93° and those that are ca. 101°. It is reasonable to suggest that the ~101° Ni–O–Ni angles lead to the antiferromagnetic interaction (J_1 = –9.1 cm^{-1}), whereas the ferromagnetic interaction is associated with the ~93° Ni–O–Ni angles (J_2 ($\approx J_3$) of ca. 18 cm^{-1}).

The use of a Weiss constant (Θ) is not apppropriate for fitting the data for $[Ni_4(OCH_3)_4(TMB)_4(OAc)_2](BPh_4)_2$, because the ground state of this complex has $S = 0$. This can be seen from a diagram in which the energies (divided by J_1) of the 19 states of this complex are plotted as a function of J_2/J_1 (Figure 8). In the present complex $J_2/J_1 = -1.92$, which is represented by the vertical dashed line. Level 19 is the ground state; examination of Table III shows that this is the state where all of the electrons are paired.

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Registry No. $[Ni_4(OCH_3)_4(TMB)_4(OAc)_2](BPh_4)_2\cdot 4CH_2Cl_2$, 77241-80-6; $[Ni_4(OCH_3)_4(DMB)_4(OAc)_2](BPh_4)_2$, 77241-82-8.

Supplementary Material Available: Tables of solvent parameters and hydrogen parameters from the structure determination, structure factors, and experimental and theoretical magnetic susceptibility data (42 pages). Ordering information is given on any current masthead page.

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Fluoro-Containing Complexes of Chromium(III). 10. Preparation, Crystal Structure, and Some Reactions of the *trans*-Fluoroamminebis(1,3-propanediamine)chromium(III) Cation[1]

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The reaction of *trans*-$[Cr(1,3\text{-}pn)_2FBr]ClO_4$ (1,3-pn = 1,3-propanediamine) with liquid ammonia at room temperature was used to prepare the *trans*-$[Cr(1,3\text{-}pn)_2FNH_3]^{2+}$ cation. The ammination reaction proceeded to yield apparently the trans isomer only. The reaction product with the empirical molecular formula $[Cr(NH_3)F(N_2C_3H_{10})_2](ClO_4)_2$ was subjected to a single-crystal X-ray structural analysis. The complex crystallizes in the monoclinic space group $P2_1/n$ with a = 10.207 (4) Å, b = 9.653 (4) Å, c = 17.599 (5) Å, β = 94.62 (3)°, Z = 4, and V = 1728 (1) Å^3. Diffraction data were collected with a computer-controlled four-circle Nicolet autodiffractometer out to a maximum 2θ Mo Kα of 51°. The structure was solved by standard heavy-atom Patterson and Fourier methods and refined by full-matrix least squares. The final discrepancy values based on the 2177 counter data having $I \geq 3\sigma(I)$ were R_1 = 0.065 and R = 0.072. The final structure revealed that the two chelate rings are nonequivalent in the solid with one of the rings adopting a chair conformation while the other is in a twist conformation. The Cr–F distance is 1.872 (3) Å, and the H_3NCrF angle is 179.0 (2)°. Reaction of the *trans*-FNH_3 complex with more liquid ammonia resulted in little additional ammination, but reaction of the complex with concentrated hydrobromic acid resulted in loss of the fluoro ligand and its replacement by water.

Introduction

In 1975 Wong and Kirk[2] reported the successful preparations of *cis*- and *trans*-$[Cr(en)_2FNH_3]^{2+}$, where en is ethylenediamine, via the reaction of *trans*-$[Cr(en)_2FBr]^+$ with a dilute solution of ammonium perchlorate in dry liquid ammonia at 0 °C. The preparative reaction was accompanied by extensive stereochemical change, and the cis/trans ratio in the crude product was estimated to be 60/40. Previous work in this laboratory[3,4] has indicated that the closely related 1,3-propanediamine complexes are more resistant to stereochemical change than the corresponding ethylenediamine complexes. Hence, the present investigation was undertaken to determine what products would be formed by the reaction of *trans*-$[Cr(1,3\text{-}pn)_2FBr]^+$ with liquid ammonia and to characterize these products.

Experimental Section

Caution! Perchlorate salts of metal complexes with reducing ligands such as amines are potentially explosive, and care should be exercised when handling these materials.

Preparation of Starting Material. *trans*-Bromofluorobis(1,3-propanediamine)chromium(III) perchlorate–0.5-water was prepared as described in the literature.[3] A solution of 1.17 g (2.9 mmol) of the crude product in 11 mL of water at 10 °C was prepared and filtered to remove a small amount of undissolved material. Solid sodium perchlorate was added to the stirred filtrate, and the precipitated product was collected, washed with acetone, and air-dried; yield 0.52 g (44%). Anal. Calcd for *trans*-$[Cr(1,3\text{-}pn)_2FBr]ClO_4\cdot 0.5H_2O$: C, 17.7; H, 5.15; N, 13.72. Found: C, 17.79; H, 5.06; N, 13.87.

Preparation of *trans*-Fluoroamminebis(1,3-propanediamine)chromium(III) Bromide Perchlorate. A 3.10-g (7.6-mmol) sample of recrystallized *trans*-$[Cr(1,3\text{-}pn)_2FBr](ClO_4)\cdot 0.5H_2O$ was placed in one leg of a modified Y-tube. The tube and its contents were cooled to about –77 °C in a dry ice–2-propanol bath. Gaseous ammonia was added to the cooled reactant until about 25 mL of liquid ammonia had condensed in the tube. The tube was sealed and allowed to stand at room temperature for 2 h. It was necessary to tip the tube several times before the starting material all dissolved in the liquid ammonia. As the starting material dissolved, the solution became deep maroon. After about 1 h at room temperature the solution was deep orange, and red-orange crystals were deposited from the solution. At the end of 2 h the empty leg of the Y-tube was placed in a dry ice–2-propanol bath, and the liquid ammonia was distilled into the cooled leg. The leg containing the liquid ammonia was cut off with a torch and set aside. The leg containing the crude product was opened, attached to a vacuum system, and pumped overnight (0.05 torr, room temperature); yield 3.10 g (98%). The electronic spectrum of the crude material in 0.2 M perchloric acid was characterized by λ_{max} 497 nm (ϵ 46.9 M^{-1} cm^{-1}), λ_{min} 418 (ϵ 13.8), and λ_{max} 366 (ϵ 31.0).

