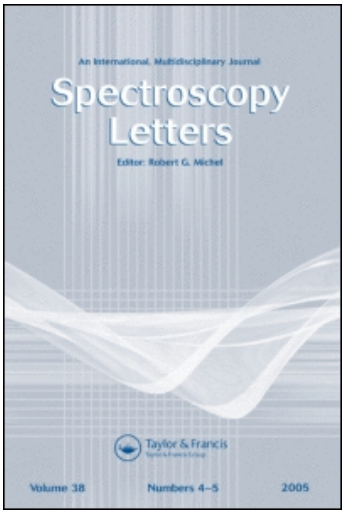

Spectroscopy Letters
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713597299

Infrared and Raman Spectra of Sulfur-Nitride Complexes of Nickel(II) and Palladium(II)

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To cite this Article Haworth, D. T. and Lin, G.(1979) 'Infrared and Raman Spectra of Sulfur-Nitride Complexes of Nickel(II) and Palladium(II)', Spectroscopy Letters, 12: 6, 451 — 465
To link to this Article: DOI: 10.1080/00387017908069172
URL: http://dx.doi.org/10.1080/00387017908069172

SPECTROSCOPY LETTERS, 12(6), 451-465 (1979)

INFRARED AND RAMAN SPECTRA OF SULFUR-NITRIDE COMPLEXES OF NICKEL(II) AND PALLADIUM(II)

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KEY WORDS: IR Spectra, Raman Spectra, Sulfur Nitride Complexes, Normal Coordinate Analysis

ABSTRACT

The infrared and Raman spectra of *trans*-$Ni(S_2N_2CH_3)_2$ and $Pd(S_3N)_2$ were measured from 4000-200 cm^{-1}. The absorption bands were assigned by comparison to the sulfur nitride complexes of nickel(II), palladium(II) and platinum(II). Normal coordinate analyses on these complexes were carried on these data using molecular parameters taken from X-ray data. To aid in band assignments, isotope shift data on *trans*-$^{62}Ni(S_2N_2CH_3)_2$ have also been carried out.

INTRODUCTION

In a previous paper we have reported on the infrared and Raman spectra of sulfur nitride square planar complexes of the type $M(S_2N_2H)_2$ with M is nickel, palladium and platinum (1). It was shown by the normal coordinate analyses that the vibrational modes associated with the sulfur nitrogen rings were strongly coupled. This data was also used to support an infrared study of the $Pt(S_2N_2H)_2$, $Pt(S_2N_2H)(S_3N)$ and $Pt(S_3N)_2$ complexes (2). This paper reports on two other sulfur nitride complexes, *trans*-$Ni(S_2N_2CH_3)_2$ and $Pd(S_3N)_2$, and makes use of the normal coordinate analysis technique combined with isotope shift data on $^{62}Ni(S_2N_2CH_3)_2$ to support assignments.

EXPERIMENTAL

1. *trans*-$Ni(S_2N_2CH_3)_2$

Trans-$Ni(S_2N_2CH_3)_2$ was prepared by reacting $Ni(S_2N_2H)_2$ with KOH and methyl iodide in tetrahydrofuran (THF) (3). The $Ni(S_2N_2H)_2$ was prepared as previously described (4,5). The THF was removed on a rotary evaporator and the residue was extracted with hot benzene. The combined benzene extracts were placed on an acid washed alumina column and the first eluate (green) contained sulfur and S_4N_4. This was followed by a red fraction, $Ni(S_2N_2H)(S_3N)$, and then after *ca.* 3 hr a green fraction, $Ni(S_2N_2CH_3)_2$. A blue fraction, $Ni(S_2N_2CH_3)(S_2N_2H)$ and a violet fraction, $Ni(S_2N_2H)_2$ were eluted with acetone. The blue and green

fractions were further purified by chromatography followed by recrystallization from benzene. $Ni(S_2N_2CH_3)_2$, mp 194°C (lit. mp 194°C(3)); $Ni(S_2N_2CH_3)(S_2N_2H)$, mp 144°C (lit. mp 144°C(3)).

