Al^{3+} ions. Thus, the idea that the stuffed condition may result from the pores, formed by the pillars, being filled by smaller species is plausible but not the only possible explanation. Why this occurs with Zr and not with titanium is an intriguing question. Electron micrographs show that the α-ZrP and α-TiP platelet retain their shape upon pillaring, but the titanium platelets exhibit a corrugated appearance about the edges, which may indicate large pores allowing access to the interior of the solid. Further investigations are in progress as is also an extension of the technique described here to other classes of layered compounds.

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Comparative Study of the Electronic Structures of *N*,*N'*-Diethyldithiocarbamate and Pyrrole-*N*-carbodithioate and Their Nickel(II) Complexes

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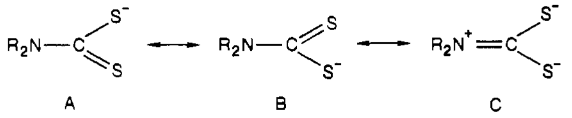
Dithiocarbamates, which can be represented by resonance forms A–C, have been used extensively as ligands in inorganic, bioinorganic, and analytical chemistry.[1] These ligands fall into two broad classes. Dialkyldithiocarbamates, such as *N*,*N'*-diethyldithiocarbamate (Et_2dtc^-), are most often employed. Recent interest, however, has focused on dithiocarbamates derived from aromatic amines such as pyrrole.[2] Because of the aromatic character of pyrrole, resonance form C is assumed to be less important in pyrrole-*N*-carbodithioate ($pdtc^-$) than it is in the dialkyl analogues.[2] This shift in electron density results in a less positive nitrogen atom and less negative sulfur atoms in pyrrole-*N*-carbodithioate. The coordination behavior of the ligand is thereby altered. It has also been proposed that pyrrole-*N*-carbodithioate forms stronger π-bonds with the metal than the dialkyl analogues.[3]



Since an increasing number of workers are using aromatic dithiocarbamate ligands to control the physical and chemical properties of dithiocarbamate complexes,[3-9] it seemed important to determine whether quantum-mechanical models agree with qualitative ideas about the relative importance of resonance forms A–C and the importance of metal–ligand π-bonding in pyrrole-*N*-carbodithioate complexes. We also wished to determine whether any insight could be gained concerning the relative reactivities of coordinated dithiocarbamate ligands from such quantum-mechanical models. Previous CNDO/2 calculations have been performed on the dimethyldithiocarbamate complex of nickel(II),[10,11] but no MO calculations have been reported on the analogous pyrrole-*N*-carbodithioate system. Results of INDO/S[12,13] calculations on the free diethyldithiocarbamate and pyrrole-*N*-carbodithioate ligands and their nickel(II) complexes are reported in this note, and the effects of including sulfur d orbitals are assessed. Details of the theoretical model are summarized in the Appendix.

Table I. Wiberg Bond Indices[a,b]

	Et_2dtc^-	$pdtc^-$
	No Point Charge	
C–S	1.21 (0.24)	1.30 (0.33)
	[1.27 (0.29)]	[1.37 (0.38)]
C–N	1.33 (0.36)	1.13 (0.18)
	[1.28 (0.33)]	[1.09 (0.16)]
	+1 Point Charge[c]	
C–S	1.13 (0.16)	1.21 (0.23)
	[1.18 (0.17)]	[1.27 (0.27)]
C–N	1.53 (0.55)	1.29 (0.32)
	[1.48 (0.53)]	[1.25 (0.31)]
	Ni(II) Complex	
C–S	1.13 (0.16)	1.20 (0.23)
	[1.17 (0.19)]	[1.25 (0.27)]
C–N	1.48 (0.51)	1.25 (0.29)
	[1.45 (0.51)]	[1.23 (0.29)]
Ni–S	0.71 (0.02)	0.70 (0.02)
	[0.81 (0.06)]	[0.79 (0.06)]

[a] Numbers in parentheses are Wiberg π-bond indices between orbitals perpendicular to the NiS_4 plane. [b] Numbers in brackets refer to results when d orbitals are included on sulfur. [c] Assuming +1 charge at the site where the metal would be located.