The reaction of *trans*-$[Cr(1,3\text{-}pn)_2FBr](ClO_4)$ with liquid ammonia in a sealed tube was repeated as described above except that the temperature of the sealed tube was maintained at 0 °C for the 2-h reaction time. The reaction mixture was worked up as described previously. The electronic spectrum of an aqueous solution of the crude product was characterized by λ_{max} 496 nm (ϵ 47.4), λ_{min} 418 (ϵ 13.6), and λ_{max} 366 (ϵ 30.0). Conversion of the crude product to

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(1) Part 9: Vaughn, J. W.; Seiler, G. J. *Inorg. Chem.* **1979**, *18*, 1509.
(2) Wong, C. F. C.; Kirk, A. D. *Can. J. Chem.* **1975**, *53*, 3388.
(3) Vaughn, J. W. *Inorg. Nucl. Chem. Lett.* **1968**, *4*, 183.
(4) DeJovine, J. M.; Mason, W. R.; Vaughn, J. W. *Inorg. Chem.* **1974**, *13*, 66.

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the corresponding diperchlorate salt gave an orange solid which had an electronic spectrum identical with that of the salt produced via the room-temperature reaction.

A 0.60-g (1.4-mmol) sample of the crude material was dissolved in ca. 1 mL of water, the clear solution was filtered, and the stirred filtrate was diluted with 4 mL of absolute alcohol. After the addition of about 2 mL of alcohol, an orange solid began to precipitate. The reaction mixture was cooled in an ice bath for 30 min before the product was collected, washed with absolute alcohol followed by ether, and air-dried; yield 0.25 g (42%). The product was dried over anhydrous magnesium perchlorate for 18 h at room temperature and 0.05 torr. Anal. Calcd for *trans*-$[Cr(1,3\text{-pn})_2FNH_3]BrClO_4$: C, 17.33; H, 5.45; N, 16.85; Cr, 12.51; F, 4.57. Found: C, 17.08; H, 5.58; N, 16.62; Cr, 12.46; F, 4.65. The electronic spectrum of the complex in 0.2 M perchloric acid was characterized by λ_{max} 498 nm (ϵ 46.1), λ_{min} 418 (ϵ 13.8), and λ_{max} 368 (ϵ 31.2).

Isolation of *trans*-Fluoroamminebis(1,3-propanediamine)chromium(III) Perchlorate. A 1.07-g (2.57-mmol) sample of the crude bromide perchlorate was dissolved in 2.5 mL of water, and the solution was filtered. The stirred filtrate was slowly diluted by the dropwise addition of 1.0 mL of concentrated perchloric acid. Orange crystals of product began to form during the addition of the acid. The reaction mixture was cooled in an ice bath for 10 min before the product was collected, washed with absolute alcohol followed by ether, and air-dried; yield 0.52 g (47%). Dilution of the filtrate with another 5 mL of perchloric acid followed by cooling in an ice bath for 30 min did not result in any additional product. Anal. Calcd for *trans*-$[Cr(1,3\text{-pn})_2FNH_3](ClO_4)_2$: C, 16.55; H, 5.28; N, 16.09; Cr, 11.95; F, 4.36. Found: C, 16.52; H, 5.24; N, 16.11; Cr, 12.00; F, 4.51.

The electronic spectrum of the complex in 0.2 M perchloric acid was characterized by λ_{max} 500 nm (ϵ 45.9), λ_{min} 418 (ϵ 13.8), and λ_{max} 368 (ϵ 31.9). Recrystallization of the compound from water did not change the elemental analyses or the spectral data.

The infrared spectrum of the compound in the 400–600-cm^{-1} region exhibited strong bands at 435, 492, and 538 cm^{-1}. A weak band was found at 410 cm^{-1}, and the Cr–F band was at 358 cm^{-1}.

The isolation of the *trans*-$[Cr(1,3\text{-pn})_2FNH_3](ClO_4)_2$ was repeated, and the filtrate was slowly diluted with 4 volumes of absolute alcohol followed by 2 volumes of ether. This resulted in the formation of an orange oil. The oil was frequently stirred over a period of 2 h. At the end of this time the oil had crystallized to an orange solid. The product was collected, washed with a little absolute alcohol followed by ether, and air-dried. Anal. Calcd for *trans*-$[Cr(1,3\text{-pn})_2FNH_3]BrClO_4$: C, 17.33; H, 5.45; N, 16.85; Cr, 12.51; F, 4.57. Found: C, 17.46; H, 5.58; N, 16.90; Cr, 12.43; F, 4.35. The electronic spectrum of the complex in 0.2 M perchloric acid was characterized by λ_{max} 497 nm (ϵ 47.3), λ_{min} 418 (ϵ 13.6), and λ_{max} 367 (ϵ 30.0).

Ion-Exchange Studies. A weighed sample of crude $[Cr(1,3\text{-pn})_2FNH_3]BrClO_4$ was dissolved in water and charged onto an ion-exchange column (Baker CGC-240, 100–200 mesh, NH_4^+, column 7.0 × 1 cm). The column was eluted 2–2.5 mL/min, successively with 200-mL volumes of 0.1, 0.6, 0.8, and 1.0 M aqueous ammonium sulfate followed by 200 mL of 6 M sulfuric acid. The fractions were collected as described previously.[4] After oxidation of the various fractions with hot alkaline peroxide and measurement of the absorbance of the solutions at 373 nm, the following results were obtained: charging fraction, 0% Cr; 0.1 M fraction, <1.0% Cr; 1.0 M fraction, 2.2% Cr; 6 M H_2SO_4 fraction, <1.40% Cr. Greater than 95% of the Cr was eluted in the 0.6 and 0.8 M fractions. *trans*-$[Cr(1,3\text{-pn})_2F_2]^+$ could be eluted from this column with 200 mL of 0.1 M aqueous ammonium sulfate while *trans*-$[Cr(1,3\text{-pn})_2F(OH_2)]^{2+}$ could not be eluted from the column until the concentration of ammonium sulfate reached 0.6 M. Concentrations of ammonium sulfate greater than 1.0 M were required to elute $[Cr(en)_3]^{3+}$ from the column.

Reaction of *trans*-$[Cr(1,3\text{-pn})_2FNH_3](ClO_4)_2$ with Liquid Ammonia. A 0.205-g (0.47-mmol) sample of analyzed *trans*-$[Cr(1,3\text{-pn})_2FNH_3](ClO_4)_2$ was placed in a Y-tube, and $NH_3(l)$ was condensed onto the sample as described previously. The tube was sealed and allowed to stand at room temperature for 18 h before the tube was opened as previously described. The electronic spectrum of the crude material in 0.2 M perchloric acid was characterized by λ_{max} 496 nm, λ_{min} 422, and λ_{max} 364.

