Table II. Half-Wave Potentials for Ruthenium(II) Complexes[a]

complex	R	oxidn	redn	
$Ru(4a)_2^{2+}$	H	+1.27 (140)	-1.21 (90)	-1.46 (100)
$Ru(4b)_2^{2+}$	CH_3	+1.26 (135)	-1.22 (85)	-1.46 (90)
$Ru(4c)_2^{2+}$	Cl	+1.26 (145)	-1.23 (110)	-1.48 (115)
$Ru(4d)_2^{2+}$	NMe_2	b	-1.25 (75)	-1.49 (85)
$Ru(4e)_2^{2+}$	NO_2	+1.28 (140)	-0.90 (185)[c]	
$Ru(5a)_2^{2+}$	H	+1.26 (170)	-1.15 (75)	-1.37 (80)
$Ru(5b)_2^{2+}$	CH_3	+1.24 (165)	-1.18 (160)	-1.42 (165)
$Ru(5c)_2^{2+}$	Cl	+1.29 (150)	-1.12 (110)	-1.34 (120)

[a] Potentials are given in volts vs SCE for 0.002 M DMSO solutions, 0.1 M in TBAP, recorded at 25 ± 1 °C. The difference between cathodic and anodic peak potentials (mV) is given in parentheses. [b] Oxidation of the NMe_2 group interferes with this process. [c] This value represents reduction of the NO_2 group, which obscured reduction of the metal center.

their bridged analogues and in the expected order $Ru(5c)_2^{2+} > Ru(5a)_2^{2+} > Ru(5b)_2^{2+}$ for both the first- and second-reduction couples. This same ordering is found for oxidation, where the influence of bridging appears to be less pronounced.

The results outlined above are consistent with our premise that the MLCT state for terpyridine complexes of Ru(II) has separate components associated with the central and distal pyridine rings of the ligand. As we change the electron-withdrawing ability of the para substituent on a 4′-phenyl ring, the energy of absorption is affected only to a small degree, while the effectiveness (intensity) of MLCT varies by a factor of about 1.5 between the extremes of **4d** and **4e**. As resonance interaction of the 4′-phenyl ring is improved by elimination of the 3,3′:5′,3″-bridges, absorption energy again remains relatively invariant, but the absorption envelope widens slightly toward longer wavelength.

Although the introduction of 4- and 4″-substituents onto terpyridine is synthetically more challenging, we are working along these lines to examine the influence of substituents on the distal rings of these complexes.

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An Update of Pseudoplanar, Bibridged $Cu_nX_{2n+2}^{2-}$, $Cu_nX_{2n+1}L^-$, and $Cu_nX_{2n}L_2$ Oligomer Stacking Patterns: Structure of 1,2-Dimethylpyridinium Bis(μ-chloro)trichloroaquadicuprate(II)

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Many copper(II) halide compounds occur as pseudoplanar, bibridged oligomeric species with formula $Cu_nX_{2n+2}^{2-}$, $Cu_nX_{2n+1}L^-$, or $Cu_nX_{2n}L_2$ ($X = Cl^-$, Br^-; L = neutral ligand). Within the oligomer the copper ions are four-coordinate and approximately planar with adjacent Cu ions linked by two bridging ligands. The Cu(II) ion frequently completes its coordintion sphere by forming longer, axial (semicoordinate) bonds to halide ions in neighboring, coplanar oligomers, thus forming stacks. Semicoordinate bond formation between oligomers requires a relative displacement of neighboring oligomers that varies periodically along the stack. The surprising variety of stacking patterns was recently tabulated by Geiser and co-workers.[1] The purpose of this note is to update this table of known stacking patterns and to report the structure of an $ACu_2Cl_5(H_2O)$ compound exhibiting an unusual stacking pattern.

