an ideal gas at 1 atm and using the harmonic-oscillator-rigid-rotor approximation.[37] These properties are given for the range 0–2000 K in Table V.

Conclusion. Except for the ^{19}F NMR data, which in the absence of relaxation time measurements[34] are inconclusive, all of the data observed for BrF_3O are in excellent agreement with the predictions made for model I of symmetry C_s. Whereas gaseous, matrix-isolated, and $FClO_3$-dissolved BrF_3O is mainly monomeric, liquid, solid, and HF-dissolved BrF_3O shows pronounced association involving bridging through the axial fluorine atoms.[38]

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Synthesis of Metallic Polythiazyl Halides from Tetrasulfur Tetranitride

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Powdered tetrasulfur tetranitride, S_4N_4, polymerizes at room temperature in the presence of Br_2 or ICl vapor to form a series of highly conducting metallic solid compounds of approximate composition $(SNBr_{0.4})_x$ and $[SN(ICl)_{0.4}]_x$, respectively. Carbon tetrachloride solutions of IBr also react with S_4N_4 at room temperature to give a conducting solid of composition $[SN(IBr)_{0.40}]_x$. When $(SNBr_{0.4})_x$ is heated with pumping at 80 °C, it is converted to a series of highly conducting copper-colored powders of composition $(SNBr_y)_x$ ($y = 0.16$–0.28). The conductivity at room temperature of compressed pellets of the compounds is in the range 10–400 Ω^{-1} cm^{-1} and is almost temperature independent down to 4.2 K.

Considerable interest has recently been shown in the synthesis and study of metallic covalent polymers such as polythiazyl, $(SN)_x$,[1] and the polythiazyl bromides, $(SNBr_y)_x$.[2] The synthesis of $(SN)_x$, from which $(SNBr_y)_x$ is made, first involves the preparation of disulfur dinitride, S_2N_2, from tetrasulfur tetranitride, S_4N_4, by a relatively complicated procedure, followed by its solid-state polymerization.

Recently, independent studies by ourselves[3] and by Street et al.[4] have shown that polycrystalline $(SNBr_y)_x$ and other metallic polythiazyl halides may be synthesized by a very simple procedure directly from S_4N_4. Thus, solid S_4N_4 reacts readily at room temperature with Br_2, ICl, and IBr vapor to give highly conducting black powders. We reported[3] that the metallic compound formed from bromine had the composition $(SNBr_{0.4})_x$ and that pumping at ca. 80 °C for ca. 4 h gave a copper-colored metallic powder of composition $(SNBr_{0.25})_x$. Street et al.[4] reported that heating $(SNBr_{0.4})_x$ at 80 °C for 18 h gave a copper-bronze-colored powder, $(SNBr_{0.07})_x$. The latter authors also stated that IBr and ICl gave conducting powders of unknown composition. In this communication we report experimental details of the synthesis of the products formed from S_4N_4 and Br_2, ICl, and IBr and some of their more important physical properties.

Experimental Section

Apparatus and Techniques. All operations were carried out under a dry argon atmosphere in a Vacuum Atmospheres Corp. Dri-Train

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or by using standard high vacuum system techniques. Pellets for conductivity measurements were made with a Perkin-Elmer infrared KBr die using a pressure of 1500 psi. The samples were first finely powdered using an agate pestle and mortar. Conductivity measurements were carried out on compressed pellets using four-probe direct-current techniques. Contacts to the samples were made with the graphite suspension Electrodag.

Reagents. Tetrasulfur tetranitride, S_4N_4, was prepared from S_2Cl_2 and ammonia.[5] It was purified by recrystallization from chloroform and pumped for several hours on the vacuum system and then sublimed in vacuo before use. The compound was checked by its infrared spectrum[6] (KBr pellet) and by its melting point[7] (found, 185.5–186 °C; lit. values 178 to 187.5 °C). Bromine (Baker analyzed reagent, 99.8%) was stored over a large excess of KBr powder in a glass pressure cell for 3–4 days to remove traces of chlorine. In order to remove traces of moisture the bromine was condensed with liquid nitrogen in the very bottom of a tube filled to a height of 5 cm with P_2O_5 and was distilled from the tube when needed.[8] Finally it was purified by trap-to-trap distillation by passing it three times through −45, −96, and −196 °C traps in a static vacuum. The fraction collecting in the −96 °C trap was used. Iodine monochloride (95+%; Alpha Products) and iodine monobromide (95+%; Alpha Products) were used without further purification. Carbon tetrachloride (5 mL) was dried by distilling under vacuum from P_2O_5 (ca. 8.5 g).

