

# Notes

CONTRIBUTION FROM ISTITUTO DI CHIMICA GENERALE ED INORGANICA, UNIVERSITY OF PADUA, PADUA, ITALY, AND ISTITUTO DI CHIMICA GENERALE, UNIVERSITY OF MESSINA, MESSINA, ITALY

## New Five-Coordinate Complexes of Cobalt(II) with Phenyl-diethoxyphosphine

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A number of five-coordinated low-spin complexes of cobalt(II) with tertiary and di(tertiary phosphines),  $[\text{Co}(\text{PR}_3)_3\text{X}_2]$  ( $\text{X}^- = \text{CN}^-$  and  $\text{NCS}^-$ ), with the donor atom set  $\text{P}_3\text{X}_2$  have been recently prepared and characterized.<sup>2</sup> The stability of such complexes has been attributed to the presence of good  $\pi$ -bonding anionic ligands which enhance the electrophilicity of the metal atom with respect to the neutral phosphine groups which are considered essentially  $\sigma$ -bonding ligands.<sup>2</sup>

Sacco and Gorrieri<sup>3</sup> reported a variety of complexes of the types  $[\text{Co}(\text{Ph}_2\text{P}(\text{CH}_2)_2\text{PPh}_2)_2]\text{XY}$  ( $\text{X}^- = \text{Cl}^-$ ,  $\text{Br}^-$ ,  $\text{I}^-$ ,  $\text{NCS}^-$ ,  $\text{NO}_3^-$ ;  $\text{Y}^- = \text{Br}^-$ ,  $\text{I}^-$ ,  $\text{NCS}^-$ ,  $\text{NO}_3^-$ ,  $\text{ClO}_4^-$ ,  $\text{BPh}_4^-$ ) which are probably five-coordinated and Venanzi and coworkers<sup>4</sup> have prepared five-coordinated cobalt(II) complexes of the type  $[\text{Co}(\text{QP})\text{X}]\text{Y}$  ( $\text{X}^- = \text{Cl}^-$ ,  $\text{Br}^-$ ,  $\text{I}^-$ ,  $\text{NCS}^-$  and  $\text{NO}_3^-$ ;  $\text{Y}^- = \text{ClO}_4^-$  and  $\text{BPh}_4^-$ ) where a trigonal-bipyramidal structure is imposed by the rigid tetradentate ligand, QP, tris(*o*-diphenylphosphinophenyl)phosphine. These seem to be the only compounds of cobalt(II) with the  $\text{P}_4\text{X}$  set of donor atoms and without strong  $\pi$ -bonding groups which have hitherto been prepared.

As an extension of our work<sup>5</sup> on the ability of phenyl-diethoxyphosphine,  $\text{PhP}(\text{OC}_2\text{H}_5)_2$ , to stabilize low oxidation states of transition metals, we have synthesized the new five-coordinated complexes  $[\text{Co}(\text{PhP}(\text{OC}_2\text{H}_5)_2)_4\text{X}]\text{BPh}_4$  ( $\text{X}^- = \text{Cl}^-$ ,  $\text{Br}^-$ ,  $\text{I}^-$ ,  $\text{N}_3^-$ , and  $\text{NCS}^-$ ) and the compound  $[\text{Co}(\text{PhP}(\text{OC}_2\text{H}_5)_2)_3(\text{NCS})_2]$  in which some of the anionic ligands are essentially  $\sigma$ -bonding groups. The chief aim was to confirm the idea that  $\text{PhP}(\text{OC}_2\text{H}_5)_2$  is a better  $\pi$ -bonding ligand than alkyl or aryl phosphines.

### Experimental Section

**Apparatus.** All the preparative work was carried out under nitrogen. The solvents were purified and dried by standard methods and distilled in a current of nitrogen just prior to the use. The complexes were kept in stoppered vials under inert atmosphere. Magnetic susceptibilities were measured on solid

samples by Gouy standard techniques using  $\text{Hg}[\text{Co}(\text{NCS})_4]$  as a calibrant.<sup>6</sup> The susceptibilities were corrected for the diamagnetism of the ligands from tables given by Figgis and Lewis.<sup>7</sup> Solution susceptibilities were measured by the  $^1\text{H}$  nmr method of Evans<sup>8</sup> using the solvent, 1,2-dichloroethane, as internal reference and a Varian A-60A at 40°. The infrared spectra were recorded by a Perkin-Elmer model 621 spectrophotometer using Nujol mulls or KBr pellets. Conductivity measurements were carried out with a LKB conductivity bridge on  $10^{-3}$  M solutions of the complexes. Electronic spectra were recorded in the range 5000–22,000  $\text{cm}^{-1}$  at room temperature with a Beckman DK-2A recording spectrophotometer. Visible and near-infrared mull spectra were obtained from Nujol mulls supported on filter paper.