A weighed sample of the compound was subjected to ion-exchange chromatography as described above. Results were as follows for the various fractions: 0% Cr; 0.1 M $(NH_4)_2SO_4$, 0% Cr; 0.6 M $(NH_4)_2SO_4$, 38.1% Cr; 0.8 M $(NH_4)_2SO_4$, 53.1%; 1.0 M $(NH_4)_2SO_4$, 7.30% Cr; 2.0 M H_2SO_4, 1.45% Cr; 6.0 M H_2SO_4, 0.11% Cr.

Table I. Crystallographic Summary

formula	$Cr(NH_3)F(N_2C_3H_{10})_2$-$(ClO_4)_2$	mol wt	435.25
crystal system	monoclinic	space groups and No.[a]	$P2_1/n$—an alternate setting of $P2_1/c$–C^5_{2h} (No. 14)
a, Å	10.207 (4)		
b, Å	9.653 (4)	V, Å^3	1728 (1)
c, Å	17.599 (5)	Z	4
β, deg	94.62 (3)	ρ_{calcd}, g cm^{-3}	1.67
μ(Mo K$\bar{\alpha}$), cm^{-1}	10.6[a]		

[a] "International Tables for X-ray Crystallography"; Kynoch Press: Birmingham, England, 1969; Vol. 1.

A weighed sample of the compound was dissolved in a minimum amount of water and converted to the insoluble diperchlorate salt by the addition of cold concentrated perchloric acid; yield 73%. The results of the elemental analysis confirmed the presence of the *trans*-$[Cr(1,3\text{-pn})_2FNH_3]^{2+}$ ion.

Reaction of *trans*-$[Cr(1,3\text{-pn})_2FNH_3](ClO_4)_2$ with Concentrated Hydrobromic Acid. A 0.56-g (1.28-mmol) sample of *trans*-$[Cr(1,3\text{-pn})_2FNH_3](ClO_4)_2$ was dissolved in 2 mL of concentrated hydrobromic acid, and the solution was filtered. The reaction mixture was allowed to stand overnight at room temperature before the product was collected, washed with acetone, and air-dried; yield 0.13 g (21%). Anal. Calcd for $[Cr(1,3\text{-pn})_2NH_3(OH_2)]Br_2ClO_4$: C, 14.56; H, 4.85; N, 14.15; Br, 32.35; Cr, 10.51. Found: C, 14.42; H, 4.81; N, 14.06; Br, 33.20; Cr, 10.60. The electronic spectrum of the complex in 0.2 M perchloric acid was characterized by λ_{max} 474 nm (ϵ 36.5), λ_{min} 408 (ϵ 14.2), and λ_{max} 359 (ϵ 39.3). The same product can also be obtained by using crude $[Cr(1,3\text{-pn})_2FNH_3]BrClO_4$ as the starting material.

Deammination of *trans*-$[Cr(1,3\text{-pn})_2FNH_3]BrClO_4$. A weighed sample of analyzed *trans*-$[Cr(1,3\text{-pn})_2FNH_3]BrClO_4$ was heated at 120 °C for 3 days. During this time the sample underwent a color change from a pale orange to a light purple and lost 3.93% of its weight. The calculated weight loss for complete removal of one NH_3 molecule per formula weight is 4.09%. Anal. Calcd for $[Cr(1,3\text{-pn})_2FBr]ClO_4$: C, 18.1; H, 5.0; N, 14.1; Cr, 13.1. Found: C, 17.7; H, 5.3; N, 13.9; Cr, 12.8. The aqueous solution electronic spectrum was characterized by λ_{max} 519 (ϵ 35.9), λ_{min} 441 (ϵ 14.8), and λ_{max} 386 (ϵ 28.3).

Analytical Methods and Instrumentation. These were the same as described previously.[4] The crystal structure was determined at Crystalytics Co., Lincoln, NE.

Structure Determination. An orange-red, multifaceted tabloid crystal of *trans*-$[Cr(1,3\text{-pn})_2FNH_3](ClO_4)_2$ suitable for the crystallographic study was obtained from a concentrated aqueous solution of the complex after slow evaporation at 4 °C. The crystal, which had minimum and maximum dimensions of 0.42 and 0.65 mm, was randomly oriented and glued to the inside of a thin glass capillary. To obtain the cell constants, we collected 15 computer-centered reflections ($2\theta > 30°$) using a computer-controlled four-circle Nicolet autodiffractometer. The data were least-squares refined to yield the values in Table I.

The data were corrected for Lorentz and polarization effects. No corrections were made for absorption or for changes in the check-reflection intensities. The structure was solved by standard heavy-atom Patterson techniques and refined by a full-matrix least-squares method. Minimization of the function $\sum w(|F_o| - |F_c|)^2$ was defined as $w = 1/\sigma_F^2$, where σ is the estimated standard deviation of the observation in question. The positions of the ammonia hydrogen atoms could be located from a difference Fourier map after the third refinement cycle. The hydrogen positions for the (1,3-pn) ligands were calculated by using idealized geometries. All hydrogen atoms were assigned isotropic thermal parameters of 7.0 Å^2. No hydrogen positional parameters or temperature factors were varied in the remaining cycles.

Structure Refinement. Anomalous dispersion corrections[5] were applied for Cr, Cl_1, and Cl_2.

Full-matrix least-squares convergence was obtained with use of only those 2177 data with $I_o/\sigma(I_o) > 3.00$. Final values were $R_1 = \sum||F_o| - |F_c|/\sum|F_o|| = 0.065$ and $R_2 = [\sum w(|F_o| - |F_c|)^2/\sum|F_o|^2]^{1/2}$

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(5) "International Tables for X-ray Crystallography"; Kynoch Press: Birmingham, England, 1974; Vol. IV, pp 149–150.

Table II. Data Collection at 20 ± 1 °C

instrument	computer-controlled four-circle Nicolet autodiffractometer
λ(Mo K$\bar{\alpha}$), Å	0.710 73
monochromator	graphite
takeoff angle, deg	4
scan	ω
scan range, deg	1.0
scan rate, deg min^{-1}	6.0 20 < 2θ < 43.0° 4.0 43 < 2θ < 51.0°
total reflctns	3219
total independent reflctns	3219
check reflctns and freq	6 remeasd after every 300
used reflctns	2177
significant criterion for $I > n(\sigma(I))$	n = 3.0

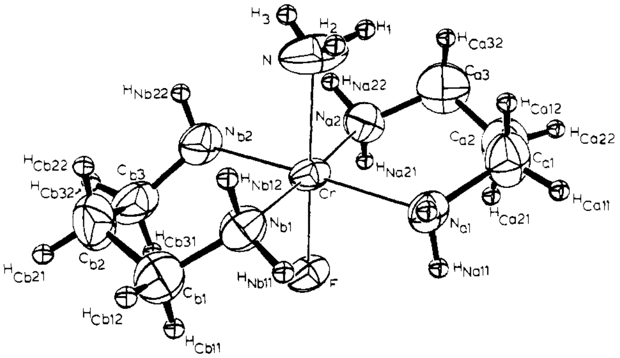

Figure 1. A perspective view of the cation.

