quantum yields was observed after 25% photolysis and was ascribed to the decay of the products in parallel with the primary process.

An interesting example has been described in the literature, illustrating the exhaustive photolysis of the ferrocyanide ion in the presence of bipyridyl or phenanthroline.[18] From this work, it became apparent that a ligand like phenanthroline could give some evidence for labile tetracyano intermediates. Due to its rigidity, 1,10-phenanthroline does not coordinate to the pentacyanoferrate(II) ion. On the other hand, the $Fe(CN)_4(H_2O)_2^{2-}$ and $Fe(CN)_4(H_2O)L^{2-}$ intermediates are expected to be labile and thus to react with 1,10-phenanthroline, forming $Fe(CN)_4phen^{2-}$.

The photolysis of the $Fe(CN)_5en^{3-}$ complex, carried out in the presence of phenanthroline, yielded $Fe(CN)_4en^{2-}$ as the major product, with an approximate quantum yield of 0.063. The $Fe(CN)_4phen^{2-}$ complex was also found, but in small amounts, with a quantum yield smaller than 0.01, along with traces of $Fe(CN)_2(phen)_2$ and $Fe(phen)_3^{2+}$ species.

Independently of the mechanism involved, one can see that the photolabilization of the cyanide ion does take place with relatively high quantum yields in the $Fe(CN)_5en^{3-}$ complex. The efficiency is comparable to the normal substitution of L in the $Fe(CN)_5L^{3-}$ complex, induced by direct ligand field excitation, as shown in Table I. On the other hand, it is certainly much higher than one would expect for reaction 2, where the regeneration of the quasi-dissociated species is likely to be more efficient than the attack of the solvent molecules. Then, it seems reasonable that, by forming an incipient chelate ring in the excited state, the ethylenediamine ligand could overcome this kind of effect in the pentacyanoferrates.[19]

An analogous behavior has also been found in the photolysis of metal hexacarbonyls,[3] where in the presence of monodentate ligands the monosubstituted species $M(CO)_5L$ are produced as the major species, while with bidentate ligands the chelate species $M(CO)_4(Chel)$ predominate.

Acknowledgment. A fellowship from CNPq (N.Y.M.I.) is gratefully acknowledged.

Registry No. $Fe(CN)_5en^{3-}$, 82482-32-4; $Fe(CN)_4en^{2-}$, 82494-75-5; $Fe(CN)_4phen^{2-}$, 17455-55-9; 1,10-phenanthroline, 66-71-7; ethylenediamine, 107-15-3.

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(19) The possibility that the photoexcited $Fe(CN)_5en^{3-}$ complex undergoes associative attack by the excess ethylenediamine is not supported by the concentration dependence experiments shown in Table I.

Communications

Direct MCD Identification of Electronic Excited States of *trans*-$Mo(CO)_4[P(n\text{-}Bu)_3]_2$ and the MO Origins and MO Fates of the Excitation Processes

Sir:

Several authors[1] recently concerned themselves with electronic excited states of substituted group 6 (Cr, Mo, W) carbonyl complexes and made direct state assignments using MCD spectroscopy. The MCD of parent molecules $M(CO)_6$ has also been reported.[2] In this publication we report the measurement and interpretation of the MCD spectrum of a trans tetracarbonyl, *trans*-$Mo(CO)_4[P(n\text{-}Bu)_3]_2$, with interesting results about the four lowest energy spin-allowed excited states. The study of this first trans carbonyl was needed because $u \leftarrow g$ charge-transfer bands can be differentiated from all d–d bands. MCD spectra are complemented with 77 and 300 K optical data. Firm state and orbital assignments are made. The results so obtained are also interesting because band polarizations cannot come from crystal spectroscopy since there is no suitable optical host for such carbonyl molecules. Furthermore, polarized crystal spectra can almost never differentiate between the details of the MO nature for two possible excited-state assignments, ${}^1E_1{}^a$ and ${}^1E_u{}^b$. This differentiation can be made by MCD if opposite-sign *A* terms are present due to their different configurational compositions. The present findings have interesting implications in the areas of photochemistry and electronic structures of carbonyl complexes.