The $^{62}Ni(S_2N_2CH_3)_2$ was prepared from $^{62}Ni(S_2N_2H)_2$, whose synthesis was previously described (1).

2. $Pd(S_3N)_2$

This compound was prepared by reacting $PdCl_2$ and S_4N_4 in methanol (6). After removal of the solvent followed by washing with water to remove chloride ion the product was dried in *vacuo*. The dry solid was taken up in cyclohexane and chromatographed on a *silica gel-G* column. The eluted blue solution was evaporated to *ca.* 5 mL and after *ca.* 12 hr $Pd(S_3N)_2$ precipitated, mp 178°C (lit. mp 179°C (6)).

3. Spectral measurements

The infrared of the compounds were recorded on a Perkin-Elmer FIS-3 far i.r. spectrophotometer from 410-33 cm^{-1} as nujol mulls on a polyethylene plate. The compounds were also run on a Beckman IR-12 i.r. spectrophotometer from 4000-200 cm^{-1} using the KBr pellet technique. Raman spectra were recorded on a Spex Model 1401 double monochrometer equipped with an ITT FW-130 phototube.

NORMAL COORDINATE ANALYSES

1. *trans*-$Ni(S_2N_2CH_3)_2$

The normal coordinate analysis on this complex was carried out on a model of C_{2h} symmetry. The molecular parameters used

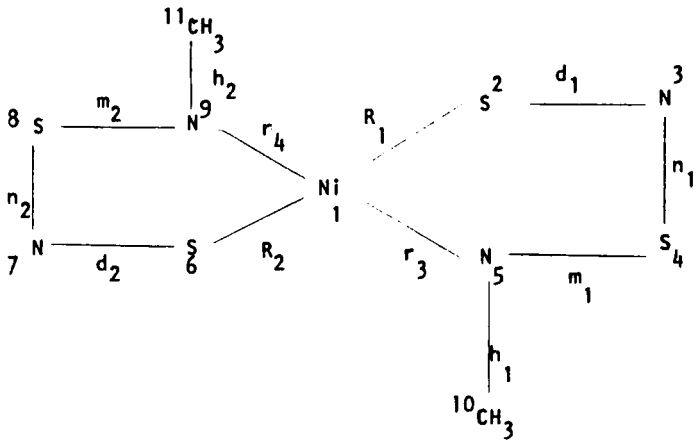
for this NCA were taken from the results of X-ray analysis (3). The twenty-seven normal vibrations can be classified into 9Ag+3Bg+5Au+10Bu; the Ag and Bg are Raman active whereas the Au and Bu modes are infrared active. In this treatment we have included all in-plane (Ag and Bu) vibrations. The programs written by Schachtschneider (7) were used in this work. The twenty-eight in-plane internal coordinates are shown in Table 1. The out-of-plane and torsion coordinates are not shown. These twenty-eight internal coordinates can be divided into twelve stretching and sixteen bending modes. The symmetry coordinates for *trans*-$Ni(S_2N_2CH_3)_2$ are shown in Table 2. Seven redundancies are included in the twenty-six symmetry coordinates. They were not easily eliminated and were included in the NCA calculations. Final results gave zero frequencies corresponding to these redundancies. The calculated force constants; stretching (K), bending (H) and repulsive (F) are displayed in Table 3. The interaction force constants (f) are not shown. Table 4 lists the observed and calculated spectra for *trans*-$Ni(S_2N_2CH_3)_2$ and its ^{62}Ni analog. Shown also are the potential energy distribution (PED) and the predominant modes.