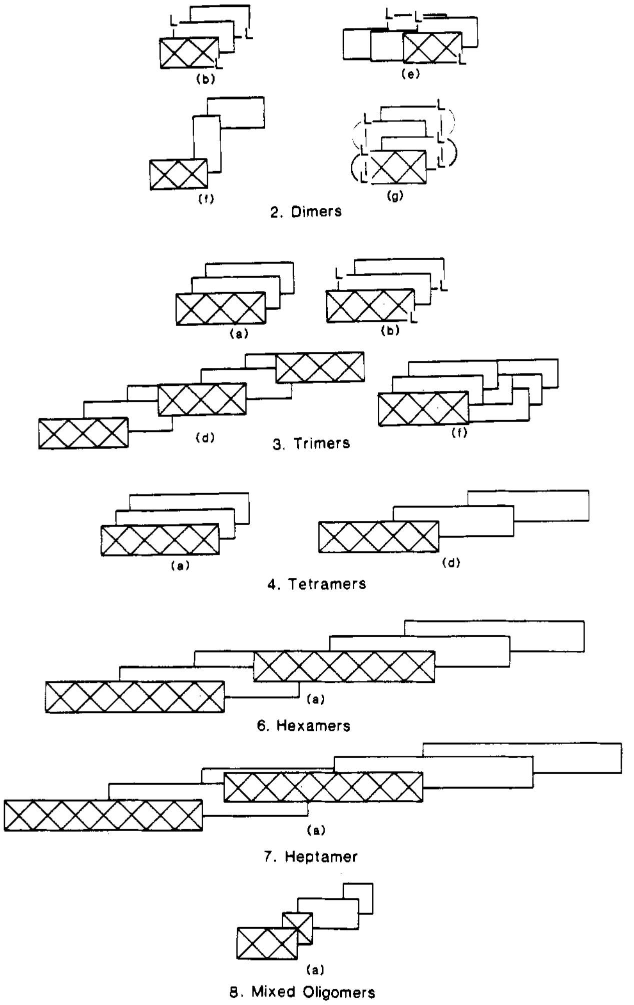

Figure 1. Envelope stacking pattern diagrams (cf. Table I).

Stacking Pattern Notation

Symbols for the various stacking patterns are devised by using the following procedure.[1] (1) The oligomer is idealized as a planar, rectangular envelope with the ligands located on the perimeter, the copper ions located in the interior, all copper–ligand distances equal, and all ligand–Cu–ligand angles either 90 or 180°. (2) The envelopes are stacked with displacements of neighboring envelopes mimicking observed oligomer displacements. (3) A stacking symbol of the form $n(m_\parallel, m_\perp, \phi)$ is constructed with the terms defined as follows: n, the number of copper ions in the oligomer; $m_\parallel d$ and $m_\perp d$, the translational displacement of the envelope taken parallel and perpendicular to the Cu–Cu axis (d = ligand–ligand distance along an edge); and ϕ, rotation of the envelope about the normal to the envelope plane. As many parenthetic symbols are concatenated as are necessary to describe the repeat unit of the stack.

It is necessary to add the following rules: (3a) $m_\parallel d$ and $m_\perp d$ are the displacements necessary to move the *central point* of one oligomer atop the *central point* of its neighbor; (4) the rotation axis is always taken through the central point; and (5) if an oligomer belongs to more than one stack, then the full symbol consists of a concatenation of the individual symbols (enclosed in brackets) for each possible stack preceded by n. Rule 3a guarantees that mixed oligomer stacking patterns will be unique, except for a uniform change of sign in $m_\parallel$ or $m_\perp$ (the value of n that most closely precedes the parenthetic symbol denotes the

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[1] Geiser, U.; Willett, R. D.; Lindbeck, M.; Emerson, K. *J. Am. Chem. Soc.* **1986**, *108*, 1173.