Method. The halogenation of S_4N_4 with Br_2, ICl and IBr was carried out in a 100-mL Erlenmeyer flask fitted with an O-ring joint and a Fischer and Porter Teflon stopcock. A tube containing the bromine was attached to this via an O-ring joint. A Teflon stopcock was located on each side of the O-ring. Ground-glass joints lubricated by Dow Corning silicone lubricant were used for the ICl reactions.

The apparatus was treated with $(CH_3)_3SiCl$ vapor to remove traces of adsorbed water and was then flamed with pumping. The Erlenmeyer reaction flask was evacuated and weighed before use.

The finely powdered S_4N_4 was placed in the flat bottom of the Erlenmeyer flask exposing as large a surface of the material as possible. *Considerable care should be exercised in grinding S_4N_4 because of the possibility of it exploding violently. Only a few milligrams should be ground at a time using an agate mortar and pestle. All appropriate precautions such as the use of a safety shield etc. should be employed.* The whole apparatus was held at room temperature (vapor pressure of Br_2 at 25 °C is ~200 Torr;[9] vapor pressure of ICl at 25 °C is ~28 Torr[10]) and the halogen vapor was permitted to enter the flask. Within 1 min, the S_4N_4 changed from orange-yellow to black. The product appeared to swell and became slightly sticky, but after 5–8 h it was a free-flowing black powder. The reaction was allowed to proceed for ca. 24 h (ca. 34 h with ICl) in order to ensure completion of reaction. Excess halogen was then removed by pumping for approximately 4 h at room temperature (ca. 2 h in the ICl reactions) and the weight of the product was determined by weighing the evacuated Erlenmeyer reaction vessel. The composition of the product could be estimated from the increase in weight of the S_4N_4 during the reaction, assuming that no volatile reaction products were formed. Elemental analyses of the products from several reactions showed that this was a valid method for obtaining the approximate composition. A faint trace of yellow-brown powder appeared on the upper walls of the reaction vessel in some experiments.

When S_4N_4 was permitted to react with bromine vapor under conditions identical with those described above for only 4 h, and was then pumped for 2 h, the product contained unreacted S_4N_4, as determined visually. This unreacted S_4N_4 was removed by repeated washing with CS_2 (dried over P_2O_5) to give a product of composition $(SNBr_{0.40})_x$.

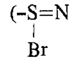
When the black solid of approximate composition $(SNBr_{0.4})_x$ was heated for about 4 h at ca. 80 °C, bromine and a small amount of brown-black solid were driven off and were collected in a trap cooled in liquid nitrogen. A small amount of S_4N_4 (identified by x-ray powder diffraction[11]) collected on the upper walls of the Erlenmeyer reaction flask. The product of lower bromine content remained as a copper-colored powder in the bottom of the flask.

In the case of the reaction involving ICl, the Erlenmeyer flask was evacuated for 2 h after ca. a 20-h reaction time and the black powder was finely powdered again in the drybox using an agate pestle and mortar and was then retreated with the ICl vapor for an additional 13.5 h. This treatment was necessary since preliminary experiments had shown that treatment of finely powdered S_4N_4 with ICl vapor at room temperature for as much as 24 h yielded a product which still contained S_4N_4 as determined visually when a particle was crushed. The final product contained no lines characteristic of S_4N_4 in the x-ray powder diffraction pattern. The most intense lines had *d* values of 6.55, 3.66, and 3.30 Å.

An essentially identical apparatus was used for the reaction of S_4N_4 with IBr, but since IBr has a very small vapor pressure at room temperature, a saturated solution of IBr in CCl_4 was placed in the side-arm tube. The powdered S_4N_4 immediately turned black when the stopcock containing the IBr solution was opened and the vapors contacted the S_4N_4. In a typical experiment, approximately 6 mL of the IBr solution was poured into the Erlenmeyer flask containing ca. 250 mg of S_4N_4. No visual change could be observed in the intensely colored dark solution or solid. After 30 h at room temperature the solution was decanted back into the side-arm tube and the solid was pumped at room temperature for 2 h. The product was a free-flowing black powder, but it still contained S_4N_4, as determined visually. The material was powdered again in the drybox and was allowed to react with the IBr solution for an additional 10 h. The x-ray powder diffraction pattern of this material contained no lines characteristic of S_4N_4; there were only two sharp lines at $d = 6.70$ Å and $d = 3.35$ Å.