2. $Pd(S_3N)_2$

The normal coordinate analysis on this planar complex was carried out on a model of C_{2v} symmetry. The molecular parameters used for the NCA were taken from X-ray analysis (8). The twenty-

TABLE I

Internal Coordinates for $Ni(S_2N_2CH_3)_2$



2-1-9	π_1	3-4-5	β_1
6-1-5	π_2	7-8-9	β_2
2-1-5	θ_1	4-5-1	α_1
6-1-9	θ_2	8-9-1	α_2
1-2-3	δ_1	4-5-10	ϕ_1
1-6-7	δ_2	8-9-11	ϕ_2
2-3-4	γ_1	1-5-10	ρ_1
6-7-8	γ_2	1-9-11	ρ_2

TABLE 2

Symmetry Coordinates for $Ni(S_2N_2CH_3)_2$

Ag:	$S_1 = \Delta R_1 + \Delta R_2$	$S_8 = \Delta\pi_1 + \Delta\pi_2 - \Delta\theta_1 - \Delta\theta_2$
	$S_2 = \Delta r_3 + \Delta r_4$	$S_9 = \Delta\delta_1 + \Delta\delta_2$
	$S_3 = \Delta d_1 + \Delta d_2$	$S_{10} = \Delta\gamma_1 + \Delta\gamma_2$
	$S_4 = \Delta m_1 + \Delta m_2$	$S_{11} = \Delta\beta_1 + \Delta\beta_2$
	$S_5 = \Delta n_1 + \Delta n_2$	$S_{12} = \Delta\alpha_1 + \Delta\alpha_2 - \Delta\phi_1 - \Delta\phi_2 - \Delta\rho_1 - \Delta\rho_2$
	$S_6 = \Delta h_1 + \Delta h_2$	$S_{13} = \Delta\phi_1 - \Delta\rho_2 - \Delta\rho_1 + \Delta\phi_2$
	$S_7 = \Delta\pi_1 + \Delta\pi_2 + \Delta\theta_1 + \Delta\theta_2$	
Bu:	$S_{14} = \Delta R_1 - \Delta R_2$	$S_{21} = \Delta\pi_1 - \Delta\pi_2 - \Delta\theta_1 + \Delta\theta_2$
	$S_{15} = \Delta r_3 - \Delta r_4$	$S_{22} = \Delta\delta_1 - \Delta\delta_2$
	$S_{16} = \Delta d_1 - \Delta d_2$	$S_{23} = \Delta\gamma_1 - \Delta\gamma_2$
	$S_{17} = \Delta m_1 - \Delta m_2$	$S_{24} = \Delta\beta_1 - \Delta\beta_2$
	$S_{18} = \Delta n_1 - \Delta n_2$	$S_{25} = \Delta\alpha_1 - \Delta\alpha_2 - \Delta\phi_1 + \Delta\phi_2 - \Delta\rho_1 + \Delta\rho_2$
	$S_{19} = \Delta h_1 - \Delta h_2$	$S_{26} = \Delta\phi_1 + \Delta\rho_2 - \Delta\rho_1 - \Delta\phi_2$
	$S_{20} = \Delta\pi_1 - \Delta\pi_2 + \Delta\theta_1 - \Delta\theta_2$	

TABLE 3

Force Constants (mdy/Å) for $Ni(S_2N_2CH_3)_2$

K(Ni-S)	1.05	H(N-S-N)	0.50
K(Ni-N)	0.60	H(Ni-N-S)	0.20
$K(S_1-N_1)$	5.40	H(S-N-C)	0.35
$K(S_2-N_2)$	3.32	H(Ni-N-C)	0.09
$K(N_1-S_2)$	2.65	F(S-S)	0.40
K(N-C)	6.20	F(N-N)	0.40
H(S-Ni-N)	0.15	F(Ni-S)	0.40
H(S-Ni-N)	0.13	F(S-C)	0.55
H(Ni-S-N)	0.30	F(Ni-C)	0.40
H(S-N-S)	0.80		