Phenyl-diethoxyphosphine (diethyl ester of phenylphosphonous acid),  $\text{PhP}(\text{OC}_2\text{H}_5)_2$ , was prepared by the method of Rabinowitz and Pellon.<sup>9</sup> The cobalt(II) halides and the thiocyanate were obtained as anhydrous salts and used without further purification. All the other salts were reagent grade chemicals.

**Preparations.**—The complexes of the type  $[\text{Co}(\text{PhP}(\text{OC}_2\text{H}_5)_2)_4\text{X}]\text{BPh}_4$  ( $\text{X}^- = \text{Cl}^-$ ,  $\text{Br}^-$ ,  $\text{I}^-$ , and  $\text{NCS}^-$ ) were prepared by the same general method. The appropriate anhydrous cobalt(II) salts (10 mmol) and  $\text{PhP}(\text{OC}_2\text{H}_5)_2$  (9.0 g, 45 mmol) were placed in 50 ml of anhydrous ethanol at room temperature and the dark-green mixture was stirred for 0.5 hr. Sodium tetraphenylboron (3.42 g, 10 mmol) was then slowly added under stirring and the precipitate, which formed immediately, was filtered off and recrystallized by dissolving it in the minimum amount of 1,2-dichloroethane and adding slowly anhydrous ethanol to the filtered solution. The green or dark-green crystals were collected by filtration, washed with ethyl ether or pentane, and dried in a stream of nitrogen.

$[\text{Co}(\text{Ph}(\text{OC}_2\text{H}_5)_2)_4\text{N}_3]\text{BPh}_4$ .—To a saturated methanolic solution of anhydrous cobalt(II) nitrate (1.73 g, 10 mmol) were slowly added at room temperature and under stirring sodium azide (0.65 g, 10 mmol) and sodium tetraphenylboron (3.42 g, 10 mmol) dissolved in the minimum amount of anhydrous methanol. After standing overnight the precipitation of  $\text{NaNO}_2$  was complete. The mother liquor was filtered and phenyl-diethoxyphosphine (9.0 g, 45 mmol) was added to give a dark-green solution. The brown-green needles of  $[\text{Co}(\text{PhP}(\text{OC}_2\text{H}_5)_2)_4\text{N}_3]\text{BPh}_4$  formed after a few hours were collected on a sintered-glass filter, washed with ethyl ether or pentane, and dried in a stream of nitrogen.

$[\text{Co}(\text{PhP}(\text{OC}_2\text{H}_5)_2)_3(\text{NCS})_2]$ .—Anhydrous cobalt(II) thiocyanate (1.75 g, 10 mmol) was placed in 80 ml of absolute ethanol and phenyl-diethoxyphosphine (6.2 g, 31 mmol) was added. The green solution, after standing at room temperature for two days, deposited the complex as green crystals. A further crop can be recovered on evaporating the mother liquor *in vacuo*.

### Results and Discussion

The complexes are all dark-green solids which can be prepared in 90–95% yields and which are quite stable under inert atmosphere. If exposed to air an oil appears at the surface of the crystals after a week indicating some decomposition. They are soluble both in polar and in nonpolar organic solvents such as acetonitrile, nitrobenzene, acetone, and 1,2-dichloroethane to give green solutions which, on contact with the air, become blue after a few hours.