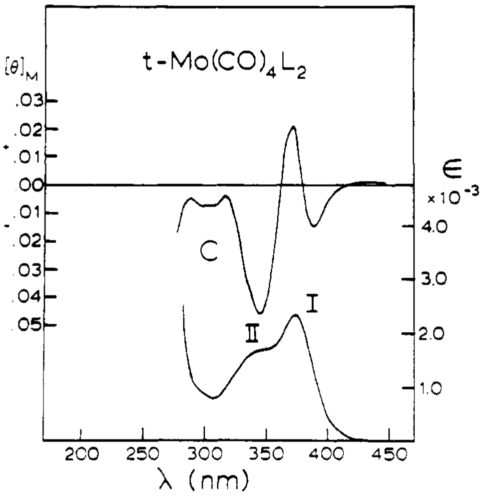

Figure 1. Room-temperature optical and MCD solution spectra of *trans*-$Mo(CO)_4[P(n\text{-}Bu)_3]_2$ in cyclohexane. $[\theta]_M$ is the molar ellipticity for a field intensity of 1 G, and ϵ has units M^{-1} cm^{-1}.

Quantities of *trans*-$Mo(CO)_4[P(n\text{-}Bu)_3]_2$ having the same properties (IR, optical, melting point) were synthesized from each of two procedures by modifying one (method A) that starts with $Mo(CO)_6$[3] and using a second one that starts with

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(1) (a) MCD data on numerous chromium, molybdenum, and tungsten pentacarbonyl amine molecules and others of A. F. Schreiner, S. Amer, and W. M. Duncan have appeared: R. M. Dahlgren, Ph.D. Dissertation, UCLA, 1978; R. M. Dahlgren and J. I. Zink, *J. Am. Chem. Soc.*, **101**, 1448 (1979); *Inorg. Chem.*, **18**, 597 (1979). (b) A. F. Schreiner, S. Amer, W. M. Duncan, R. M. Dahlgren, and J. I. Zink, Abstracts, 178th Natinal Meeting of the American Chemical Society, Washington, D.C., Sept. 1979, No. INOR 219. (c) G. Boxhoorn, D. J. Stufkens, P. J. F. M. van de Coolwijk, and A. M. F. Hezemans, *J. Chem. Soc., Chem. Commun.*, 1075 (1979). (d) A. F. Schreiner, S. Amer. W. M. Duncan, R. M. Dahlgren, and J. I. Zink, *J. Am. Chem. Soc.*, **102**, 6871 (1980). (e) A. F. Schreiner, S. Amer, W. M. Duncan, and R. M. Dahlgren, *J. Phys. Chem.*, **84**, 2688 (1980). (f) G. Boxhoorn, D. J. Stufkens, P. J. F. M. van de Coolwijk, and A. M. F. Hezemans, *Inorg. Chem.*, **20**, 2778 (1981).

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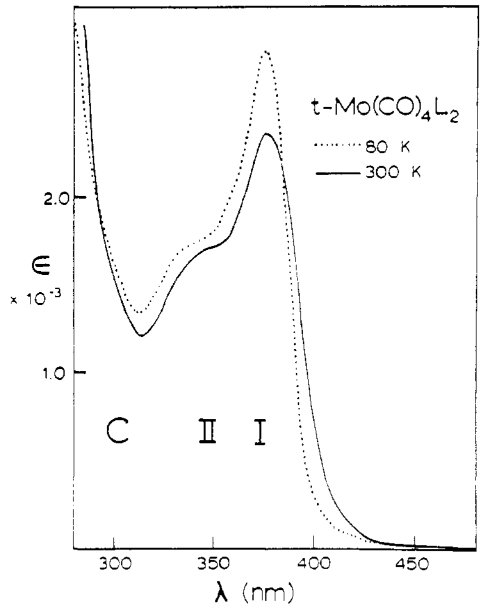