Table I. New Pseudoplanar, Bibridged $Cu_nX_{2n+2}^{2-}$, $Cu_nX_{2n+1}L^-$, and $Cu_nX_{2n}L_2$ Oligomer Stacking Patterns[a]

pattern	symbol	oligomer	compd	ref
		Dimers		
2b	$2(^1/_2, {}^1/_2, 180°)$	$Cu_2Cl_5(H_2O)^-$	$(C_6H_8N)Cu_2Cl_5(H_2O)$	8
			$(C_5H_9N_2O)Cu_2Cl_5(H_2O)$	8
2e	$(^1/_2, {}^1/_2, 180°)(-^3/_2, -^1/_2, 180°)$	$Cu_2Cl_5(H_2O)^-$	$(C_7H_{10}N)Cu_2Cl_5(H_2O)$	this work
2f	$2(1, 1, 90°)(1, 1, -90°)$	$Cu_2Cl_6^{2-}$	$[(CH_3)_2NH_2]CuCl_3$	12
			$[(C_6H_5)CH_2C_5H_9NH_2]CuCl_3$	13
			$(C_5H_{10}NH_2)CuCl_3$	11
		$Cu_2Br_6^{2-}$	$(C_5H_{10}NH_2)CuBr_3$	15
2g	$2(^1/_2, {}^1/_2, 180°)(-^1/_2, {}^1/_2, 180°)$	$C_{12}H_{12}N_2Cu_2Cl_4$	$C_{12}H_{12}N_2Cu_2Cl_4$	16
		Trimers		
3a	$3(^1/_2, {}^1/_2)$	$Cu_3Cl_8^{2-}$	$(C_6H_9N_2)_2Cu_3Cl_8$	3
		$Cu_3Br_8^{2-}$	$(C_6H_8N_2Br)_2Cu_3Br_8$	3
3b	$3(^1/_2, {}^1/_2, 180°)$	$Cu_3Cl_7(C_2H_5OH)^-$	$(C_9H_{14}N)Cu_3Cl_7(C_2H_5OH)$	3
3d	$3[(^3/_2, {}^1/_2)][(-^5/_2, -^1/_2)]$	$Cu_3Cl_6(CH_3CN)_2$	$Cu_3Cl_6(CH_3CN)_2$	10
3f	$3(^1/_2, {}^1/_2)^2(^1/_2, -^1/_2)^2$	$Cu_3Br_8^{2-}$	$(C_6H_8N)_2Cu_3Br_8$	4
		Tetramers		
4a	$4(^1/_2, {}^1/_2)$	$Cu_4Cl_{10}^{2-}$	$(C_6H_9N_2)_2Cu_4Cl_{10}$	14
4d	$4(^5/_2, {}^1/_2)$	$Cu_4Cl_{10}^{2-}$	$(C_6H_8N)_2Cu_4Cl_{10}$	4
		Hexamers		
6a	$6[(^5/_2, {}^1/_2)][(-^9/_2, -^1/_2)]$	$Cu_6Cl_{14}^{2-}$	$(C_7H_{10}N)_2Cu_6Cl_{14}$	4
		Heptamers		
7a	$7[(^7/_2, {}^1/_2)][(-^9/_2, -^1/_2)]$	$Cu_7Cl_{16}^{2-}$	$(C_7H_{10}N)_2Cu_7Cl_{16}$	4
		Mixed Oligomers		
8a	$2(1, {}^1/_2)1(1, {}^1/_2)$	$Cu_2Cl_4(C_6H_7NO)_2$; $CuCl_2(H_2O)_2$	$Cu_3Cl_6(C_6H_7NO)_2(H_2O)_2$	5

[a] The numeric and alphabetic labels for the stacking patterns in this table refer to their order of appearance in the comprehensive table in the supplementary material.

size of the oligomer being displaced). Adoption of this rule does not change the symbols for any of the uniform oligomer stacking patterns, except for the $2(^3/_2, {}^1/_2, 90°)(^3/_2, {}^1/_2, -90°)$ pattern (original notation) in which the rotation axis is not taken through the central point (cf. Figure 1, 2f). Application of rule 4 to this pattern, however, gives the same symbol in both new and original systems of notation, i.e. $2(1, 1, 90°)(1, 1, -90°)$. Rule 5 enables description of systems in which any oligomer forms semicoordinate bonds with more than two coplanar oligomers, i.e. the trimer system in Figure 1, 3d, designated $3[(^3/_2, {}^1/_2)][(-^5/_2, -^1/_2)]$) and the hexamer and heptamer systems (Figure 1, 6 and 7, respectively), resulting in multiple-stack systems.