Good analytical data have been obtained (Table I) even without recrystallization of the products. The equivalent conductivities of  $10^{-3}$  M solutions in nitrobenzene lie in the range 17.3–18.0  $\text{cm}^{-2}$   $\text{ohm}^{-1}$   $M^{-1}$  and are characteristic of 1:1 electrolytes. Similar values have been reported by other authors for  $[\text{Co}$

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TABLE I  
 ANALYSIS AND PROPERTIES OF SOME  $[\text{Co}(\text{PhP}(\text{OC}_2\text{H}_5)_2)_4\text{X}]\text{BPh}_4$  COMPLEXES

Compound	Color	Dec pt, <sup>a</sup> °C	$\Delta,^b$ $\text{cm}^2$ $\text{ohm}^{-1}$ $M^{-1}$	C, %		H, %		Others, %		
				Calcd	Found	Calcd	Found	Calcd	Found	
$[\text{Co}(\text{PhP}(\text{OC}_2\text{H}_5)_2)_4\text{Cl}]\text{BPh}_4^c$	Green	93-94	17.5	63.72	63.60	6.63	6.52	Cl	2.94	3.04
$[\text{Co}(\text{PhP}(\text{OC}_2\text{H}_5)_2)_4\text{Br}]\text{BPh}_4^d$	Green	104	17.4	61.45	61.35	6.40	6.42	Br	6.40	6.44
$[\text{Co}(\text{PhP}(\text{OC}_2\text{H}_5)_2)_4\text{I}]\text{BPh}_4$	Dark-green	88-89	17.3	59.22	59.02	6.17	6.13	I	9.78	9.83
$[\text{Co}(\text{PhP}(\text{OC}_2\text{H}_5)_2)_4\text{N}_3]\text{BPh}_4$	Green	61-63	18.0	63.37	63.23	6.60	6.55	N	3.46	3.36
$[\text{Co}(\text{PhP}(\text{OC}_2\text{H}_5)_2)_4\text{NCS}]\text{BPh}_4^e$	Green	87-88	17.6	63.52	63.43	6.51	6.53	N	1.14	1.21
$[\text{Co}(\text{PhP}(\text{OC}_2\text{H}_5)_2)_3(\text{NCS})_2]^f$	Brown-green	91-92		49.93	49.81	5.85	5.86	N	3.64	3.63

<sup>a</sup> All the decomposition points are uncorrected. <sup>b</sup> For  $10^{-3} M$  solutions in nitrobenzene at 25°. <sup>c</sup> Calcd: P, 10.28. Found: P, 10.16. <sup>d</sup> Calcd: P, 9.92. Found: P, 9.78. <sup>e</sup> Calcd: S, 2.61. Found: S, 2.68. <sup>f</sup> Calcd: S, 8.32. Found: S, 8.48.

 TABLE II  
 MAGNETIC AND ELECTRONIC ABSORPTION DATA FOR SOME  $[\text{Co}(\text{PhP}(\text{OC}_2\text{H}_5)_2)_4\text{X}]\text{BPh}_4$  COMPLEXES

Compound	Solution	$\mu_{\text{eff}},^a$ BM	Band maxima, $\text{cm}^{-1}{}^b$			
			7700 (75)	12,300 sh	15,200 (490)	16,900 (570)
$[\text{Co}(\text{PhP}(\text{OC}_2\text{H}_5)_2)_4\text{Cl}]\text{BPh}_4$	Solution	2.32	7700 (75)	12,300 sh	15,200 (490)	16,900 (570)
	Solid	2.21	7700	12,300 sh	15,200	16,800
$[\text{Co}(\text{PhP}(\text{OC}_2\text{H}_5)_2)_4\text{Br}]\text{BPh}_4$	Solution	2.20	7400 (65)	11,500 sh	14,500 (475)	16,900 (650)
	Solid	2.15	7500	11,500 sh	14,600	16,700
$[\text{Co}(\text{PhP}(\text{OC}_2\text{H}_5)_2)_4\text{I}]\text{BPh}_4$	Solution	2.11	7200 (55)	11,300 sh	14,200 (430)	16,500 (590)
	Solid	2.13	7200	11,300 sh	14,100	16,300
$[\text{Co}(\text{PhP}(\text{OC}_2\text{H}_5)_2)_4\text{N}_3]\text{BPh}_4$	Solution	2.02	9500 (125)	11,600 sh	15,000 (410)	
	Solid		7900	11,700 sh	15,200	16,800 sh
$[\text{Co}(\text{PhP}(\text{OC}_2\text{H}_5)_2)_4\text{NCS}]\text{BPh}_4$	Solution	2.02	9000 (190)		15,000 (265)	17,400 (350)
	Solid	2.05	9000		14,900	17,800
$[\text{Co}(\text{PhP}(\text{OC}_2\text{H}_5)_2)_3(\text{NCS})_2]$	Solution	2.06	6550 (230)		15,100 (205)	23,400 (850)
	Solid		7500		16,100	

<sup>a</sup> Solid sample at 23° and solution at 40°. <sup>b</sup> Spectra measured at room temperature, molar extinction coefficients in parentheses.