Figure 2. Optical spectra of *trans*-$[Mo(CO)_4[P(n\text{-}Bu)_3]_2$ in a poly(vinyl chloride) matrix at 80 K (···) and 300 K (—). The optical absorptivity scale is only a relative one in that ϵ_{max} of band I is arbitrarily given the same value as is found in cyclohexane solution.

the cis isomer.[4,5] Method A consisted of the reaction between $Mo(CO)_6$ and the ligand in 1:2 molar ratio in diglyme at 130°C under N_2, monitoring the amount of CO displaced, filtering, and vacuum evaporating the filtrate solvent. The oily composition contained the cis and trans isomers. The isomers were cleanly separated on an alumina gravity column with petroleum ether (bp 37.1–57.8 °C), subsequently evaporated to yield an oil, which from CH_2Cl_2–CH_3OH solution at 0 °C gave the pure trans isomer, the properties of which are distinct from those of the cis isomer or their mixture. The solid was stored in the dark under N_2, and fresh solutions were kept in the dark until spectra were obtained. Follow-up spectral scans showed that there was no solution decomposition. MCD measurements were made as previously described.[6]

The results and their discussion include the 300 K optical and MCD spectra (cyclohexane solvent) shown in Figure 1 and the optical spectra at 300 and 80 K (poly(vinyl chloride) matrix) shown in Figure 2. While weak band C (~300 nm) appears in all three spectra, it is best defined in the MCD. Figure 2 shows[7] that bands I (375 nm) and II (345 nm) are electric dipole allowed u ← g excitations, since the conditions of constant optical intensity and $\epsilon_{max}(77\ K) > \epsilon_{max}(300\ K)$ are each characteristics of allowed u ← g optical bands in centric molecules. These must consequently be CT bands in view of the centric coordination about Mo, so that this rules out d–d (g ← g) identities for bands I and II. Furthermore, D_{4h} static electric dipole selection rules allow only $^1E_u \leftarrow {}^1A_{1g}$ and $^1A_{2u} \leftarrow {}^1A_{1g}$ excitations for such closed-shell molecules. For this reason bands I and II can in principle involve excitation to (i) $^1E_u{}^a$ and $^1E_1{}^b$, (ii) $^1A_{2u}{}^a$ and $^1A_{2u}{}^b$ or (iii) $^1A_{2u}$ and ^1E_u, in the absence of knowing at this point how to associate the members of any of these pairs with bands I and II. However, Figure 1 shows that there is a distinct *A* term for band I and positive B terms (negative ellipicity) for bands II and C. The *presence* of this *A* term is direct evidence that band I is of in-plane (*xy*) polarization, and therefore excitation $^1E_u \leftarrow {}^1A_{1g}$, which establishes the symmetry of the excited state. The *positive* sign of the *A* term is discussed below in terms of its open-shell excited MO configuration.

The MO natures of CT bands I and II are assigned to MTLCT transitions generally (π^* CO ← "t_{2g}" MO), but specifically to a_{2u}(100% π^* CO) ← $e_g{}^4$(~$4d_{xz}$ Mo), or state transition $^1E_u \leftarrow {}^1A_{1g}$ (presence of the MCD *A* term), and e_u(~π^* CO > 5p Mo) ← $e_g{}^4$(~$4d_{xz}$, d_{yz} Mo), or state transitions $^1A_{2u} \leftarrow {}^1A_{1g}$ (presence of MCB *B* term and absence of *A* term). The reason for this selection of the excitation orbital origin is that PES data showed[8] e (~$4d_{xz}$, d_{yz}) > b_2(~$4d_{xz}$), within the Koopmans approximation, and that this is the order for monosubstitution already, $M(CO)_5L(N,P)$. Furthermore, the assignment of our band I to $a_{2u} \leftarrow e_g$, or $^1E_u \leftarrow {}^1A_{1g}$, is based on the total of four reasons: (i) the presence of the observed MCD *A* term implies an excited state ^1E_u, (ii) ^1E_u must be of excited open-shell origin $a_{2u}{}^1e_g{}^3$ or $e_u{}^1b_{2g}{}^1$ (vide infra), (iii) but a_{2u} is the LUMO here as in $Pt(CN)_4{}^{2-}$[9] because the latter is valence-shell isoelectronic with our $Mo(CO)_4$ beginning with $b_{2g}{}^2$(~$4d_{xy}$) and moving to higher energy (d_{z^2} of Pt is assumed to be nonbonding, *and* MOs of the two Mo–P σ bonds of the trans complex are bonding MOs of expected energy below $b_{2g}{}^2$(~$4d_{xy}$)), and (iv) this transition (I) is predicted, as experimentally found for $Pt(CN)_4{}^{2-}$ and here for *trans*-$Mo(CO)_4L_2$, to have a *positive A* term for this frontier-orbital MTLCT transition, $a_{2u} \leftarrow e_g{}^4$, or $^1E_u \leftarrow {}^1A_{1g}$.