Results and Discussion

In 1896 Clever and Muthmann obtained garnet red crystals of composition $S_4N_4Br_6$ by the reaction of bromine vapor with solid S_4N_4 during several days.[12] They also stated that a bronze-colored solid compound, $S_4N_4Br_4$, was formed by the reaction of excess bromine with a solution of S_4N_4 in CS_2 after 24 h. Because of the insolubility of $S_4N_4Br_4$ in organic solvents and its deep bronze color, Goehring considered $S_4N_4Br_4$ to be a linear polymer[13]



$$(\underset{\underset{Br}{|}}{-S}=N-)_x$$

Heal later reinvestigated the reaction of bromine with S_4N_4 in CS_2 solution and concluded that the bronze-colored material could be $S_3N_2Br_2$.[14]

In the present study we have found that finely powdered orange-yellow S_4N_4 reacts instantly at room temperature with approximately 200 Torr of bromine vapor and immediately changes to a dark black powder (see Table I). A slow gas–solid reaction occurs at room temperature and after a 24-h reaction time, x-ray powder studies cannot detect the presence of unreacted S_4N_4. An apparently labile equilibrium

$$(SNBr_{1.5})_x(s) \rightleftarrows (SNBr_y)_x(s) + (x(1.5 - y)/2)Br_2(g)$$

exists at room temperature as indicated by the observation that when the increase in weight of the product is determined under a vapor pressure of ca. 200 Torr of bromine, its composition is ca. $(SNBr_{1.5})_x$. Pumping at room temperature for ca. 4 h shifts the equilibrium to the right to give black powders where $y \approx 0.4$. The actual composition of the product finally obtained varies slightly according to the actual time and temperature of pumping. There is no obvious change in appearance of the solid during pumping at room temperature.

When the product is pumped at ca. 80 °C for ca. 4 h, a greater amount of bromine is liberated together with a small amount of black-brown solid of unknown composition and a trace of S_4N_4. Since no S_4N_4 could be detected in the $(SNBr_{0.4})_x$ employed, it appears that some irreversible decomposition of the $(SNBr_{0.4})_x$ to S_4N_4 and Br_2 occurs during heating. The black solid changes to a copper-colored powder during this process. The value of *y* in the final product varies from 0.16 to 0.28 depending on minor variations in the time and temperature of heating. It appears that bromine can be removed constantly and controllably since Street et al.[4] note that $(SNBr_{0.07})_x$ is obtained after 18 h of pumping at 80 °C.

Powders of composition $y = 0.40$–0.43 and $y = 0.16$–0.28 can be easily compressed into black-blue and dark copper-colored disk-shaped pellets, respectively. *On occasions, the powder will explode during compression.*

Table I. Synthesis and Conductivity of Halogen Derivatives of S_4N_4

Wt of S_4N_4, g (mmol)	Halogen	Reacn time, h	Compn from wt increase	Compn from anal.[a]	Compn from anal. after heating at ca. 80 °C for ca. 4 h	Conductivity[o] (25 °C) before heating, Ω^{-1} cm^{-1}	Conductivity[o] (25 °C) after heating, Ω^{-1} cm^{-1}
0.1786 (0.97)	Br_2	24	$SNBr_{0.46}$		$SNBr_{0.25}$ [b,c]		99
0.4745 (2.60)	Br_2	24	$SNBr_{1.50}$ [d] $SNBr_{0.41}$		$SNBr_{0.25}$ [e]		86
0.2846 (1.55)	Br_2	24	$SNBr_{0.46}$	$SNBr_{0.43}$ [c,f]			
0.3195 (1.74)	Br_2	24	$SNBr_{0.40}$	$SNBr_{0.41}$ [g]	$SNBr_{0.165}$ [h]	350	100
0.4962 (2.70)	Br_2	24	$SNBr_{0.41}$	$SNBr_{0.40}$ [i]	$SNBr_{0.28}$ [j]	350	
0.5100 (2.77)	Br_2	24	$SNBr_{0.40}$		$SNBr_{0.22}$ [k]		350
0.4014 (2.18)	ICl	33.5	$SN(ICl)_{0.37}$	$SN(ICl)_{0.43}$ [l]		11	
0.2663 (1.44)	ICl	30	$SN(ICl)_{0.32}$	$SN(ICl)_{0.43}$ [m]			
0.3805 (2.065)	IBr	30	$SN(IBr)_{0.35}$	$SN(IBr)_{0.40}$ [n]		50	