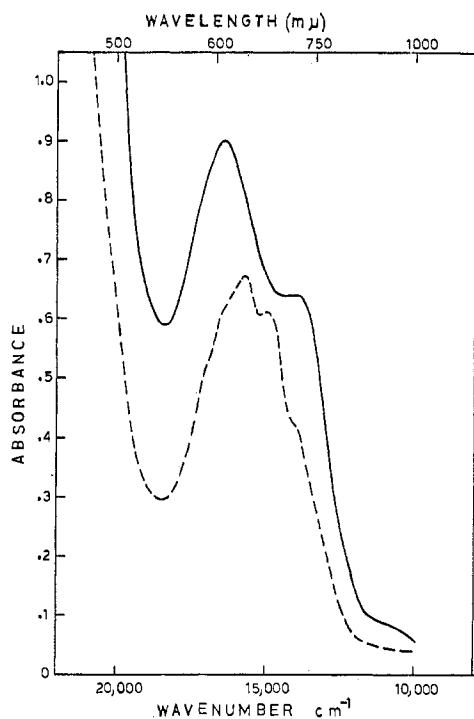


Figure 1.—Absorption spectra: —,  $[\text{Co}(\text{PhP}(\text{OC}_2\text{H}_5)_2)_4\text{I}]\text{BPh}_4$ ,  $1.5 \times 10^{-3} M$  solution in 1,2-dichloroethane containing  $10^{-1} M$   $\text{PhP}(\text{OC}_2\text{H}_5)_2$ ; - - - - - ,  $1.5 \times 10^{-3} M$  complex without free phenyldiethoxyphosphine.

$(\text{QP})\text{X}]\text{BPh}_4^4$  and  $[\text{Co}(\text{Ph}_2\text{P}(\text{CH}_2)_2\text{PPh}_2)_2\text{X}]\text{Y}^3$  complexes.

The magnetic moment data, reported in Table II, range from 2.00 to 2.30 BM, which are intermediate between those of low-spin octahedral (1.73–2.0 BM) and square-planar (2.3–2.9 BM) cobalt(II) complexes. Intermediate values are expected<sup>10</sup> and usually