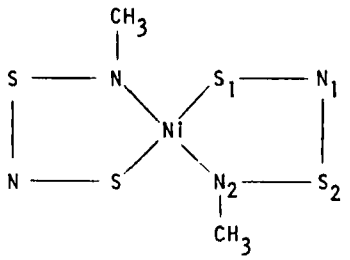


TABLE 4

Comparison of Observed and Calculated Frequencies and Band Assignments for $Ni(S_2N_2CH_3)_2$ (cm^{-1})

		Observed	Δν*	Calculated	Δν*	Predominant mode	Potential energy distribution (%)
Ag:	ν_1	1262	0.0	1266.0	0.0	$\nu(N\text{-}CH_3)$	$S_6(90)$
(R)	ν_2	1023	0.0	1026.5	0.0	$\nu(S_1\text{-}N_1)+\nu(N_1\text{-}S_2)$	$S_3(83)+S_5(12)$
	ν_3	824	0.0	831.1	0.0	$\nu(S_2\text{-}N_2)+\delta(S\text{-}N\text{-}CH_3)$	$S_4(72)+S_{13}(14)$
	ν_4	717	0.0	715.1	0.0	$\nu(N_1\text{-}S_2)+\delta(S\text{-}N\text{-}S)$	$S_5(68)+S_{10}(18)$
	ν_5	463	0.0	459.3	0.0	$\delta(S\text{-}N\text{-}S)+\delta(N\text{-}S\text{-}N)+\delta(Ni\text{-}N\text{-}S)+\delta(S\text{-}N\text{-}CH_3)$	$S_{10}(20)+S_{11}(28)+S_{12}(22)+S_{13}(11)$
	ν_6	382	0.0	379.6	0.0	$\nu(Ni\text{-}N)+\delta(S\text{-}N\text{-}S)+\delta(S\text{-}N\text{-}CH_3)$	$S_2(18)+S_{10}(16)+S_{13}(44)$
	ν_7	297	0.0	294.5	0.0	$\nu(Ni\text{-}S)+\delta(N\text{-}S\text{-}N)+\delta(Ni\text{-}N\text{-}S)+\delta(S\text{-}N\text{-}CH_3)$	$S_1(27)+S_{11}(13)+S_{12}(15)+S_{13}(27)$
	ν_8	-	-	216.0	0.0	$\nu(Ni\text{-}S)+\nu(Ni\text{-}N)$	$S_1(43)+S_2(37)$
	ν_9	-	-	156.6	0.0	$\nu(Ni\text{-}S)+\nu(Ni\text{-}N)+\delta(S\text{-}Ni\text{-}N)+\delta(Ni\text{-}N\text{-}S)$	$S_1(11)+S_2(23)+S_8(32)+S_{12}(18)$