A list of new or previously untabulated stacking patterns is presented in Table I, with the corresponding envelope stacking diagrams being presented in Figure 1 (a comprehensive table and figure appear in the supplementary material).[2] This table contains several points of interest. (1) The trimer stacking pattern found in $(C_6H_9N)_2Cu_3Br_8$ corresponds to a stacking arrangement conjectured to occur 20% of the time in the disordered 3-methyl-2-aminopyridinium octachlorotricuprate(II) structure.[3] (2) The first known hexameric and heptameric systems have been synthesized.[4] (3) We have identified a compound, $Cu_3Cl_6(pyNO)_2(H_2O)_2$, that contains stacks of alternating $Cu_2Cl_4(pyNO)_2$ dimers and $CuCl_2(H_2O)_2$ monomers (pattern 8a).[5]

Experimental Section

Dichroic (orange–green) needle crystals of the title compound were grown by slow evaporation of a concentrated HCl solution equimolar in 1,2-dimethylpyridinium chloride and copper(II) chloride dihydrate. A crystal of dimensions 0.21 × 0.25 × 0.27 mm³ was mounted on a Syntex P2₁ diffractometer (Mo Kα radiation, graphite monochromator) upgraded to Nicolet R3m specifications. Reflections measured: 3437 total $(0 \le h \le 10; -11 \le k \le 11; -17 \le l \le 17; 2\theta_{max} = 55°$; bisecting geometry; 1.0° ω scans), 3172 independent, 2711 observed $(|F| > 3\sigma(|F|))$. Numerical absorption, Lorentz–polarization, and crystal decay corrections were applied. The structure was solved by direct methods, with final refinement of 155 least-squares parameters. Table II gives an abbreviated listing of the crystallographic data.

Table II. Crystallographic Data for $ACu_2Cl_5(H_2O)$ (A = 1,2-Dimethylpyridinium)

$C_7H_{12}Cl_5Cu_2NO$
$a = 7.077\ (1)$ Å
$b = 8.308\ (1)$ Å
$c = 12.655\ (2)$ Å
$\alpha = 87.63\ (1)°$
$\beta = 78.90\ (1)°$
$\gamma = 71.37\ (1)°$
$V = 691.8\ (2)$ Å³
$M_r = 430.53$
$P\bar{1}$ (No. 2)
$T = 293$ K
$\lambda = 0.71069$ Å
$\rho_{calc} = 2.07$ g/cm³
$\mu = 40.39$ cm⁻¹
$T_{max} = 0.48$, $T_{min} = 0.36$
$R(F_o) = 0.0325$
$R_w(F_o) = 0.0391$

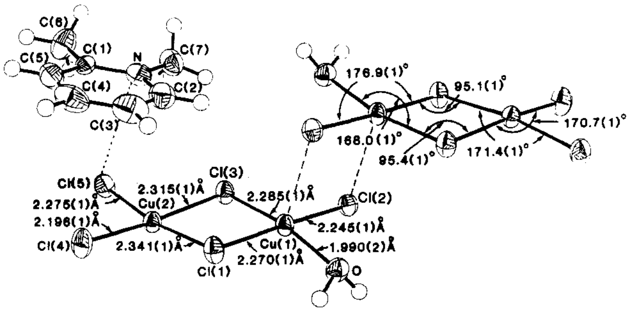

Figure 2. View of a portion of the title structure. The dotted line represents N–Cl interaction; the dashed lines represent Cu–Cl semicoordinate bonds.