[a] Analyses performed by Galbraith Laboratories, Inc., Knoxville, Tenn. [b] Anal. Calcd for $SNBr_{0.25}$: S, 48.54; N, 21.21; Br, 30.25. Found: S, 48.36; N, 21.42; Br, 30.10 (total 99.88). [c] X-ray diffraction data for this material are reported in Table II. [d] Weighed under 200 Torr vapor pressure of Br_2. [e] Found: S, 48.76; N, 21.04; Br, 30.15 (total 99.95). [f] Calcd for $SNBr_{0.43}$: S, 39.87; N, 17.40; Br, 42.73. Found: S, 40.00; N, 17.51; Br, 42.63 (total 100.14). [g] Calcd for $SNBr_{0.41}$: S, 40.67; N, 17.77; Br, 41.56. Found: S, 40.83; N, 17.86; Br, 41.16 (total 99.85). [h] Calcd for $SNBr_{0.165}$: S, 54.11; N, 23.64; Br, 22.25. Found: S, 54.27; N, 23.47; Br, 22.21 (total 99.95). [i] Calcd for $SNBr_{0.40}$: S, 41.09; N, 17.95; Br, 40.96. Found: S, 41.25; N, 18.03; Br, 41.00 (total 100.28). [j] Calcd for $SNBr_{0.28}$: S, 46.82; N, 20.46; Br, 32.72. Found: S, 46.79; N, 20.41; Br, 32.92 (total 100.12). [k] Calcd for $SNBr_{0.22}$: S, 50.37; N, 22.00; Br, 27.63. Found: S, 50.27; N, 21.72; Br, 28.06 (total 100.05). [l] Calcd for $SN(ICl)_{0.43}$: S, 27.66; N, 12.09; I, 47.09; Cl, 13.16. Found: S, 27.86; N, 12.22; I, 46.89; Cl, 13.19 (total 100.16). [m] Calcd for $SN(ICl)_{0.43}$: S, 27.66; N, 12.09; I, 47.09; Cl, 13.16. Found: S, 27.64; N, 12.14; I, 46.96; Cl, 13.07 (total 99.81). [n] Calcd for $SN(IBr)_{0.40}$: S, 24.90; N, 10.88; I, 39.41; Br, 24.81. Found: S, 24.93; N, 11.03; I, 39.50; Br, 24.70 (total 100.16). [o] The values given are typical average values.

Table II. X-Ray Powder Diffraction Data[a] for $(SNBr_y)_x$ (d Spacing, Å; Cu Kα)

$(SNBr_{0.25})_x$[b] from $(SN)_x$ + Br_2	$(SNBr_{0.25})_x$ from S_4N_4 + Br_2	$(SNBr_{0.43})_x$ from S_4N_4 + Br_2
3.66 s, br	3.68 s, br	3.35[d] w, sh
3.16 s, br	3.18 vs, br	3.18 s, br
2.82 ms, br	2.84 ms, br	
2.43[c] vvw, br		
	2.30[d] w, sh	
	2.15[d] w, sh	
2.08 vvw, br	2.08 w, br	2.09 w, br
2.00[d] vvw, sh		
1.90 w, br		
1.75 vw, br		
1.58 w, br	1.58 vw, br	

[a] Abbreviations: s, strong; m, medium; w, weak; v, very; br, broad; sh, sharp. [b] This compound was prepared by heating brominated $(SN)_x$ of composition $(SNBr_{0.4})_x$ at ca. 90 °C with pumping for ca. 50 h. Anal. Calcd for $SNBr_{0.25}$: S, 48.55; N, 21.21; Br, 30.24. Found: S, 48.69; N, 21.34; Br, 30.09 (total 100.12). See also ref 2. [c] Poorly resolved reflection. [d] These sharp lines differ in intensity from all other lines in the pattern which are diffuse and broad.