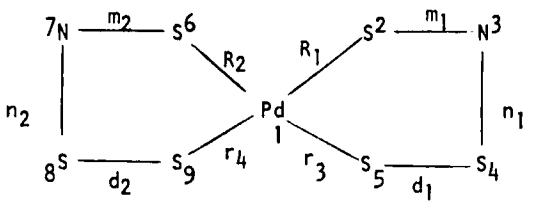
Bu:	ν_{10}	1268	0.0	1266.0	0.0	$\nu(N\text{-}CH_3)$	$S_{19}(90)$
(IR)	ν_{11}	1032	0.0	1026.6	0.0	$\nu(S_1\text{-}N_1)+\nu(N_1\text{-}S_2)$	$S_{16}(83)+S_{18}(12)$
	ν_{12}	834	0.0	831.6	0.0	$\nu(S_2\text{-}N_2)+\delta(S\text{-}N\text{-}CH_3)$	$S_{17}(72)+S_{26}(14)$
(IR)	ν_{13}	710	0.0	714.9	0.0	$\nu(N_1\text{-}S_2)+\delta(S\text{-}N\text{-}S)$	$S_{18}(67)+S_{23}(18)$
	ν_{14}	465	0.0	466.3	0.45	$\nu(Ni\text{-}N)+\delta(S\text{-}N\text{-}S)+\delta(N\text{-}S\text{-}N)+\delta(Ni\text{-}N\text{-}S)$	$\nu_{15}(10)+S_{23}(17)+S_{24}(25)+S_{25}(23)$
	ν_{15}	400	2.0	402.2	2.19	$\nu(Ni\text{-}S)+\nu(Ni\text{-}N)+\delta(S\text{-}N\text{-}S)+\delta(S\text{-}N\text{-}CH_3)$	$S_{14}(15)+S_{15}(37)+S_{23}(13)+S_{26}(24)$
	ν_{16}	321	3.0	323.0	4.40	$\nu(Ni\text{-}S)+\nu(Ni\text{-}N)$	$S_{14}(42)+S_{15}(35)$
	ν_{17}	310	1.0	311.3	1.25	$\nu(Ni\text{-}S)+\delta(N\text{-}S\text{-}N)+\delta(S\text{-}N\text{-}CH_3)$	$S_{14}(30)+S_{24}(12)+S_{26}(43)$
	ν_{18}	249	2.7	248.7	2.86	$\delta(S\text{-}Ni\text{-}N)+\delta(Ni\text{-}S\text{-}N)+\delta(S\text{-}N\text{-}S)+\delta(Ni\text{-}N\text{-}S)$	$S_{20}(12)+S_{22}(10)+S_{23}(14)+S_{25}(36)$
	ν_{19}	-	-	90.4	0.61	$\delta(S\text{-}Ni\text{-}N)+\delta(S\text{-}Ni\text{-}N)$	$S_{20}(54)+S_{21}(38)$

$\Delta\nu^*$ $=\Delta(^{NA}Ni - {}^{62}Ni)$ NA - natural abundance

one normal modes are classified as 8 A_1 + 3 A_2 + 3 B_1 + 7 B_2 and all modes are infrared and Raman active except A_2 which is only Raman active. In this treatment only the in-plane (A_1 and B_2) modes were considered and these twenty-two internal coordinates are shown in Table 5. The twenty-one symmetry coordinates as shown in Table 6 include five redundancies which were not easily

TABLE 5

Internal Coordinates for $Pd(S_3N)_2$



2-1-6 = Π_1	2-3-4 = γ_1
5-1-9 = Π_2	6-7-8 = γ_2
2-1-5 = θ_1	3-4-5 = β_1
6-1-9 = θ_2	7-8-9 = β_2
1-2-3 = δ_1	4-5-1 = α_1
1-6-7 = δ_2	8-9-1 = α_2

TABLE 6

Symmetry Coordinates for $Pd(S_3N)_2$

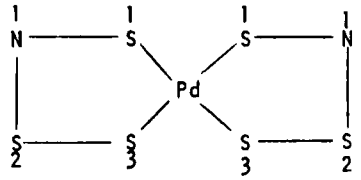
A_1:	$S_1=\Delta R_1+\Delta R_2$	$S_6=\Delta\Pi_1+\Delta\Pi_2-\Delta\theta_1-\Delta\theta_2$
	$S_2=\Delta r_3+\Delta r_4$	$S_7=\Delta\delta_1+\Delta\delta_2$
	$S_3=\Delta m_1+\Delta m_2$	$S_8=\Delta\gamma_1+\Delta\gamma_2$
	$S_4=\Delta n_1+\Delta n_2$	$S_9=\Delta\beta_1+\Delta\beta_2$
	$S_5=\Delta d_1+\Delta d_2$	$S_{10}=\Delta\alpha_1+\Delta\alpha_2$
B_2:	$S_{11}=\Delta R_1-\Delta R_2$	$S_{17}=\Delta\Pi_1-\Delta\Pi_2-\Delta\theta_1+\Delta\theta_2$
	$S_{12}=\Delta\gamma_3-\Delta r_4$	$S_{18}=\Delta\delta_1-\Delta\delta_2$
	$S_{13}=\Delta m_1-\Delta m_2$	$S_{19}=\Delta\gamma_1-\Delta\gamma_2$
	$S_{14}=\Delta n_1-\Delta n_2$	$S_{20}=\Delta\beta_1-\Delta\beta_2$
	$S_{15}=\Delta d_1-\Delta d_2$	$S_{21}=\Delta\alpha_1-\Delta\alpha_2$
	$S_{16}=\Delta\Pi_1-\Delta\Pi_2+\Delta\theta_1-\Delta\theta_2$	