Results and Discussion

The dimers (Figure 2) stack parallel to the *a* axis with pattern $2(^1/_2, {}^1/_2, 180°)(-^3/_2, -^1/_2, 180°)$. Dimers are linked within the stack by semicoordinate Cu–Cl bonds and by hydrogen bonding between the water molecule and Cl(5). Hydrogen bonding between the water molecule and a chloride ion (Cl(5)) in a neighboring stack defines layers of linked stacks parallel to the *ab* plane. Cu(1) exhibits a 4 + 1 + 1′ coordination geometry $((Cu(1)\text{-}Cl_{eq})_{av}$

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[2] A complete listing of the monomeric stacking patterns is given in a separate publication: Manfredini, T.; Pellacani, G. C.; Bonamartini-Corradi, A.; Battaglia, L. P.; Guarini, G. G. T.; Giusti, J. G.; Willett, R. D.; West, D. X. *Inorg. Chem.*, submitted for publication.

[3] Grigereit, T.; Ramakrishna, B. L.; Place, H.; Willett, R. D.; Pellacani, G. C.; Manfredini, T.; Menabue, L.; Bonamartini-Corradi, A.; Battaglia, L. P. *Inorg. Chem.* **1987**, *26*, 2235.

[4] Bond, M. R. Unpublished work.

[5] Sager, R. S.; Watson, W. H. *Inorg. Chem.* **1968**, *7*, 2035.

= 2.27 (2) Å; Cu(1)–O = 1.990 (2) Å; Cu–Cl$_{ax}$ = 2.832 (1) and 3.206 Å), and Cu(2) exhibits a 4 + 1 coordination geometry ((Cu–Cl$_{eq}$)$_{av}$ = 2.28 (6) Å; Cu–Cl$_{ax}$ = 2.898 (1) Å). The difference between terminal and bridging Cu–Cl distances ((Cu–Cl$_{term}$)$_{av}$ = 2.24 (4) Å; (Cu–Cl$_{br}$)$_{av}$ = 2.30 (3) Å) and the divergence in lengths of the semicoordinate bonds as one distance shortens are generally observed.[6,7]

The cations and anions do not form segregated stacks;[8] instead, the cation is approximately coplanar with the anion and located over the region containing Cu(2). The presence of the cation prevents Cu(2) from forming a semicoordinate bond with one neighbor, forcing the ($-^3/_2$, $-^1/_2$) displacement, while it is still free to form a semicoordinate bond with the other neighbor, resulting in the ($^1/_2$, $^1/_2$) displacement. A weak interaction between N and Cl(5), as pictured in Figure 2, is responsible for the partial insertion of the cation in the stack. Similar interactions have been observed by Prout and Murray-Rust in a series of paraquat halometalate salts in which N···Cl interaction distances and interaction vector angles range from 3.44 Å and 90° (for the $Cu_2Cl_6^{2-}$ salt) to 3.83 Å and 61° (for the $CoCl_4^{2-}$ salt) (interaction vector angle taken with respect to the cation plane).[9] The interaction distances and vector angles for the title compound (3.70 Å, 75°) compare favorably to these.

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Registry No. $(C_7H_{10}N)Cu_2Cl_5(H_2O)$, 120881-60-9; Cu, 7440-50-8.

Supplementary Material Available: A table of data collection conditions and parameters, a complete description of the experimental conditions, tables of non-hydrogen atomic coordinates and isotropic thermal parameters, anisotropic thermal parameters, hydrogen atom coordinates, bond lengths, and bond angles, a stereographic view of the contents of a unit cell, and a comprehensive table and diagram of known pseudoplanar, bibridged $Cu_nX_{2n+2}^{2-}$, $Cu_nX_{2n+1}L^-$, and $Cu_nX_{2n}L_2$ oligomer stacking patterns (16 pages); a table of observed and calculated structure factors (19 pages). Ordering information is given on any current masthead page.