Results and Discussion

Wiberg bond indices[14] for *N*,*N'*-diethyldithiocarbamate, pyrrole-*N*-carbodithioate, and their corresponding nickel(II) complexes are listed in Table I. Since the Wiberg bond index has approximately the same value as the traditional bond order (i.e., a single bond has a Wiberg index of 1.0, and a double bond 2.0), it is easily related to classical resonance forms. In agreement with previous arguments, the C–S index is higher and the C–N index lower in $pdtc^-$ and $Ni(pdtc)_2$ than in Et_2dtc^- and $Ni(Et_2dtc)_2$.[15] Bonding to the metal has a comparable effect on the two ligands. The C–S index is lowered and the C–N index raised, implying that resonance form C contributes more to the bonding in the complex than in the free ligand. Experimentally, this leads to an increase in the C–N stretching frequency upon coordination to the metal.[16] The effect of coordination to nickel(II) on the Wiberg indices of the two ligands can be mimicked by the presence of a +1 point charge at the site where the metal is located. This suggests that much of the change in electron distribution upon coordination is a result of the electrostatic influence of the metal. The magnitudes of the Wiberg indices are changed slightly by including d orbitals on sulfur, but the trends are identical.

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Table II. Net Atomic Charges[a–c]

	Et_2dtc^-	$pdtc^-$	$Ni(Et_2dtc)_2$	$Ni(pdtc)_2$
q(S)	−0.66 (−0.68)	−0.61 (−0.62)	−0.60 (−0.30)	−0.55 (−0.25)
	[−0.56 (−0.68)]	[−0.49 (−0.60)]	[−0.52 (−0.32)]	[−0.46 (−0.26)]
q(C)	0.29 (0.25)	0.29 (0.26)	0.32 (0.30)	0.34 (0.34)
	[0.04 (0.18)]	[0.03 (0.19)]	[0.04 (0.24)]	[0.05 (0.27)]
q(N)	−0.25 (−0.10)	−0.15 (0.03)	−0.20 (−0.05)	−0.15 (0.03)
	[−0.22 (−0.07)]	[−0.12 (0.06)]	[−0.15 (0.02)]	[−0.11 (0.08)]
q(Ni)			1.33 (0.04)	1.35 (0.06)
			[1.43 (0.06)]	[1.46 (0.08)]

[a] Based on Mulliken populations with the assumption that the INDO basis set represents a symmetrically orthogonalized set of atomic orbitals. [b] Numbers in parentheses are based on the ZDO bond order matrix. [c] Numbers in brackets correspond to calculations including d orbitals on sulfur.

Table III. Atomic Orbital Populations[a,b]

	$Ni(Et_2dtc)_2$	$Ni(pdtc)_2$		$Ni(Et_2dtc)_2$	$Ni(pdtc)_2$
			Nickel		
4s	0.30 (0.57)	0.27 (0.55)	$d_{x^2-y^2}$	1.93 (1.92)	1.94 (1.93)
$4p_x$	−0.23 (0.36)	−0.24 (0.35)	d_{xy}	0.89 (0.89)	0.88 (0.89)
$4p_y$	−0.07 (0.36)	−0.08 (0.36)	d_{xz}	1.96 (1.96)	1.96 (1.96)
$4p_z$	−0.01 (0.06)	−0.01 (0.05)	d_{yz}	1.97 (1.97)	1.97 (1.97)
d_{z^2}	1.84 (1.85)	1.85 (1.86)			
			Sulfur		
4s	2.13 (1.82)	2.13 (1.82)	$d_{x^2-y^2}$	0.03 (0.05)	0.03 (0.05)
$4p_x$	1.35 (1.33)	1.38 (1.36)	d_{xy}	0.00 (0.04)	0.00 (0.04)
$4p_y$	1.12 (1.18)	1.10 (1.17)	d_{xz}	0.02 (0.02)	0.02 (0.02)
$4p_z$	1.85 (1.83)	1.79 (1.77)	d_{yz}	0.02 (0.03)	0.02 (0.03)
d_{z^2}	0.00 (0.01)	0.00 (0.01)			

[a] Based on Mulliken populations with the assumption that the INDO basis set represents a symmetrically orthogonalized set of atomic orbitals. Basis set includes d orbitals on sulfur. [b] Numbers in parentheses are based on the ZDO bond order matrix.