eliminated and were included in the calculations. These gave zero frequencies in the final results. The calculated force constants; stretching (K), bending (H) and repulsive (F) are shown in Table 7. The interaction force constants (f) are not shown. Table 8 shows the infrared and Raman spectra for $Pd(S_3N)_2$, the predominant modes as well as the percent potential energy distribution (PED).

TABLE 7

Force Constants (mdy/Å) for $Pd(S_3N)_2$

$K(Pd\text{-}S_1)$	1.08	H(S-N-S)	0.73
$K(Pd\text{-}S_3)$	0.98	H(N-S-S)	0.90
$K(S_1\text{-}N_1)$	5.50	H(Pd-S-S)	0.30
$K(N_1\text{-}S_2)$	2.68	$F(S_1\text{-}S_1)$	0.76
$K(S_2\text{-}S_3)$	1.52	$F(S_1\text{-}S_3)$	0.29
H(S-Pd-S)	0.90	F(Pd-N)	0.10
$H(S_1\text{-}Pd\text{-}S_3)$	0.90	$F(S_1\text{-}S_2)$	0.42
H(Pd-S-N)	0.20	F(N-S)	0.20
		F(Pd-S)	0.40



RESULTS AND DISCUSSION

Replacement of the N-H hydrogens of *cis*-$Ni(S_2N_2H)_2$ with methyl groups results in a *trans*-$Ni(S_2N_2CH_3)_2$ (See figure in Table 3). Two of the three ring S-N stretching modes are similiar to those found in *cis*-$Ni(S_2N_2H)_2$: $\nu(S_1\text{-}N_1)$, 1032 cm^{-1} and

TABLE 8

Comparison of Observed and Calculated Frequencies and Band Assignments for $Pd(S_3N)_2$ (cm^{-1})

