10a] the fact that Mo–P distances for 1 are actually longer than comparable Mo–P(phosphite) values may be attributed to the considerably greater steric demand of the C_6F_5 units.[15]

The most significant structural parameters of 1 to compare with other crystallographically characterized *trans*-$CpML_2(X)(Y)$

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complexes are the *trans*-L–M–L and *trans*-X–M–Y angles. When L = CO, the L–M–L angles are all similar, 108 ± 3°, with extreme values being noted for CpMo$(CO)_3$(ZnBr) (100.9)[2l] and $[CpMn(CO)_2(PPh_3)(SnCl_3)]^+$ (115.1°).[2i] When L is a more sterically-demanding phosphine ligand, a wider range of values is found; some representative examples are given in Table IV. Because of the counteracting influences of the apical Cp group, which would favor small P–M–P angles, and the basal X and Y groups, which favor large P–M–P angles, no clear correlation of P–M–P angles with phosphorus cone angles is apparent. In the extreme where X = Y = H and basal steric conflicts are minimized, an unusually small P–M–P angle of 108.6° is in fact observed for *trans*-$CpRe(PPh_3)_2H_2$.[2j] This tendency is consistent with the previously noted preference for trans stereochemistry in hydride chelates, $CpM(\overset{\frown}{P\ P})(H)_2$.[7,8]

The trans coordination mode of **1** may be similarly attributed to steric interactions. All cis complexes $CpM(\overset{\frown}{L\ L})(X)(Y)$ previously reported in the literature incorporate relatively small chelating groups. To the best of our knowledge dppe is the largest chelate previously reported, with a cone angle θ of 125°, which is considerably less than that of dfppe (θ = 151°). Given identical X and Y basal groups, one would anticipate a greater driving force for trans coordination in **1** to minimize interactions between dfppe and the cyclopentadienyl group.

Establishing a steric "crossover" point between *cis*- and *trans*-$CpM(\overset{\frown}{L\ L})(X)(Y)$ coordination geometries would provide a useful predictive guideline in future $CpML_4$ studies. In comparing NMR spectroscopic data of **1** with *cis*-CpMo(dppe)(CO)Cl, we noted the presence of a minor cyclopentadienyl resonance (≈8%) in the ^1H NMR spectrum of CpMo(dppe)(CO)Cl at δ 5.13 along with a corresponding singlet in the ^{31}P NMR at δ 81.9 that is very close to the average of the phosphorus chemical shifts of the cis isomer. It seems probable that these resonances are due to *trans*-CpMo(dppe)(CO)Cl and that in this particular system the free energies of the cis and trans isomers are closely comparable.

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Supplementary Material Available: A table of complete data collection parameters and full listings of atomic distances and bond angles, anisotropic thermal parameters, and hydrogen atom coordinates and isotropic thermal parameters (9 pages); a listing of calculated and observed structure factors (17 pages). Ordering information is given on any current masthead page.

Additions and Corrections

1989, Volume 28

David P. Fairlie, W. Gregory Jackson,* and George M. McLaughlin: Cobalt-Induced Facile Degradation of Phenylurea to Ammonia, Carbon Dioxide, and Anilinium Ion and Other Reactions of Linkage Isomeric Cobalt(III) Complexes of Phenylurea.

Page 1983. In the abstract, the space group *C*2/*c* with *Z* = 8 is given incorrectly. It should read space group *Cc*, *Z* = 4.—W. Gregory Jackson

1990, Volume 29

Ludwig Bär, Helmut Englmeier, Günter Gliemann,* Ulrich Klement, and Klaus-Jürgen Range*: Luminescence at High Pressures and Magnetic Fields and the Structure of Single-Crystal Platinum(II) Binuclear Complexes $M_x[Pt_2(POP)_4]\cdot nH_2O$ (M_x = Ba_2, $[NH_4]_4$; POP = $P_2O_5H_2^{2-}$).

Pages 1162–1168. The supplementary material was omitted from the original paper. It has now been included, as indicated in the following paragraph.

Supplementary Material Available: Tables of atomic positional and isotropic thermal parameters, anisotropic thermal parameters, and bond lengths and angles (5 pages); listings of observed and calculated structure factors, the last refinement cycle, and difference Fourier syntheses (25 pages). Ordering information is given on any current masthead page. —Ulrich Klement