Table IV. HOMO's and LUMO's of $Ni(Et_2dtc)_2$[a]

MO	energy, au	symm	type
55	0.009	b_g	46.8% Ni_{xy} + 42.0% $S_{x,y}$
54	−0.027	b_g	50.8% C_z + 22.1% N_z + 15.4% S_z
53	−0.030	b_u	51.1% C_z + 22.9% N_z + 13.6% S_z
52	−0.313	a_g	64.2% S_z + 34.6% Ni_{yz}
51	−0.316	b_g	42.7% S_z + 37.9% Ni_{xz} + 15.3% N_z
50	−0.325	a_g	73.5% Ni_{z^2} + 14.8% Ni_s
49	−0.335	a_u	99.4% S_z
48	−0.343	b_u	79.0% $S_{x,y}$
47	−0.353	b_u	52.8% S_z + 36.1% N_z
46	−0.360	a_u	79.7% $S_{x,y}$ + 10.4% Ni_x

[a] Basis set includes d orbitals on sulfur.

Table V. HOMO's and LUMO's of $Ni(pdtc)_2$[a,b]

MO	energy, au	symm	type
49	−0.019	b_{1g}	47.2% Ni_{xy} + 41.9% $S_{x,y}$
48	−0.070	b_{2g}	47.3% C_z + 21.8% S_z + 16.2% C_z'
47	−0.074	b_{1u}	48.5% C_z + 19.2% S_z + 16.2% C_z'
46	−0.326	b_{3g}	94.8% C_z'
45	−0.326	a_u	99.4% C_z'
44	−0.336	b_{3g}	65.0% S_z + 28.9% Ni_{yz}
43	−0.341	b_{2g}	35.0% S_z + 32.3% Ni_{xz} + 16.3% N_z + 14.1% C_z'
42	−0.351	a_g	72.9% Ni_{z^2} + 14.3% Ni_s
41	−0.356	a_u	99.2% S_z
40	−0.367	b_{1u}	37.4% C_z' + 31.0% S_z + 30.4% N_z

[a] C′ refers to a C atom on the pyrrole ring. [b] Basis set includes d orbitals on sulfur.

Table VI. Calculated ΔE(SCF) Ionization Energies and Electron Affinities[a]

	no S d	inc S d	one-hole state
	IE, eV		
$Ni(Et_2dtc)_2$	6.39	6.53	^2A_g (Ni z^2)
$Ni(pdtc)_2$	7.01	7.18	^2A_g (Ni z^2)
	EA, eV		
$Ni(Et_2dtc)_2$	0.28	1.23	^2B_u (ligand)
	0.07	0.80	^2B_g (Ni xy)
$Ni(pdtc)_2$	1.48	2.43	$^2B_{1u}$ (ligand)
	0.77	1.62	$^2B_{1g}$ (Ni xy)

[a] A positive electron affinity corresponds to an exothermic electron attachment energy.

Because the π-bond index between the nickel and the sulfur atoms is very small and the indices are identical for both complexes, any difference in properties between the nickel(II) complexes of Et_2dtc^- and $pdtc^-$ cannot be attributed to differences in metal–ligand π-bonding. Previous CNDO/2 calculations on $Ni(Me_2dtc)_2$[10] indicated very little metal–ligand π-back-bonding, but significant $S(p_z) \rightarrow Ni(p_z)$ π-bonding was implicated.

Atomic charges on the sulfur, carbon, nitrogen, and nickel atoms are given in table II. Mulliken charges are much more sensitive to the inclusion of sulfur d orbitals than are the charges obtained from the bond order matrix. If resonance form C contributes more in Et_2dtc^- than in $pdtc^-$, one would expect the charge on N to be more positive in Et_2dtc^-. This is not supported by our calculations. Most significant is the similarity of charges in the two complexes. One would not expect a large difference in reactivity based on the calculated charges. There is also no significant difference in the atomic orbital populations on Ni or S in the two complexes (Table III). It is noteworthy that the populations of the d_{xz} and d_{yz} orbitals on the metal are close to 2.00, and the population of the Ni p_z orbital is close to zero, again indicating that there is little π-bonding between the metal and the ligand. This contrasts with the calculated orbital populations in bis(ethylene-1,2-dithiolato)nickel(II), $Ni(S_2C_2H_2)_2$, in which there is an appreciable loss of electron density in the d_{xz} orbital of the metal.[13]