X-ray powder patterns of two samples of brominated S_4N_4 are given in Table II. For comparison the pattern of $(SNBr_{0.25})_x$ obtained from brominated $(SN)_x$ is also included. All three of these compounds give x-ray powder patterns which have broad, diffuse lines, indicating poor crystallinity of the products. The powder patterns of the two compounds $(SNBr_{0.25})_x$ obtained from heating the product of $(SN)_x$ with bromine and the product of S_4N_4 with bromine, respectively, are almost identical, in both line positions and in relative intensities. This strongly suggests that these two products are identical, or at least have very similar polymeric structures. The presence of (–SN–) polymer chains has been confirmed by the x-ray studies of Iqbal et al.[15] and of Street et al.[2] in brominated $(SN)_x$. The two sharp lines in the pattern of $(SNBr_{0.25})_x$ synthesized from S_4N_4 do not appear in the brominated $(SN)_x$ analogue. However, it has not been possible to determine if these lines arise from impurity or are intrinsic to the compound.

It should be noted that the powder patterns of $(SNBr_{0.43})_x$ obtained from S_4N_4 and Br_2 are of poorer quality than those of $(SNBr_{0.25})_x$. The two lines in the former compound at d = 3.18 and 2.09 Å are also present in materials of composition $(SNBr_{0.25})_x$. However, the absence of the strong line at d = 3.66 Å in the powder patterns of $(SNBr_{0.43})_x$ compound suggests that there are significant structural differences between it and the compounds of lower bromine content.

Tetrasulfur tetranitride also reacts readily with ICl and IBr to give $[SN(ICl)_{0.43}]_x$ and $[SN(IBr)_{0.40}]_x$, respectively. It is interesting to note that these interhalogen derivatives contain twice the number of halogen atoms per SN unit as does $(SNBr_{0.4})_x$. This might imply that only the smaller halogen (Cl or Br) in these interhalogen compounds can occupy the positions occupied by bromine in $(SNBr_{0.4})_x$. Furthermore, it should be noted that the halogenation of S_4N_4 apparently occurs more readily than the halogenation of $(SN)_x$. Thus, treatment of S_4N_4 with bromine gives a maximum bromine content before pumping corresponding to $SNBr_{1.5}$ whereas the composition of the bromine derivative formed from $(SN)_x$ under comparable conditions is only $SNBr_{0.5}$.[2] Also, treatment of $(SN)_x$ crystals with ICl or IBr results in only superficial surface reaction under conditions which yield extensive halogenation with S_4N_4. When $[SN(IBr)_{0.4}]_x$ is heated at 80 °C with pumping, it loses iodine and bromine in equimolar amounts, apparently as IBr.

Since $(SN)_x$[1,16] and $(SNBr_y)_x$ (y = 0.4, 0.27)[15] contain chains of multiply bonded S–N groups, it is apparent that the reaction of solid S_4N_4 which has a cradle-like cyclic alternating S–N structure[17] undergoes an irreversible ring opening and polymerization during reaction with halogen. It is possible that the materials derived from the reaction with S_4N_4 may contain some cross-linking between S–N chains. The very high conductivities observed for compressed pellets (see Table I) strongly suggest long S–N polymeric chains in these materials. The conductivity results are therefore qualitatively in agreement with the earlier single-crystal conductivity studies[2] of brominated $(SN)_x$ and imply metallic behavior. The conductivities of $(SN)_x$ compressed pellets and $(SNBr_y)_x$ compressed pellets synthesized from S_4N_4 were studied as a function of temperature with results indicating nearly temperature-independent values down to 4.2 K. These data and the absolute values listed in Table I imply that the halogen derivatives synthesized by the techniques described in this paper have a high intrinsic metallic conductivity with the compressed pellet values limited by anisotropy and interparticle contacts.

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Registry No. S_4N_4, 28950-34-7; Br_2, 7726-95-6; ICl, 7790-99-0; IBr, 7789-33-5; $SNBr_{0.25}$, 65995-70-2; nitrogen bromide sulfide, 65995-69-9; nitrogen chloride iodide sulfide, 65995-68-8; nitrogen bromide iodide sulfide, 65995-67-7.