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(6) Willett, R. D. *Transition Met. Chem.* (*London*) **1987**, *12*, 410.
(7) Willett, R. D. *Acta Crystallogr.* **1988**, *B44*, 503.
(8) Bond, M. R.; Willett, R. D. *Acta Crystallogr.* **1987**, *C43*, 2304.
(9) Prout, C. K.; Murray-Rust, P. *J. Chem. Soc. A* **1969**, 1520.
(10) Willett, R. D.; Rundle, R. E. *J. Chem. Phys.* **1964**, *40*, 838.
(11) Manfredini, T. Ph.D. Thesis, Universities of Modena and Bologna, Italy, 1984. Battaglia, L. P.; Bonomartini-Corradi, A.; Geiser, U.; Willett, R. D.; Motori, A.; Sandrolini, F.; Antolini, A.; Manfredini, T.; Menabue, L.; Pellacani, G. C. *J. Chem. Soc., Dalton Trans.* **1988**, 265.
(12) Willett, R. D. *J. Chem. Phys.* **1966**, *44*, 39.
(13) Battaglia, L. P.; Bonamartini-Corradi, A.; Marcotrigiano, G.; Menabue, L.; Pellacani, G. C. *Inorg. Chem.* **1980**, *19*, 125.
(14) Halvorson, K. E.; Grigereit, T.; Willett, R. D. *Inorg. Chem.* **1987**, *26*, 1716.
(15) Patyal, B.; Scott, B.; Willett, R. D. Manuscript in preparation.
(16) Pon, G.; Willett, R. D. Private communication, 1989.

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Cis and Trans Isomers of $Mo(N_2)_2(PMe_2Ph)_4$[1]

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There are many six-coordinate, bis(dinitrogen) complexes of chromium, molybdenum, and tungsten known of the type $[M(N_2)_2L_4]$, where the ligands L are usually[4] some combination of mono-, bi-, and tridentate organophosphines.[6] In the majority of examples the N_2 ligands adopt a trans configuration. However, there are a few examples of complexes with the cis configuration specifically when L ligands are small, e.g., *cis*-$[M(N_2)_2(PMe_3)_4]$ (M = Cr,[7] Mo,[8] W[8]) and *cis*-$[M(N_2)_2(PMe_2Ph)_4]$ (M = Mo,[9] W[10]). Recently, a relatively stable mixture of *cis*- and *trans*-$[Mo(N_2)_2(Me_2PCH_2CH_2PMe_2)_2]$ was isolated.[11] We wish to report the first example of the effective separation of the cis and trans isomers of a bis(dinitrogen) complex, $[Mo(N_2)_2(PMe_2Ph)_4]$.

Experimental Section

All manipulations were carried out under an inert atmosphere with standard Schlenk techniques,[12] in the dark (wrapping with aluminum foil) wherever possible. Solvents were dried by standard procedures and distilled under dry dinitrogen prior to use. Infrared spectra were recorded with a Perkin-Elmer 283 instrument. ^{31}P NMR spectra were recorded in C_6D_6 on a Varian VXR-200 spectrometer operating at 80.894 MHz. $MoCl_3(THF)_3$ was prepared according to published procedures.[13]

Preparation of *cis*- and *trans*-$[Mo(N_2)_2(PMe_2Ph)_4]$. Into a 250-mL Fischer–Porter bottle was placed THF (70 mL), 2.282 g (16.5 mmol) of PMe_2Ph, 2.040 g (4.88 mmol) of $MoCl_3(THF)_3$, and a magnetic stirbar. After the mixture was stirred briefly, 100 g of 1% sodium amalgam was added. The bottle was closed and pressurized with N_2 (75 psi) and the mixture stirred (17 h). The light red color changed to green within 1 h and eventually turned dark yellow-brown. After the bottle was depressurized, the solution was decanted and filtered through diatomaceous earth (acid washed) and washed with Et_2O (5 × 5 mL). A brown solid remained. The combined filtrate was evaporated to dryness in vacuo to yield a black oily residue. The residue was dissolved in Et_2O (40 mL) and filtered through diatomaceous earth. Solvent was removed from the filtrate by bubbling N_2 through the solution to produce a red-brown residue. Pentane (50 mL) was added to the flask and the suspension stirred (1 h). The yellow-brown solid was filtered off, washed with pentane (2 × 10 mL), and dried in vacuo. The yield of product was 1.672 g (2.4 mmol, 58%). IR (KBr): $\nu(N_2)$ 2010 (s), 1940 (s) cm^{-1}. ^{31}P NMR: cis, δ 4.5 (m, 27); trans, δ 3.3 (s, 1).