The eigenvalues of the HOMO's and LUMO's of the two complexes are listed in Tables IV and V. The respective eigenvalues are all somewhat lower for $Ni(pdtc)_2$. This suggests that $Ni(Et_2dtc)_2$ is more readily oxidized and $Ni(pdtc)_2$ more readily reduced. No electrochemical data have been reported for $Ni(pdtc)_2$; however, $pdtc^-$ is known to stabilize low oxidation states in iron complexes.[3] Electrochemical studies indicate that $Ni(Et_2dtc)_2$ undergoes a quasi-reversible one-electron reduction to form $Ni(Et_2dtc)_2^-$.[17] Since the lowest energy virtual orbital contains no metal character in either system, one would predict that reduction of either complex would place the odd electron primarily on the ligand. ESR results on $Ni(Et_2dtc)_2^-$, however, indicate that the odd electron resides primarily on the metal.[18] Because of relaxation effects, the relative energies of the molecular orbitals in a Hartree–Fock calculation are often not a good indicator of the relative energies of the one-hole cation or anion states. This is particularly true when orbitals containing considerable metal d character are involved.[19] Therefore, ΔE(SCF) calculations were carried out to obtain the electron affinities of the complexes. The results, listed in Table VI, confirm that the electron affinity is higher for $Ni(pdtc)_2$, in agreement with

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qualitative considerations. Inclusion of sulfur d orbitals increases the calculated electron affinities by approximately 0.8 eV in both systems. In contrast to the ESR results, addition of an electron to a ligand orbital is favored by 0.2–0.4 eV over the addition of the electron to the Ni d_{xy} orbital in Ni(Et$_2$dtc)$_2$.[20] Since the geometry of the anion may be sensitive to the nature of the MO containing the odd electron, differential relaxation of geometry in the two anion states could cause the energies to change by the small amount necessary to alter the ground state of the anion.[21] On the basis of the calculated electron affinities, Ni(pdtc)$_2^-$ is less likely to contain the odd electron in a metal-based orbital than is Ni(Et$_2$dtc)$_2^-$.

Our ΔE(SCF) calculations confirm that Ni(Et$_2$dtc)$_2$ is more readily oxidized than Ni(pdtc)$_2$ (Table VI). The lowest energy one-hole state results from removal of the electron from a lower energy d_{z^2} orbital, not the highest occupied MO. This indicates that the complex Ni(Et$_2$dtc)$_2^+$ produced initially upon oxidation contains nickel(III). Ni(R$_2$dtc)$_2$ complexes are known to undergo irreversible oxidation to Ni(R$_2$dtc)$_3^+$ by means of nickel(III) intermediates.[17,22] It is not clear, however, whether direct oxidation to Ni(R$_2$dtc)$_2^+$ is important in the mechanism.

Our calculations suggest that neither complex will be susceptible to electrophilic decomposition. The two highest occupied MO's in Ni(pdtc)$_2$ are composed primarily of p_z orbitals on the carbon atoms of the pyrrole rings. Electrophilic attack involving these MO's does not directly affect the Ni–S fragment of the molecule. Furthermore, the HOMO's of Ni(Et$_2$dtc)$_2$ contain appreciable Ni–S antibonding character. Therefore, attack by an electrophile should *strengthen*, not weaken, the Ni–S bond in this system.

The LUMO's of Ni(Et$_2$dtc)$_2$ are composed primarily of C–N p_z antibonding character, whereas the N p_z orbital contributes much less to the LUMO of Ni(pdtc)$_2$. The C–S antibonding characters of the LUMO's are similar in the two complexes. Attack of a nucleophile should weaken the C–N bond of Ni(Et$_2$dtc)$_2$ but not affect the C–N bond of Ni(pdtc)$_2$.