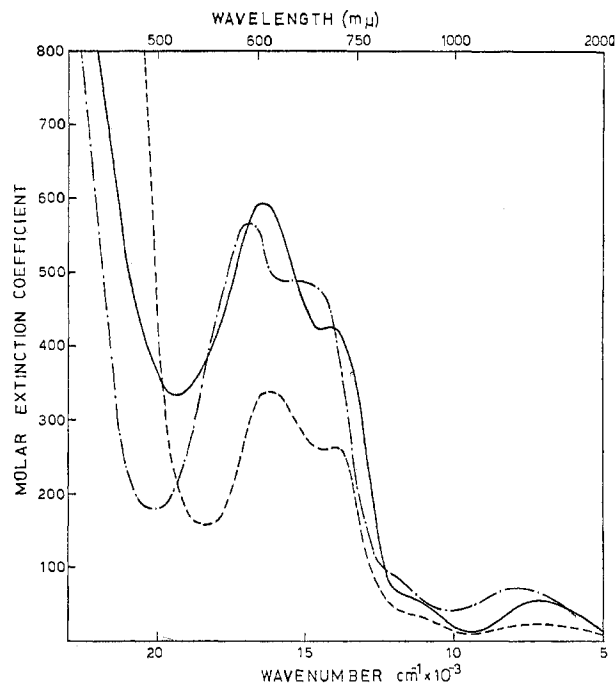


Figure 2.—Electronic absorption spectra: —,  $[\text{Co}(\text{PhP}(\text{OC}_2\text{H}_5)_2)_4\text{I}]\text{BPh}_4$ ,  $1.5 \times 10^{-3} M$  in 1,2-dichloroethane containing  $10^{-1} M$   $\text{PhP}(\text{OC}_2\text{H}_5)_2$ ; - - - - - ,  $[\text{Co}(\text{PhP}(\text{OC}_2\text{H}_5)_2)_4\text{I}]\text{BPh}_4$  solid on Nujol mull (arbitrary scale); - · - · - ,  $[\text{Co}(\text{PhP}(\text{OC}_2\text{H}_5)_2)_4\text{NCS}]\text{BPh}_4$ ,  $2 \times 10^{-3} M$  in 1,2-dichloroethane containing  $10^{-1} M$   $\text{PhP}(\text{OC}_2\text{H}_5)_2$ . The spectra of the other  $[\text{Co}(\text{PhP}(\text{OC}_2\text{H}_5)_2)_4\text{X}]\text{BPh}_4$  complexes have been omitted for clarity.

found<sup>3,4,11,12</sup> for five-coordinate cobalt(II) compounds. The agreement of the bulk susceptibility in solid and that in solution, measured by the <sup>1</sup>H nmr technique, is fairly good. This may indicate that the coordination is the same in both cases.

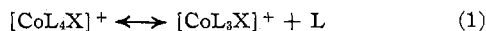
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These evidences seem to suggest that the complexes of the type  $[\text{Co}(\text{PhP}(\text{OC}_2\text{H}_5)_2)_4\text{X}]\text{BPh}_4$  ( $\text{X}^- = \text{Cl}^-$ ,  $\text{Br}^-$ ,  $\text{I}^-$ ,  $\text{N}_3^-$ ,  $\text{NCS}^-$ ) are five-coordinate and they appear to be the first compounds of cobalt(II) with the donor atom set  $\text{P}_4\text{X}$  in which the phosphine ligands are monodentate.

The solution data have been obtained by dissolving the complexes in the presence of an excess of free ligand to force the dissociative process to the left



( $\text{L} = \text{PhP}(\text{OC}_2\text{H}_5)_2$ ). Evidence for the existence of such equilibrium has been also obtained from the study of the electronic spectra in 1,2-dichloroethane or nitrobenzene in the visible region. When free ligand is not present the absorption spectrum, as for example the spectrum of  $[\text{Co}(\text{PhP}(\text{OC}_2\text{H}_5)_2)_4\text{I}]\text{BPh}_4$  (Figure 1), shows a multiple band that, on the basis of the similarity with other four-coordinated phosphine cobalt(II) complexes, has been tentatively attributed to the  $[\text{Co}(\text{PhP}(\text{OC}_2\text{H}_5)_2)_3\text{X}]^+$  species. Since Beer's law is obeyed in the range  $10^{-4}$  to  $5 \times 10^{-3} M$  both in polar and nonpolar organic solvents it is probable that in these conditions the equilibrium 1 lies completely to the right. Addition of free  $\text{PhP}(\text{OC}_2\text{H}_5)_2$  to the solution causes the spectrum in the range 5000–22,000  $\text{cm}^{-1}$  to change. When there is approximately a 50-fold molar excess of phosphine with respect to complex, further addition of ligand has no effect upon the spectrum which now closely resembles that of the complex in its solid state (Figure 2). The spectral data reported in Table II and Figure 2 relate to solutions containing nearly a 100-fold molar excess of the phosphine. The similarity of both the electronic spectra and the magnetic moments in solution and in solid suggests that, under such conditions, the coordination and probably also the geometrical structure around the cobalt atom could be the same.

A comparison of the electronic spectra of the complexes reported here with those of the already known compounds<sup>4</sup>  $[\text{Co}(\text{QP})\text{X}]\text{BPh}_4$  which have a trigonal-bipyramidal structure and those of  $[\text{Co}(\text{AP})_2\text{X}]\text{ClO}_4$  complexes<sup>11</sup> ( $\text{AP} =$  diphenyl(*o*-diphenylarsinophenyl)-phosphine) for which Dyer and Meek proposed a square-pyramidal structure shows rough similarities. Studies are in progress to correlate the position of the band maxima in the 5000–22,000  $\text{cm}^{-1}$  region to the d-d transitions and to find a relationship between electronic spectra and geometrical structure.

However, further useful information can be obtained from the infrared absorption spectra of the solids which are reported in Table III.