Band II is assigned on the basis of the following. First, e_u(~π^* CO > 5p Mo) is the MO above LUMO a_{2u},[9] so that four ~π^* CO ← ~4d Mo excited configurations and their states result from $b_{2g}{}^2$(~$4d_{xy}$) < $e_g{}^4$(~$4d_{xz}$, d_{yz}) < a_{2u}(in-plane π^* CO) < e_u(~π^* CO > 5p Mo):

$e_u{}^1b_{2g}{}^1$	$^1E_u{}^b$
$a_{2u}{}^1b_{2g}{}^1$	–($^1B_{1u}$)
$e_u{}^1e_g{}^3$	$^1A_{2u}$ ($^1A_{1u}$, $^1B_{1u}$, $^1B_{2u}$)
$a_{2u}{}^1e_g{}^3$	$^1E_u{}^a$

Assignment of band II to the $e_u{}^1e_g{}^3$ configuration and $^1A_{2u}(e_u{}^1e_g{}^3) \leftarrow {}^1A_{1g}$ can be made by exclusion; viz., it is consistent with (i) the absence of an MCD *A* term and presence of *B* term in our tetracarbonyl MCD spectrum, (ii) the expectation of a positive *A* term, not experimentally observed at band II, for $^1E_u{}^b$ of the above fourth configuration, and (iii) the group-theoretical finding that the third configuration leads to a *vibronically* allowed MTLCT excited state, $^1B_{1u}$, in contradiction to the requirement of the 80 K/300 K optical band II behavior. It is an interesting feature of I and II that these ~π^* CO ← ~4d Mo CT bands are at lower energy for *trans*-$Mo(CO)_4[P(n\text{-}Bu)_3]_2$ (370 and 360 nm) than the lowest energy MTLCT transition of $Mo(CO)_6$ (ca. 290 nm), and the expected higher electron density at Mo in the tetracarbonyl must be important contributor to this MTLCT energy lowering.

Band C at ca. 300 nm is assigned to be d–d because of its very much lower area and peak intensity ($\epsilon_{max} < 200$) compared to those of the adjacent u ← g CT bands I and II, its narrowness, and its energy. The Wentworth–Piper (W–P) empirical MO and CF models, in the absence of strong state interactions, predict,[10,11] and in practice it is approximately found, that the energies of parent $^1T_{1g}[(t_{2g}{}^5e_g{}^1); ML_6]$ and daughter $^1A_{2g}[(b_{2g}{}^1(\sim nd_{xy})b_{1g}{}^1(\sim nd_{x^2-y^2}); ML_5L']$ states are equal. Indeed, $^1T_{1g} \leftarrow {}^1A_{1g}$ of $Mo(CO)_6$ has been assigned

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most often to the 313-nm band,[12] and the presently assigned $^{1}A_{2g} \leftarrow {}^{1}A_{1g}$ d–d band of *trans*-$Mo(CO)_4[P(n\text{-}Bu)_3]_2$ is at 300 nm. Using the W–P MO model, one also predicts that the second d–d daughter state of $^{1}T_{1g}$, or $^{1}E_g[e_g{}^3(\sim 4d_{xz}, d_{yz})a_{1g}{}^1(\sim 4d_{z^2}); {}^{1}T_{1g}]$, is under band I and/or II, or $^{1}E_g < {}^{1}A_{2g}$, since its transition energy equals $^{1}T_{1g}[M(CO)_6] + {}^{1}/_{2}\delta(10Dq)$. The quantity $\delta(10Dq)$ is the difference $10Dq'[P(n\text{-}Bu)_3] - 10Dq^{\circ}(CO)$, for which the magnitude of $10Dq^{\circ}$ and the inequality $10Dq' < 10Dq^{\circ}$ are known but $10Dq'$ is not known quantitatively.