The red-brown pentane filtrate was evaporated to dryness by blowing N_2 across the surface of the solution. Methanol (30 mL) was added and the suspension stirred (1.5 h). The orange-brown solid was collected by filtration, washed with methanol (2 × 5 mL), and dried in vacuo. The product (yield 0.40 g, 0.57 mmol, 14%), was a 1:1 mixture of isomers.

Pentane (20 mL) was added to 0.372 g of the 1:1 mixture and the suspension stirred (1 h). The yellow cis isomer was filtered off, washed with pentane (2 × 10 mL), and dried in vacuo to yield 0.149 g (0.21 mmol). IR (KBr): $\nu(N_2)$ 2010 (vs), 1935 (vs) cm^{-1}. The filtrate was stripped to dryness in vacuo. Deoxygenated reagent methanol (10 mL) was added to the residue. After 1 h the orange trans isomer was isolated by filtration and washed with methanol (10 mL) to yield 0.13 g (0.18 mmol). IR (KBr): $\nu(N_2)$ 2010 (m), 1923 (vs) cm^{-1}. ^{31}P NMR: cis, δ 4.5 (m, 1); trans, δ 3.3 (s, 7.2).

Electrochemistry. Cyclic voltammetry measurements were carried out in a two-compartment, three-electrode cell fitted with a Luggin probe using a Hi-Tek potentiostat, Type DT2101, a Chemical Electronics (Birtley) Ltd. waveform generator, Type 01, and a Philips PM8041 X-Y recorder. The cell was fitted with a platinum-wire working electrode, a

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(1) Reactions of Coordinated Dinitrogen. 23. Part 22: Gebreyes, K.; George, T. A.; Koczon, L. M.; Ma, L.; Shaikh, S. N.; Tisdale, R. C.; Zubieta, J. Submitted for publication in *Polyhedron*.
(2) University of Nebraska.
(3) University of Sussex.
(4) For examples in which one or more ligating atoms are not phosphorus, see ref. 5.
(5) (a) Aresta, M.; Sacco, A. *Gazz. Chim. Ital.* **1972**, *102*, 755. (b) Lazarowych, N. J.; Morris, R. H.; Ressner, J. M. *Inorg. Chem.* **1986**, *25*, 3926. (c) George, T. A.; Jackson, M. A. *Inorg. Chem.* **1988**, *27*, 924. (d) Yoshida, T.; Adachi, T.; Kaminaka, M.; Ueda, T.; Higuchi, T. *J. Am. Chem. Soc.* **1988**, *110*, 4872.
(6) George, T. A. In *Homogeneous Catalysis with Metal Phosphine Complexes*; Pignolet, L. H., Ed.; Plenum: New York, 1983; pp 405–441.
(7) Karsch, H. H. *Angew. Chem. Int. Ed. Engl.* **1977**, *16*, 56.
(8) Carmona, E. *J. Organomet. Chem.* **1988**, *358*, 283.
(9) George, T. A.; Seibold, C. D. *Inorg. Chem.* **1973**, *12*, 2544.
(10) Chatt, J.; Heath, G. A.; Richards, R. L. *J. Chem. Soc., Dalton Trans.* **1974**, 2074.
(11) Fong, L. K.; Fox, J. R.; Foxman, B. M.; Cooper, N. J. *Inorg. Chem.* **1986**, *25*, 1880.
(12) Shriver, D. F. *Manipulations of Air Sensitive Compounds*; McGraw-Hill: New York, 1969.
(13) Dilworth, J. R.; Zubieta, J. A. *J. Chem. Soc., Dalton Trans.* **1983**, 397.

0020-1669/89/1328-3269$01.50/0