= 0.072, and the goodness of fit for the last cycle was $[\sum w(|F_o| - |F_c|)^2/(NO - NV)]^{1/2} = 3.50$ for NO = 2177 observations and NV = 208 variables.

In the final cycle of refinement the average shift/error for all nonhydrogen atom parameters was $0.001\sigma_p$.

There were no peaks present in the final difference Fourier map above the noise level.

Figure 1 shows a perspective ORTEP drawing of the $Cr(NH_3)F(N_2C_3H_{10})_2{}^{2+}$ cation.

Discussion

The previously unknown compound *trans*-$[Cr(1,3\text{-}pn)_2FNH_3]BrClO_4$ was prepared by reacting *trans*-$[Cr(1,3\text{-}pn)_2FBr]ClO_4$ with liquid ammonia in a sealed tube for 2 h at room temperature. The product of the reaction is not temperature dependent in the range 0–20 °C, since the identical product was obtained at both 0 and 20 °C. That the *trans*-FNH_3 isomer could be prepared via this reaction is not surprising, since the ammination of *trans*-$[Cr(en)_2FBr]^+$ under similar conditions yields *trans*-$[Cr(en)_2FNH_3]^{2+}$ as one of the reaction products.[2] However, in the case of the ethylenediamine complex the preparative reaction was accompanied by extensive stereochemical change with a final cis/trans ratio of 60/40 whereas the 1,3-pn system apparently reacts with little or no stereochemical change.

The lack of stereochemical change with the 1,3-pn complex with respect to that of the corresponding en complex is similar to the lack of stereochemical change observed when *trans*-$[Cr(AA)_2F(OH_2)]X_2$ (X = Cl^-, Br^-) is thermally dehydrated.[3] The trans AA = 1,3-pn complexes yield *trans*-FX isomers while the corresponding trans AA = en complexes yield mainly *cis*-FX isomers.[6]

Known cases of cis isomers of the type $[Cr(1,3\text{-}pn)_2X_2]^{n+}$ are rare with apparently the only well-characterized cases being X = H_2O[7] and X = Cl.[8] The ion *cis*-$[Cr(1,3\text{-}pn)_2F(OH_2)]^{2+}$ has been characterized in solution[4] but has yet to be isolated as a solid salt.

Table III. Electronic Spectral Data for Some Chromium(III)–Fluoro Complexes

complex	λ,[a] ε[b]	λ, ε	λ, ε
$[Cr(NH_3)_5F]^{2+}$ [c]	503, 41.7	366, 20.4	
trans-$[Cr(1,3\text{-}pn)_2FNH_3]^{2+}$ [d]	500, 45.9	368, 31.9	
trans-$[Cr(en)_2FNH_3]^{2+}$ [e]	490, 45.7	356, 24.1	
cis-$[Cr(en)_2FNH_3]^{2+}$ [c]	494, 64.9	360, 34.4	
trans-$[Cr(1,3\text{-}pn)_2F(OH_2)]^{2+}$ [f]	533, 20.9	460, 22.4	375, 37.8
cis-$[Cr(1,3\text{-}pn)_2F(OH_2)]^{2+}$ [f]	511, 27.0	376, 30.8	

[a] Wavelength in nm. [b] Molar absorptivities in M^{-1} cm^{-1}. [c] Zinato, E.; Lindholm, R.; Adamson, W. A. *Inorg. Nucl. Chem.* **1969**, *31*, 449. [d] This work. [e] Wong, C. F. C.; Kirk, A. D. *Can. J. Chem.* **1975**, *53*, 3388. [f] DeJovine, J. M.; Mason, W. R.; Vaughn, J. W. *Inorg. Chem.* **1974**, *13*, 66.

The results of the ion-exchanging chromatography experiments indicate that greater than 95% of the Cr(III)-containing species found in crude $[Cr(1,3\text{-}pn)_2FNH_3]BrClO_4$ have a charge of 2+. As a result, the ammination of the bromo ligand is complete, and substitution of F^- by NH_3 to yield ions of the type $[Cr(1,3\text{-}pn)_2(NH_3)_2]^{3+}$ does not occur. The observation that the fraction that contained the 2+ species could be eluted from the column as a single band would support the supposition that the crude material consisted of only one isomer.

The possibility of the replacement of the 1,3-pn ligands by NH_3 to yield the dipositive ion $[Cr(1,3\text{-}pn)(NH_3)_3F]^{2+}$ cannot be excluded by the ion-exchange data. However, such a result can be excluded by the analytical data.

It is expected that the electronic spectra of *cis*- and *trans*-$[Cr(1,3\text{-}pn)_2FNH_3]^{2+}$ should be rather similar if one considers only the CrN_5F core since both isomers have C_{4v} symmetry. Hence, large shifts in the wavelengths of the d → d transitions are not anticipated, nor would one expect any difference in the number of spin-allowed electronic transitions.

As pointed out by Wong and Kirk[2] for the ethylenediamine case, the electronic spectrum of the 1,3-pn complex should closely resemble that of $[Cr(NH_3)_5F]^{2+}$, which has C_{4v} symmetry.

The data in Table III clearly show that the wavelengths of the electronic transitions for *trans*-$[Cr(1,3\text{-}pn)_2FNH_3]^{2+}$ are very close to those found for $[Cr(NH_3)_5F]^{2+}$. In fact, the average ligand fields at the Cr(III) centers are almost identical in the two complexes. Wong and Kirk[2] found for the cis–trans pair of $[Cr(en)_2FNH_3]^{2+}$ that the molar absorptivity of the first spin-allowed transition for the cis isomer is about 50% higher than that associated with the corresponding transition in the trans complex. Such comparisons are difficult in the 1,3-pn system because of the lack of cis isomers, but the pattern does hold up for *cis*- and *trans*-$[Cr(1,3\text{-}pn)_2F(OH_2)]^{2+}$, where molar absorptivities differ by 30%, as well as *cis*- and *trans*-$[Cr(1,3\text{-}pn)_2(OH_2)_2]^{3+}$. However, no significance should be attached to the fact that the difference in molar absorptivities of the 1,3-pn cis–trans pair is only 30% instead of the 50% found in the en case, since the cis–trans members of the reference pairs $[Cr(1,3\text{-}pn)_2F(OH_2)]^{2+}$ and $[Cr(1,3\text{-}pn)_2(OH_2)_2]^{3+}$, unlike *cis*- and *trans*-$[Cr(1,3\text{-}pn)_2FNH_3]^{2+}$, belong to different symmetry groups if only local site symmetry is considered.