The red-most weak optical shoulder near ~415 nm and its net positive MCD ellipticity are attributed to a spin-forbidden transition, $^{3}\Gamma \leftarrow {}^{1}A_{1g}$, which either is the partner, $^{3}E_u(a_{2u}{}^1e_g{}^3)$, of band I ($^{1}E_u(a_{2u}{}^1e_g{}^3)$), or is of "$^{3}T_{1g}$" ($t_{eg}{}^5e_g{}^1$) origin ($^{3}E_g[e_g{}^3(\sim 4d_{xz}, d_{yz})a_{1g}{}^1(\sim 4d_{z^2}); {}^{3}T_{1g}]$), the partner of $^{1}E_g(T_{1g})$.

In summary, MO origins, MO fates, excited-state symmetries, and excitation polarization assignments were made. Bands I and II are π^* CO ← ~$4d^6$ Mo MTLCT transitions to respectively excited states $^{1}E_u[a_{2u}{}^1(\pi^*\ CO)e_g{}^3(\sim 4d_{xz}, d_{xz}\ Mo)]$ and $^{1}A_{2u}[e_u{}^1(\sim \pi^*\ CO > 5p\ Mo)e_g{}^3(\sim 4d_{xz}, d_{yz}\ Mo)]$. Band C is a d–d transition involving the excited state $^{1}A_{2g}[b_{2g}{}^1(\sim 4d_{x^2-y^2}); {}^{1}T_{1g}]$, and the d–d transition to $^{1}E_g[e_g{}^3(\sim 4d_{xz}, d_{yz})a_{1g}{}^1(\sim 4d_{z^2}); {}^{1}T_{1g}]$ is at lower energy than $^{1}A_{2g}$ by $^{1}/_{2}\delta(10Dq)$ (under band I and/or II).

This excitation information is also photochemically interesting, and especially so since the two lowest energy long-lived spin-forbidden triplets are expected to be the d–d state $^{3}E_g[e_g{}^3(\sim 4d_{xz}, d_{yz})a_{1g}{}^1(\sim 4d_{z^2}); {}^{3}T_{1g}]$ and the MTLCT state $^{3}E_u[a_{2u}{}^1(\pi^*\ CO)e_g{}^3(\sim 4d_{xz}, d_{yz}\ Mo)]$ as a consequence of the above excitation findings. In state $^{3}E_g$ the molecule will be preferentially z axis (P–Mo–P) labilized. It is suggested that a wavelength-dependent quantitative photochemistry study (labeled ligand exchange or substitution) be undertaken in order to locate $^{1}E_g$, and preferentially $^{3}E_g$, more accurately. The photosubstitution study[13] of a similar complex, *trans*-$Mo(CO)_4(PPh_3)_2$, was carried out already at two wavelengths (366 and 313 nm) and gave results compatible with the present findings. For example, quantum yields for PPh_3 loss followed by CO entry are 0.30 and 0.20 at 366 and 313 nm, respectively, and *both* CO (QY = 0.30) *and* PPh_3 (QY = 0.33) photosubstitutions take place with irradiation at 366 nm (band I). Molecular series studies are under way.

Registry No. *trans*-$Mo(CO)_4[P(n\text{-}Bu)_3]_2$, 17652-79-8.