$[\text{Co}(\text{PhP}(\text{OC}_2\text{H}_5)_2)_4\text{NCS}]\text{BPh}_4$  and  $[\text{Co}(\text{PhP}(\text{OC}_2\text{H}_5)_2)_3(\text{NCS})_2]\text{ClO}_4$  have a strong peak at 2060 and 2066  $\text{cm}^{-1}$ , respectively, assigned to the CN stretching vibrations of NCS terminal groups bonded to the metal atom through the nitrogen.<sup>13</sup> The presence of only one band in the second complex indicates that the two thiocyanato groups are trans.

The absence of bands in the 2150–2180- $\text{cm}^{-1}$  region indicates<sup>13</sup> the absence of bridging NCS groups and therefore the complexes can be considered to be five-coordinate. The lower CN stretching frequencies of the

TABLE III

INFRARED DATA FOR SOME COBALT(II) COMPLEXES IN SOLID

Compound	$\nu(\text{CN})$ , $\text{cm}^{-1}$	$\nu(\text{N}_3)$ , $\text{cm}^{-1}$
$[\text{Co}(\text{PhP}(\text{OC}_2\text{H}_5)_2)_4\text{N}_3]\text{BPh}_4$		2062
$[\text{Co}(\text{PhP}(\text{OC}_2\text{H}_5)_2)_4\text{NCS}]\text{BPh}_4$	2060	
$[\text{Co}(\text{PhP}(\text{OC}_2\text{H}_5)_2)_3(\text{NCS})_2]^a$	2066	
$[\text{Co}(\text{Ph}_2\text{P}(\text{CH}_2)_2\text{PPh}_2)_2\text{NCS}]\text{ClO}_4^b$	2083	
$[\text{Co}(\text{PR}_3)_5(\text{NCS})_2]^c$	2080	

<sup>a</sup> In 1,2-dichloroethane solution with  $[\text{PhP}(\text{OC}_2\text{H}_5)_2] = 0.1 M$ ,  $\nu(\text{CN})$  is 2064  $\text{cm}^{-1}$ . <sup>b</sup> From ref 1. <sup>c</sup> From ref 2.  $\text{PR}_3$  is alkyl or aryl phosphine.

second and third compounds reported in Table III, compared with the  $\nu(\text{CN})$  frequencies of the last two thiocyanato complexes, are probably due to the fact that  $\pi$  interactions from the metal to the phosphine ligands in the case of alkyl or aryl tertiary phosphines or diphosphines are weaker than in the case of phenyldiethoxyphosphine.

On substitution of one neutral ligand in the complex  $[\text{Co}(\text{PhP}(\text{OC}_2\text{H}_5)_2)_4\text{NCS}]\text{BPh}_4$  by thiocyanate to give  $[\text{Co}(\text{PhP}(\text{OC}_2\text{H}_5)_2)_3(\text{NCS})_2]$ , the CN stretching frequency does not change very much (from 2060 to 2066  $\text{cm}^{-1}$ ). This may indicate that the electronic density on the central metal atom is roughly the same in both cases.

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## 1-(Chlorosilyl)pentaborane(9)<sup>1</sup>

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Previously, we reported the preparation and properties of 2- and  $\mu$ -(halosilyl)pentaboranes(9).<sup>2</sup> Recently, our studies have been extended to include 1-silylpentaboranes. In this connection, we have prepared and characterized 1-( $\text{ClSiH}_2$ ) $\text{B}_5\text{H}_8$ . These results are shown below. In addition, vapor tension *vs.* temperature data for 1- $\text{SiH}_3\text{B}_5\text{H}_8$  are reported which serve to complete the description of its physical properties.<sup>3</sup>

### Experimental Section

**Apparatus and Techniques.**—All work was carried out in a standard high-vacuum system.<sup>4,5</sup> Vapor pressure *vs.* temperature data were obtained using an all-glass immersible tensimeter which had been predried with  $\text{SiH}_2\text{Br}$  for a period of 12 hr. Samples were frozen to  $-196^\circ$  between pressure measurements to minimize decomposition. Melting points were obtained by the Stock method. Infrared spectra were obtained with Beckman IR-12 and Perkin-Elmer Model 337 spectrometers on gaseous samples in a 10-cm cell fitted with KBr windows. Proton nuclear magnetic resonance spectra were recorded at 60.0 MHz

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