After removal of the least soluble diperchlorate salt from the crude reaction mixture, an orange bromide perchlorate could be obtained by diluting the reaction mixture with absolute alcohol and ether.

The wavelengths of the electronic transitions in this product were within experimental error of those found for the crude

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(6) Vaughn, J. W.; Magnuson, V. E.; Stran, O. J., Jr. *Inorg. Chem.* **1968**, *7*, 736.

(7) Cauldwell, M. C.; House, D. A. *Inorg. Chem.* **1972**, *11*, 2025.

(8) Pederson, E. *Acta Chem. Scand.* **1970**, *24*, 3362.

Table IV. Atomic Coordinates in Crystalline Cr(NH
$Cr(NH_3)F(N_2C_3H_{10})_2(ClO_4)_2$[a]

name[b]	fractional coordinates *x*	*y*	*z*
Cr	0.19647 (11)	0.28011 (10)	0.15813 (6)
F	0.22031 (34)	0.11456 (34)	0.21292 (19)
N	0.17104 (76)	0.46606 (62)	0.09906 (35)
N_{a1}	0.36641 (53)	0.35718 (52)	0.21721 (29)
N_{a2}	0.30093 (60)	0.19924 (58)	0.07124 (31)
N_{b1}	0.08664 (54)	0.36732 (56)	0.24047 (30)
N_{b2}	0.02602 (58)	0.19940 (60)	0.10198 (30)
C_{a1}	0.48470 (75)	0.39317 (83)	0.17459 (42)
C_{a2}	0.51927 (85)	0.28279 (105)	0.12038 (48)
C_{a3}	0.42500 (93)	0.26383 (98)	0.05131 (43)
C_{b1}	−0.02756 (80)	0.29422 (91)	0.26699 (42)
C_{b2}	−0.12371 (76)	0.24858 (97)	0.20189 (47)
C_{b3}	−0.07308 (80)	0.14310 (82)	0.14950 (45)
Cl_1	0.26139 (21)	0.32747 (20)	0.44109 (11)
O_{11}	0.28087 (68)	0.26717 (63)	0.36796 (28)
O_{12}	0.36198 (59)	0.42635 (59)	0.46032 (35)
O_{13}	0.13796 (56)	0.39387 (64)	0.43539 (39)
O_{14}	0.26639 (69)	0.22199 (59)	0.49710 (28)
Cl_2	0.14826 (21)	0.33951 (23)	−0.12294 (11)
O_{21}	0.17083 (102)	0.20240 (83)	−0.10992 (59)
O_{22}	0.26897 (67)	0.40442 (86)	−0.12815 (45)
O_{23}	0.09044 (72)	0.39487 (108)	−0.06271 (37)
O_{24}	0.06132 (65)	0.35759 (62)	−0.18815 (30)
H_1	0.25344 (0)	0.45166 (0)	0.06400 (0)
H_2	0.19127 (0)	0.53540 (0)	0.11718 (0)
H_3	0.12752 (0)	0.47923 (0)	0.06391 (0)
H_{Na11}	0.39633 (0)	0.28858 (0)	0.25260 (0)
H_{Na12}	0.34297 (0)	0.43841 (0)	0.24186 (0)
H_{Na21}	0.31871 (0)	0.10800 (0)	0.08392 (0)
H_{Na22}	0.24037 (0)	0.20460 (0)	0.02627 (0)
H_{Nb11}	0.14463 (0)	0.38229 (0)	0.28434 (0)
H_{Nb12}	0.05375 (0)	0.45281 (0)	0.22079 (0)
H_{Nb21}	0.05083 (0)	0.12642 (0)	0.07006 (0)
H_{Nb22}	−0.01502 (0)	0.27114 (0)	0.07192 (0)
H_{Ca11}	0.55545 (0)	0.41093 (0)	0.20985 (0)
H_{Ca12}	0.46250 (0)	0.47520 (0)	0.14516 (0)
H_{Ca21}	0.51771 (0)	0.19567 (0)	0.14852 (0)
H_{Ca22}	0.60342 (0)	0.29886 (0)	0.10654 (0)
H_{Ca31}	0.46517 (0)	0.20908 (0)	0.01457 (0)
H_{Ca32}	0.40845 (0)	0.35523 (0)	0.02845 (0)
H_{Cb11}	0.00474 (0)	0.21195 (0)	0.29302 (0)
H_{Cb12}	−0.06970 (0)	0.35105 (0)	0.29958 (0)
H_{Cb21}	−0.19881 (0)	0.21121 (0)	0.22335 (0)
H_{Cb22}	−0.14910 (0)	0.32850 (0)	0.17268 (0)
H_{Cb31}	−0.03508 (0)	0.06950 (0)	0.17972 (0)
H_{Cb32}	−0.14521 (0)	0.10877 (0)	0.11752 (0)

[a] The numbers in parentheses are the estimated standard deviations in the last significant figure. For all H atoms B_{iso} = 7.00 (0) Å^2. [b] Atoms labeled in agreement with Figure 1.

$[Cr(1,3\text{-}pn)_2FNH_3]BrClO_4$, and the corresponding molar absorptivities in the two cases differed by no more than 4%.

The results of the ion-exchange studies strongly suggest that the chromium(III) fraction of charge 2+ consists of only one isomer. This conclusion is supported by the observation that the product obtained after the removal of the diperchlorate exhibited electronic spectral characteristics very close to those of the bromide perchlorate.

The infrared spectrum of the least soluble diperchlorate in the 400–600-cm^{-1} region is consistent with the published spectra[9] for *trans*-$[Cr(1,3\text{-}pn)_2X_2]^{n+}$ complexes except for the weak band at 410 cm^{-1}. Similar behavior[2] has been observed in the ethylenediamine system and found not to exclude the presence of the trans isomer.

The results of the X-ray structural determination confirm the trans structure for the least soluble diperchlorate salt obtained from the reaction of *trans*-$[Cr(1,3\text{-}pn)_2FBr]ClO_4$ with liquid ammonia. The positional and thermal parameters for *trans*-$[Cr(1,3\text{-}pn)_2FNH_3](ClO_4)_2$ are given in Tables IV and V. Selected bond distances and bond angles are contained in Tables VI and VII.