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X-ray Crystal and Molecular Structure of a Platinum(II) Complex Containing a PtS_4 Ring, $[(C_6H_5)_3P]_2PtS_4 \cdot CHCl_3$

Sir:

We report here the first crystal and molecular structure of a compound containing a PtS_4 ring although compounds containing PtS_5 rings have been known for nearly 100 years.[1] The only MS_4 rings that have known structures are for M = Mo and W (Table I).[2] Our interest in the reactivity of tansition-metal complexes containing catenated sulfur[3] prompted this investigation. The title compound was originally prepared by Chatt and Mingos[4] from tetrakis(triphenylphosphine)platinum(0) and elemental sulfur. The sample used in the current study was prepared by the method of Kreutzer et al.[5] according to eq 1.[6–10]

$$(NH_4)_2Pt(S_5)_3 + 12(C_6H_5)_3P \rightarrow (NH_4)_2S + [(C_6H_5)_3P]_2PtS_4 + 10(C_6H_5)_3PS \quad (1)$$

Table I. Bond Lengths for S–S Bonds in Compounds with S_4 Structural Units (in Å)[a]

	S(1)–S(2)	S(2)–S(3)	S(3)–S(4)
$(Ph_3P)_2PtS_4$	2.024 (8)	2.022 (10)	2.081 (10)
$[AsPh_4][Mo_2S_{10}]$(a)[b]	2.019 (5)	1.970 (6)	2.115 (5)
$[AsPh_4]_2[Mo_2S_{10}]$(b)[b]	2.096 (16)	1.936 (19)	2.169 (14)
$(Et_4N)_2MoS_9$[c]	2.107 (1)	2.012 (1)	2.166 (1)
$(\eta^5\text{-}C_5H_5)_2MoS_4$[d]	2.081 (8)	2.018 (9)	2.085 (7)
$(\eta^5\text{-}C_5H_5)_2WS_4$[e]	2.105 (7)	2.016 (8)	2.116 (9)
$BaS_4 \cdot H_2O$(a)[f]	2.069 (4)	2.063 (4)	2.069 (4)
$BaS_4 \cdot H_2O$(b)[f]	2.079 (3)	2.062 (4)	2.079 (3)
$[C_6H_2(OEt)_2S_4]_2$(a)[g]	2.028 (5)	2.068 (5)	2.027 (5)
$[C_6H_2(OEt)_2S_4]_2$(b)[g]	2.034 (5)	2.067 (5)	2.024 (5)

[a] Abbreviations: Ph, C_6H_5; Et, C_2H_5. [b] Reference 2a. The S_4 ring is disordered with (a) the major component and (b) the minor one. [c] Reference 2d. [d] Reference 2b. [e] Reference 2c. [f] Reference 17. There were two independent S_4 chains (a and b). [g] Reference 18. The molecule has two S_4 units (a and b).

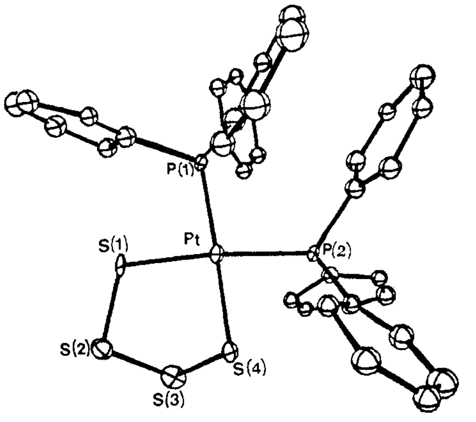

Figure 1. ORTEP diagram of $(Ph_3P)_2PtS_4$ (50% probability thermal ellipsoids). Important bond lengths (Å) and angles (deg): Pt–S(1), 2.360 (6); Pt–S(4), 2.366 (5); S(1)–S(2), 2.024 (8); S(2)–S(3), 2.022 (10); S(3)–S(4), 2.081 (10); Pt–P(1), 3.300 (5); Pt–P(2), 2.283 (5); P–C(av), 1.85 (2); C–C(av), 1.41 (3); S(1)–Pt–S(4), 92.0 (3); P(1)–Pt–P(2), 99.3 (2); S(1)–Pt–P(1), 82.6 (3); S(4)–Pt–P(2), 87.2 (3). The atom S(2) is 0.966 (7) Å removed from the least-squares P_2PtS_4 plane with no other atom more than 0.17 Å from this plane.

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