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Appendix

Parameters were taken from ref 12 and 13 with the following exceptions: β_{3d}(Ni) = −28.0 eV; β_{3d}(S) = −4.0 eV; f_π = 0.610. The coordinates for Ni(Et$_2$dtc)$_2$ were taken from the published crystal structure[23] and idealized to C_{2h} symmetry. Bond lengths used were Ni–S = 2.20 Å, C–S = 1.706 Å, and C–N = 1.326 Å. The coordinates of the pdtc group in Ni(pdtc)$_2$ were based on the crystal structure of Fe(pdtc)$_3$.[4] The geometry of the complex was idealized to D_{2h} symmetry. Bond lengths used were Ni–S = 2.20 Å, C–S = 1.681 Å, and C–N = 1.378 Å. The same bond lengths were used in the free-ligand calculations as in the metal complexes. No geometry optimizations were performed. All calculations on the closed-shell systems were of the restricted Hartree–Fock (RHF) type. Unrestricted Hartree–Fock (UHF) calculations were performed on the open-shell systems. The odd electron was assigned to various molecular orbitals to obtain energies of different one-hole states. Reported energies involving UHF calculations do not include spin annihilation. Ionization energies and electron affinities are obtained from ΔE(SCF) calculations by taking the difference in total energy between the molecular ion and the neutral molecule.

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(20) There is some spin contamination in both UHF states of Ni(Et$_2$dtc)$_2^-$. The values of S^2 are 0.76 and 0.80 for the ^2B_g and ^2B_u states when no sulfur d orbitals are included and 0.78 and 0.80 when sulfur d orbitals are present in the calculation. However, the energy of the ^2B_g state is still 0.3 eV higher than that of the ^2B_u state even after spin annihilation.

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Synthesis and Coordination Chemistry of 2-(Diisopropoxyphosphino)pyridine *N,P*-Dioxide. Crystal and Molecular Structure of Bis[2-(diisopropoxyphosphino)pyridine *N,P*-dioxide]lanthanum Nitrate

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Ligands with donor oxygen atoms in 1,3-positions are normally found to be good chelating groups for hard transition metal ions, and classical examples are found in the family of acetylacetonate ligands. For the trivalent lanthanide and actinide ions, methylenediphosphonates and (carbamoylmethyl)phosphonates with two P=O or P=O and C=O groups in 1,3-positions have been found to form many coordination complexes, as well as display useful extraction applications.[2-6] Extending this concept, there has been interest in developing new families of ligands containing a P=O group and a functionality, X—O where X is N or S, since these combinations might be expected to show selective coordination properties. Following this reasoning, we recently reported on the synthesis of 2-(diethoxyphosphino)pyridine *N,P*-dioxide and 2-(diphenylphosphino)pyridine *N,P*-dioxide.[7] These ligands were found to form complexes with the uranyl ion, UO$_2$(NO$_3$)$_2$·L, in which L was bonded to uranium in a bidentate coordination mode. we report here on the preparation of 2-(diisopropoxyphosphino)pyridine *N,P*-dioxide and its coordination chemistry with the early lanthanide La(NO$_3$)$_3$.

Experimental Section

General Information. Pyridine *N*-oxide, dimethyl sulfate, *n*-butyllithium, and triisopropyl phosphite were purchased from Aldrich Chemical Co., and La(NO$_3$)$_3$·6H$_2$O was obtained from Alfa Products (Ventron). All reactions, unless specified otherwise, were performed under dry nitrogen in Schlenk flasks or in glovebags. Infrared spectra were recorded on a Nicolet Model 6000 FTIR spectrometer, and NMR spectra were measured on a Varian FT-80A spectrometer. NMR standards were 85% H$_3$PO$_4$ (^{31}P) and Me$_4$Si (^{1}H, ^{13}C).

Ligand Preparation. The synthesis employed was similar to that described previously for the ethoxy and phenyl analogues.[7] However, there are several important differences, and the procedure is outlined here. Pyridine *N*-oxide (26.0 g, 0.273 mol) in a 250-mL Schlenk flask equipped with a stir bar was submerged in a water bath maintained at 20 °C. Under a nitrogen purge, dimethyl sulfate (25.9 mL, 0.273 mol) was slowly added to the vigorously stirred pyridine *N*-oxide. After addition was complete (1 h), the water bath temperature was increased to 60 °C and held for 2 h. The resulting brown oil was transferred to a dropping

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