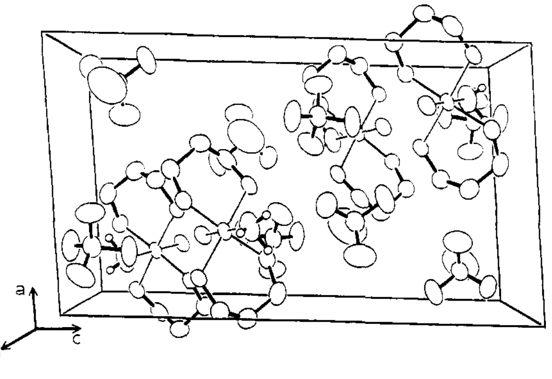

Figure 2. Packing of the molecules in the unit cell as viewed along the *b* axis.

When viewed in accord with Figure 1, ring b involves a chair conformation while ring a is in the slightly higher energy twist conformation.

The Cr–F distance is 1.872 Å, which is in agreement with that found for CrF_6^{3-} (1.90).[10] The Cr–ammine distance does not differ significantly from the Cr–N distance in the molecule, and the ammine–chromium–nitrogen (ring a) angles are very near the anticipated values of 90°. However, the ammine–chromium–nitrogen (ring b) angles show deviation from 90° with the $NCrN_{b1}$ angle being compressed by about 3°. This compression of the bond angle is also manifested in the F–N nonbonded distances and in the $FCrN_{b1}$ angle. Calculation of close contacts involving ring hydrogen atoms in their idealized positions with the fluoro ligand of the nearest cation in the unit cell produced an H···F distance of 2.01 Å and a $N_{a1}H_{Na12}F$ angle of 175°. All other close contact distances involving either the ring hydrogens or the ammine hydrogens and the oxygen atoms of the perchlorate groups as acceptor atoms are greater than those found for the fluorine–hydrogen interaction. However, the calculations indicate that considerable hydrogen bonding could occur in the solid.

As can be seen from Figure 2 the asymmetric units are arranged in a zigzag pattern in the unit cell. The NH_3–Cr–F axis of unit 1 is essentially parallel to the same axis in unit 4, but the NH_3–Cr–F axis of unit 4 has been rotated 180° with respect to the same axis in unit 1. In a similar fashion the NH_3–Cr–F axes of units 2 and 3 are parallel but are rotated by 180° with respect to one another.

Although the perchlorate anions are arranged on the same side of the chelate rings, they are not positioned over the rings; rather they are dihedrally arranged, with respect to the rings, closer to the NH_3 ligand than to the F^- group.

The nonequivalent conformations of the two chelate rings are unusual in that those bis(1,3-propanediamine) complexes of Co(III) whose structures are known from single-crystal X-ray studies involve chair conformations for both rings. That is, *trans*-$[Co(1,3\text{-}pn)_2Cl_2]Cl{\cdot}HCl{\cdot}2H_2O$,[11] *trans*-$[Co(1,3\text{-}pn)_2(NO_3)_2]NO_3$,[12] and $(-)_{589}$-*cis*-$[Co(1,3\text{-}pn)_2(NCS)_2]$-[Sb-(+)-tart]$\cdot 2H_2O$[13] all involve a chair conformation. Since *trans*-$[Co(1,3\text{-}pn)_2Cl_2]ClO_4$ and *trans*-$[Cr(1,3\text{-}pn)_2Cl_2]ClO_4$ are isomorphous[14] and the Co(III) complex involves a chair

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Table V. Anisotropic Thermal Parameters in Crystalline $Cr(NH_3)F(N_2C_3H_{10})_2(ClO_4)_2$[a]

name[b]	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Cr	3.81 (5)	2.23 (4)	2.33 (4)	−0.34 (4)	−0.63 (3)	0.11 (4)
F	4.38 (19)	2.16 (15)	3.60 (17)	−0.10 (14)	−0.93 (14)	0.26 (13)
N	9.40 (48)	3.42 (30)	4.24 (32)	0.12 (32)	−1.18 (31)	0.98 (25)
N_{a1}	3.59 (26)	2.97 (26)	3.06 (24)	−0.44 (21)	−0.55 (21)	−0.35 (19)
N_{a2}	4.73 (31)	3.84 (30)	3.48 (26)	−1.16 (25)	−0.04 (23)	−0.47 (22)
N_{b1}	3.86 (28)	3.71 (28)	3.34 (26)	0.19 (23)	−0.15 (22)	−0.32 (21)
N_{b2}	4.34 (29)	4.36 (32)	3.19 (25)	−0.82 (25)	−0.94 (22)	−0.61 (23)
C_{a1}	4.45 (39)	5.37 (42)	4.33 (38)	−1.00 (35)	0.20 (31)	−1.46 (33)
C_{a2}	5.05 (45)	9.07 (61)	4.91 (43)	−0.84 (46)	0.85 (36)	−0.54 (45)
C_{a3}	6.77 (52)	7.84 (57)	3.58 (38)	−0.29 (46)	1.12 (35)	−1.24 (37)
C_{b1}	4.57 (40)	7.12 (51)	3.91 (37)	−0.83 (39)	−0.07 (30)	−0.35 (36)
C_{b2}	3.53 (37)	8.22 (60)	5.25 (44)	−1.74 (38)	0.05 (32)	−0.20 (41)
C_{b3}	4.83 (41)	4.78 (43)	4.71 (40)	−1.16 (34)	−1.74 (33)	0.72 (32)
Cl_1	5.73 (11)	4.05 (9)	3.81 (9)	0.39 (8)	−0.17 (8)	0.15 (7)
O_{11}	10.94 (46)	7.07 (35)	2.93 (23)	1.95 (34)	0.13 (27)	−0.15 (24)
O_{12}	6.65 (34)	5.23 (31)	8.38 (38)	−1.48 (28)	−2.74 (29)	1.47 (28)
O_{13}	4.53 (30)	6.93 (36)	10.02 (44)	−0.02 (28)	0.94 (29)	−0.66 (32)
O_{14}	12.02 (48)	4.66 (27)	3.54 (25)	−1.86 (32)	1.38 (28)	0.84 (22)
Cl_2	4.83 (10)	5.18 (11)	4.17 (10)	0.37 (9)	−0.44 (8)	0.18 (8)
O_{21}	15.94 (81)	6.52 (45)	18.42 (84)	0.69 (48)	−4.09 (66)	4.95 (51)
O_{22}	6.32 (41)	11.06 (54)	12.07 (55)	−2.14 (39)	−0.06 (37)	0.05 (45)
O_{23}	7.54 (43)	23.02 (90)	4.97 (34)	1.31 (53)	0.98 (31)	−4.54 (47)
O_{24}	9.34 (43)	7.27 (37)	4.24 (28)	0.46 (33)	−2.13 (28)	−0.09 (26)

[a] The numbers in parentheses following each B_{ij} value are the estimated standard deviations in the last significant figure. The form of the anisotropic thermal parameters is $\exp[-0.25(B_{11}h^2a^{*2} + B_{22}k^2b^{*2} + B_{33}l^2c^{*2} + 2B_{12}hka^*b^* + 2B_{13}hla^*c^* + 2B_{23}klb^*c^*)]$. [b] Atoms are labeled in agreement with Figure 1.