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Effect of Para Substituent on Rates of Phenyl Ring Rotation in Gallium Complexes of Para-Substituted Tetraphenylporphyrins

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Rates of phenyl ring rotation in gallium chloro complexes of para-substituted tetraphenylporphyrins were studied by variable-temperature ^1H NMR of the phenyl resonances. Para substituents examined were trifluoromethyl, chloro, methyl, isopropyl, methoxy, and diethylamino. Activation parameters obtained by total line shape analysis are in the ranges $\Delta G^{\ddagger}_{298} = 12.8\text{–}15.0$ kcal/mol, $\Delta H^{\ddagger} = 9.2\text{–}12.5$ kcal/mol, and $\Delta S^{\ddagger} = -12.4$ to -8.1 eu. Rates of ring rotation for the gallium complexes are the fastest observed to date for metallotetraphenylporphyrin complexes. Rates are faster for electron-donating substituents than for electron-withdrawing substituents but do not give a linear correlation with Hammett σ_p values.

The effect of para substituent and metal ion on the rate of phenyl ring rotation in ruthenium carbonyl, indium chloro, and titanyl complexes of para-substituted tetraphenylporphyrins has been reported in a previous paper of this series.[1] Herein we report the rates of phenyl ring rotation for gallium complexes of the same series of para-substituted tetraphenylporphyrins.

Experimental Section

Physical Measurements. Infrared spectra were recorded as Nujol or halocarbon mulls on Perkin-Elmer 237 or 337 spectrophotometers. Visible spectra were obtained in chloroform solutions on Cary 14 or Beckman Acta V spectrophotometers. Data are given below with wavelengths in nm, and log ϵ is enclosed in parentheses. ^1H NMR spectra were run at power levels well below saturation on a Varian HA-100 spectrometer equipped with a variable-temperature probe. Spectra were obtained on samples in 1:3 v/v trichloroethylene/1,1,2,2-tetrachloroethane (1:3 $C_2HCl_3/C_2H_2Cl_4$), unless otherwise noted, with the spectrometer locked on the $C_2H_2Cl_4$ resonance. ^1H NMR chemical shifts were measured with respect to $C_2H_2Cl_4$ and are reported in ppm downfield of Me_4Si using a correction of 5.96 ppm for the chemical shift of $C_2H_2Cl_4$. In order to indicate slow-exchange chemical shift differences, values of chemical shifts reported below are at −40 °C, unless otherwise noted.

Preparation of Compounds. Porphyrin free bases were prepared and characterized by literature methods: $H_2(p\text{-}CF_3\text{-}TPP)$;[2,3] $H_2(p\text{-}R\text{-}TPP)$, R = Cl, Me, OMe;[4,5] $H_2(p\text{-}i\text{-}Pr\text{-}TPP)$;[6] $H_2(p\text{-}Et_2N\text{-}TPP)$;[7] $H_2(o\text{-}Me\text{-}TPP)$.[8]

Gallium Complexes. The procedure used to synthesize the gallium complexes was analogous to that reported for the indium complexes.[3] Porphyrin (0.5 mmol) and $(NH_4)Ga(SO_4)_2\cdot 12H_2O$ (1.0 mmol) were refluxed in 250 mL of acetic acid containing excess sodium acetate (0.10 mol). The reaction was monitored by the disappearance of the free-porphyrin band at ca. 515 nm in the visible spectrum. When the reaction was complete, the acetic acid was removed in vacuo and the product purified by chromatography on activity IV Baker 0537 alumina. Details of reaction time, chromatography conditions, yield, and characterization data are given below for individual complexes.

$Ga(p\text{-}CF_3\text{-}TPP)OH$. The reaction time was 12 h. The crude product was chromatographed on a column in $CHCl_3$. Elution with $CHCl_3$ gave a trace of free porphyrin followed by the product. Recrystallization was from $CHCl_3$/heptane; yield 84%. Visible spectrum: 400 sh (4.60), 420 (5.82), 515 sh (3.48), 551 (4.37), 590 (3.43). ^1H NMR (−40 °C): pyrrole H, 9.09, singlet; *o*-H, 8.54, 8.26, doublets; *m*-H, 8.12, 8.05, doublets. Anal. Calcd for $C_{48}H_{25}N_4F_{12}GaO$: C, 59.35; H, 2.59; N, 5.77; Cl, 0.0. Found: C, 59.16; H, 2.76; N, 5.84; Cl, 0.0.

$Ga(p\text{-}CF_3\text{-}TPP)Cl$. A total of 150 mg of $Ga(p\text{-}CF_3\text{-}TPP)OH$ was boiled in 15 mL of $C_2H_2Cl_4$ for 3.5 h. The conversion from axial

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