		Observed	Calculated	Predominant mode	Potential energy distribution (%)
A_1:	ν_1	1060	1059.9	$\nu(S_1\text{-}N_1)$	$S_3(81)$
(R)	ν_2	700	701.7	$\nu(N_1\text{-}S_2)$	$S_4(68)$
	ν_3	515	512.4	$\nu(S\text{-}S)+\delta(S\text{-}Pd\text{-}S)+\delta(N\text{-}S\text{-}S)$	$S_5(45)+S_6(15)+S_9(16)$
	ν_4	444	444.0	$\nu(S\text{-}S)+\delta(S\text{-}N\text{-}S)+\delta(N\text{-}S\text{-}S)+\delta(Pd\text{-}S\text{-}S)$	$S_5(25)+S_8(19)+S_9(14)+S_{10}(11)$
	ν_5	350	353.3	$\nu(Pd\text{-}S_3)+\delta(S\text{-}Pd\text{-}S)+\delta(Pd\text{-}S\text{-}S)$	$S_2(54)+S_6(10)+S_{10}(13)$
	ν_6	335	335.7	$\nu(Pd\text{-}S_1)+\nu(Pd\text{-}S_3)$	$S_1(40)+S_2(40)$
	ν_7	220	220.6	$\nu(Pd\text{-}S_1)+\nu(Pd\text{-}S_3)$	$S_1(35)+S_2(55)$
	ν_8	-	9.4	$\delta(Pd\text{-}S\text{-}N)+\delta(Pd\text{-}S\text{-}S)$	$S_7(29)+S_{10}(54)$
B_2:	ν_9	1050	1050.6	$\nu(S_1\text{-}N_1)$	$S_{13}(81)$
(IR)	ν_{10}	700	698.3	$\nu(N_1\text{-}S_2)$	$S_{14}(67)$
	ν_{11}	474	476.2	$\nu(N_1\text{-}S_2)+\nu(S\text{-}S)+\delta(S\text{-}N\text{-}S)+\delta(N\text{-}S\text{-}S)$	$S_{14}(11)+S_{15}(36)+S_{19}(14)+S_{20}(24)$
	ν_{12}	394	396.6	$\nu(S\text{-}S)+\delta(S\text{-}N\text{-}S)+\delta(Pd\text{-}S\text{-}S)$	$S_{15}(55)+S_{19}(13)+S_{21}(12)$
	ν_{13}	354	351.5	$\nu(Pd\text{-}S_1)+\nu(Pd\text{-}S_3)$	$S_{11}(69)+S_{12}(22)$
	ν_{14}	272	269.3	$\nu(Pd\text{-}S_1)+\delta(S\text{-}Pd\text{-}S)+\delta(S_1\text{-}Pd\text{-}S3)+\delta(Pd\text{-}S\text{-}S)$	$S_{11}(11)+S_{16}(28)+S_{17}(28)+S_{21}(18)$
	ν_{15}	248	250.1	$\nu(Pd\text{-}S_3)+\delta(S\text{-}Pd\text{-}S)$	$S_{12}(13)+S_{16}(49)$
	ν_{16}	191	189.6	$\nu(S\text{-}S)+\delta(S\text{-}Pd\text{-}S)+\delta(S_1\text{-}Pd\text{-}S_3)$	$S_{15}(22)+S_{16}(33)+S_{17}(33)$

$\nu(N_1\text{-}S_2)$, 717 cm^{-1}. The $\nu(N_2\text{-}S_2)$ band is shifted from 890 cm^{-1} in *cis*-$Ni(S_2N_2H)_2$ to 834 cm^{-1} in *trans*-$Ni(S_2N_2CH_3)_2$. Most bands in *trans*-$Ni(S_2N_2CH_3)_2$ as well as in *cis*-$Ni(S_2N_2H)_2$ are strongly coupled. The metal isotope substitution $^{NA}Ni/^{62}Ni$ is expected to shift all the metal-nitrogen and metal-sulfur stretching modes.

It is particularly significant that the calculations due to ^{62}Ni isotope substitution are in good agreement with those observed for the Ni-N and Ni-S stretching and skeletals bending modes (Table 4). Band assignments for the observed frequencies and isotopic shift are in good agreement and show an average error of less than one percent.

For planar $Pd(S_3N)_2$ (See figure in Table 7) the ring S-N assignments can be compared to our previous synthesis and infrared study of $Pt(S_3N)_2$ (2). In the platinum complex $\nu(S_1\text{-}N_1)$ is shifted from 1025 cm^{-1} to 1050 cm^{-1} in the palladium complex. The $\nu(N_1\text{-}S_2)$ vibration shows a small change shift, 712 cm^{-1} for platinum to 700 cm^{-1} for the palladium complex. While no NCA was carried out on $Pt(S_3N)_2$, the $Pd(S_3N)_2$ complex shows strong coupling for most of the stretching and bending modes. Band assignments show an average error of 0.05 percent.

ACKNOWLEDGEMENT

This work was supported in part by the Marquette University Committee on Research. G. Lin acknowledges support from the Arthur J. Schmitt Foundation and we are grateful to Professor

H. J. Emeleus for his interest in this work while he was a senior NSF fellow at Marquette University.

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Received 4-4-79
Accepted 5-1-79