Table VI. Bond Lengths, Polyhedral Edge Lengths, and Bond Angles Subtended at the Cr Atom in the Coordination Group of Crystalline $Cr(NH_3)F(N_2C_3H_{10})_2(ClO_4)_2$[a]

type[b]	length, Å	type[b]	length, Å
Cr–F	1.872 (3)	$F\cdots N_{a1}$	2.774 (6)
Cr–N	2.080 (6)	$F\cdots N_{a2}$	2.809 (6)
$Cr–N_{a1}$	2.086 (5)	$F\cdots N_{b1}$	2.856 (6)
$Cr–N_{a2}$	2.085 (6)	$F\cdots N_{b2}$	2.791 (7)
$Cr–N_{b1}$	2.080 (5)	$N_{a1}\cdots N_{a2}$	3.016 (7)
$Cr–N_{b2}$	2.082 (6)	$N_{b1}\cdots N_{b2}$	2.951 (8)
		$N_{a1}\cdots N_{b1}$	2.919 (8)
		$N_{a2}\cdots N_{b2}$	2.900 (9)

type[b]	angle, deg	type[b]	angle, deg
FCrN	179.0 (2)	$NCrN_{a1}$	90.4 (2)
$N_{a1}CrN_{b2}$	178.3 (2)	$NCrN_{a2}$	90.4 (2)
$N_{a2}CrN_{b1}$	176.9 (2)	$NCrN_{b1}$	86.9 (2)
$FCrN_{a1}$	88.8 (2)	$NCrN_{b2}$	91.1 (2)
$FCrN_{a2}$	90.3 (2)	$N_{a1}CrN_{a2}$	92.6 (2)
$FCrN_{b1}$	92.4 (2)	$N_{b1}CrN_{b2}$	90.3 (2)
$FCrN_{b2}$	89.7 (2)	$N_{a1}CrN_{b1}$	89.0 (2)
		$N_{a2}CrN_{b2}$	88.2 (2)

[a] The numbers in parentheses are the estimated standard deviations in the last significant figure. [b] Atoms are labeled in agreement with Figure 1.

Table VII. Bond Lengths and Bond Angles Involving Nonhydrogen Atoms in Crystalline $Cr(NH_3)F(N_2C_3H_{10})_2(ClO_4)_2$[a]

type[b]	length, Å	type[b]	length, Å
$N_{a1}–C_{a1}$	1.512 (9)	$Cl_1–O_{11}$	1.441 (6)
$N_{a2}–C_{a3}$	1.479 (11)	$Cl_1–O_{12}$	1.423 (6)
$N_{b1}–C_{b1}$	1.470 (10)	$Cl_1–O_{13}$	1.410 (6)
$N_{b2}–C_{b3}$	1.468 (10)	$Cl_1–O_{14}$	1.415 (6)
$C_{a1}–C_{a2}$	1.491 (12)	$Cl_2–O_{21}$	1.360 (8)
$C_{a2}–C_{a3}$	1.500 (12)	$Cl_2–O_{22}$	1.392 (8)
$C_{b1}–C_{b2}$	1.513 (11)	$Cl_2–O_{23}$	1.363 (8)
$C_{b2}–C_{b3}$	1.494 (12)	$Cl_2–O_{24}$	1.404 (6)

type[b]	angle, deg	type[b]	angle, deg
$CrN_{a1}C_{a1}$	120.0 (4)	$O_{11}Cl_1O_{12}$	109.6 (4)
$CrN_{a2}C_{a3}$	121.0 (5)	$O_{11}Cl_1O_{13}$	107.9 (4)
$CrN_{b1}C_{b1}$	120.8 (5)	$O_{11}Cl_1O_{14}$	109.4 (4)
$CrN_{b2}C_{b3}$	117.1 (5)	$O_{12}Cl_1O_{13}$	109.6 (4)
$N_{a1}C_{a1}C_{a2}$	113.1 (6)	$O_{12}Cl_1O_{14}$	109.4 (4)
$N_{a2}C_{a3}C_{a2}$	111.4 (7)	$O_{13}Cl_1O_{14}$	111.0 (4)
$N_{b1}C_{b1}C_{b2}$	112.4 (7)	$O_{21}Cl_2O_{22}$	108.1 (5)
$N_{b2}C_{b3}C_{b2}$	112.8 (7)	$O_{21}Cl_2O_{23}$	109.2 (6)
$C_{a1}C_{a2}C_{a3}$	115.9 (7)	$O_{21}Cl_2O_{24}$	110.3 (5)
$C_{b1}C_{b2}C_{b3}$	115.5 (7)	$O_{22}Cl_2O_{23}$	108.4 (5)
		$O_{22}Cl_2O_{24}$	113.3 (4)
		$O_{23}Cl_2O_{24}$	107.6 (5)

[a] The numbers in parentheses are the estimated standard deviations in the last significant figure. [b] Atoms are labeled in agreement with Figure 1.

conformation, one would expect the Cr(III) complex to have both rings in the chair form.

The twist conformation of ring a in the solid could well be due to a combination of two effects: The first is a nonbonded repulsive interaction between the F ligand and the axial protons of $N_{a1}(H_{Na11})$ and $N_{a2}(H_{Na21})$. Such an interaction would tend to push the axial protons away and slightly twist the ring. Such an interaction is not found in ring b since the axial protons are on the opposite side of the chelate ring away from the fluoro ligand. The second effect is hydrogen bonding of the fluoro ligand with H_{Na12} of the nearest neighboring cation. This would tend to reinforce the twisting produced by the nonbonded interaction. In the case where both trans ligands are the same, one would not have unequal nonbonded interactions and both rings will take over the low-energy chair conformation.

The reaction of *trans*-$[Cr(1,3\text{-pn})_2FNH_3](ClO_4)_2$ with liquid ammonia for 18 h at room temperature resulted in some ammination of the starting material. The elution profile of the crude reaction mixture indicated at least one unidentified minor fraction of charge 2+ that eluted from the column slightly ahead of the point where *trans*-$[Cr(1,3\text{-pn})_2FNH_3]^{2+}$ normally eluted from the column. About 8.5% of the chromium was contained in the ions that were eluted from the column with 1.0 M $(NH_4)_2SO_4$, 2 M H_2SO_4 and 6 M H_2SO_4. In the case of crude *trans*-$[Cr(1,3\text{-pn})_2FNH_3]^{2+}$, only about 4% of the total chromium was found in these higher fractions. At this point it would appear that further replacement of the

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ligands by NH_3 to produce a 3+ ion does not occur to any appreciable extent.

The reaction of *trans*-$[Cr(1,3\text{-pn})_2FNH_3](ClO_4)_2$ with concentrated hydrobromic acid resulted, as anticipated, in the removal of the fluoro ligand and its replacement by water. The conclusion that the complex is not anated by the bromide ion is supported by the fact that the electronic spectrum of the reaction product resembles that of $[Cr(NH_3)_5(OH_2)]^{3+}$[15] very closely rather than resembling that of $[Cr(NH_3)_5Br]^{2+}$.[16]

Heating of solid *trans*-$[Cr(1,3\text{-pn})_2FNH_3]BrClO_4$ at 120 °C for 3 days resulted in the loss of 1 mol of ammonia/formula weight of the complex. However, the electronic spectral data for this product do not match those of *trans*-$[Cr(1,3\text{-pn})_2FBr]ClO_4$, one of the anticipated products of the deammination reaction, nor does the aqueous solution spectrum change rapidly with time. Rapid changes in the electronic spectrum would be anticipated if the product were *cis*- or *trans*-$[Cr(1,3\text{-pn})_2FBr]ClO_4$. Thus, the deammination reaction is more complex than it would first appear. Identification and characterization of the products of this reaction are currently under way.

Registry No. *trans*-$[Cr(1,3\text{-pn})_2FNH_3](ClO_4)_2$, 77462-46-5; *trans*-$[Cr(1,3\text{-pn})_2FNH_3]BrClO_4$, 77462-45-4; *trans*-$[Cr(1,3\text{-pn})_2FBr]ClO_4$, 19200-03-4; $[Cr(1,3\text{-pn})_2NH_3(OH_2)]Br_2ClO_4$, 77462-43-2.

Supplementary Material Available: Listings of the observed and calculated structure factors (10 pages). Ordering information is given on any current masthead page.

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Oxy and Thio Phosphorus Acid Derivatives of Tin. 7. Crystal and Molecular Structure of α-(Phenylphosphonato)trimethyltin(IV) at 138 K. A Unique, One-Dimensional, Helical $\{[(CH_3)_3Sn^+][C_6H_5(OH)P(O)OSn(CH_3)_3OP(O)(OH)C_6H_5]^-\}_n$ Polymer[1]

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α-(Phenylphosphonato)trimethyltin(IV) ($C_9H_{15}O_3PSn$) forms colorless crystals, mp = 201 °C dec, in the tetragonal space group $I4_1/acd$ with $a = b = 15.310$ (6) Å, $c = 42.620$ (59) Å, $V = 9989.96$ Å^3, $Z = 32$, and $\rho_{calcd} = 1.706$ g cm^{-3}. The structure was solved by the heavy-atom method to an R value of 0.046 for the 2578 reflections collected at 138 ± 2 K on an Enraf-Nonius CAD/4 automatic diffractometer with the use of Mo K$\bar{\alpha}$ radiation. The molecular units associate to form an infinite, one-dimensional polymer in which planar trimethyltin(IV) groups are axially bridged by –O–P–O– linkages of the phenylphosphonate ligand to yield an almost perfect trigonal bipyramid at tin. The chains are linear at the tin atoms but bent through a bite angle of 115.3 (3)° at phosphorus. The chains propagate helically through the crystal, and each phenylphosphonate P=O and P—OH group is hydrogen bonded, holding adjacent helixes, turning in opposite directions, into two-dimensional sheets. The phenyl groups point outward from the center of the propagating helixes, separating the sheets from one another. Tin atoms occupy two nonequivalent environments, alternating metal atoms forming either two short or two long bonds to oxygen, an arrangement best described as a combination of trimethyltin(IV) cations and bis(phenylphosphonato)trimethyltin(IV) anions, which alternate in the helical array.

We have been investigating the structures of organotin derivatives of thio phosphorus acids[2-4] which combine biocidal activity[5] in both halves of the molecule. The biocidal activity of the organotin moiety is now well established, and such species have found extensive commerical application in the fields of fungicides, insecticides, bacteriocides, and the protection of surfaces (ship hulls, pier pilings, etc.) from attack by marine organisms.[5] Particularly attractive is the idea of combining the organotin species with ligands which are themselves biocidally active with the potential for enhanced activity in the compounds thus formed. The importance of phosphorus acids in in vivo systems has thus led us to investigate the structures of organotin derivatives of ligands deriving from thio or oxy phosphorus acids.[2-4]

The possible structures such systems can adopt are depicted in A–E. The simplest is a monomeric structure containing a monodentate ligand and a four-coordinated tin atom as we have found in the solid (*O,O'*-diethyl dithiophosphato)triphenyltin(IV) $[(C_6H_5)_3SnSP(S)(OC_2H_5)_2]$[3] shown in A, but this configuration is virtually unique among the metal derivatives of such ligands.[6] Much more common is the chelated, five-coordinated monomer structure which can exist with anisobidentate or symmetrical chelation as shown in B and C, respectively. We have found severely anisobidentate chelation in the dimeric tin(II) derivative bis(*O,O'*-diphenyl dithiophosphato)tin(II) $(\{Sn[SP(S)(OC_6H_5)_2]_2\}_2)$[7] as well as

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(1) For Part 6 of this series see: Molloy, K. C.; Hossain, M. B.; van der Helm, D; Zuckerman, J. J., Mullins, F. P. *Inorg. Chem.* **1981**, *20*, 2172.

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(8) The monomeric units in bis(*O,O'*-diphenyl dithiophosphato)tin(II) are held together in part by a weak bridging interaction between a sulfur atom of one chelate ligand and the adjacent tin atom to give the dimeric $[Sn[SP(S)(OC_6H_5)_2]_2]_2$ which contains three-coordinated bridging sulfur atoms in a planar Sn_2S_2 ring.[6]

0020-1669/81/1320-2402$01.25/0