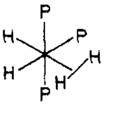
et al.[3] have shown that $[FeH(H_2)dpe_2]^+$ has a nonclassical structure. $[OsH_4(PMe_2Ph)_3]$ on the other hand has a classical structure[5] according to neutron diffraction work; although a nonclassical model was probably not considered, it would not be expected to have escaped detection. We therefore turned our attention to the problem of determining the true structure of the title complexes by the T_1 method.[4b]

$FeH_4(PEtPh_2)_3$ (**1**) was prepared by the method of Aresta et al.[6] This complex shows a quartet at δ -11.5 (^2J(P,H) = 27 Hz) in the ^1H NMR (toluene-d_8) at 20 °C under H_2, but at low temperature, the multiplet collapses to a broad feature. At 205 K, the apparent T_1 for this resonance at 250 MHz is 24 ms.[4d] This is in the range previously identified as appropriate for a nonclassical structure.[4b,c] The short T_1's are not due to fast exchange between **1** and free H_2 because irradiation of the resonance for free H_2 seen at δ 4.56 did not affect the appearance of the metal hydride resonance.[4e] As shown by Aresta et al.,[6] the IR of **1** shows not only bands due ν(classical Fe–H) but also a band at 2380–2400 cm^{-1}, not present in $FeH_2(PEtPh_2)_3$. These bands could not be assigned by Aresta et al., but by analogy with similar bands in known dihydrogen complexes,[1,2] we can now assign them to ν-(Fe(H_2)). The fact that a simple quartet is observed at 25 °C in the ^1H NMR shows that exchange between the Fe(H_2) and Fe–H groups is fast at this temperature but may begin to be frozen out on cooling.

$RuH_4(PPh_3)_3$ (**2**)[7] has a broad RuH resonance at 25 °C in the ^1H NMR (toluene-d_8) at δ -7.06. the T_1 of this resonance at 205 K is 38 ms, again suggesting a nonclassical structure. The IR spectrum shows classical Ru–H bands at 1942 cm^{-1}. The feature corresponding to ν(Ru(H_2)) is probably buried under the C–H vibrations or is too weak to be seen. These results confirm the speculation by Ashworth and Singleton[7] that this complex might be better described as an H_2 complex.

$OsH_4(PPh_3)_3$[8] was too insoluble for the T_1 to be determined at 205 K. The more soluble derivative OsH_4(Pp-tolyl$_3$)$_3$ (**3**) gave the value 820 ms, appropriate to a classical structure. **3** is much less reactive than **1** and **2** and does not bind N_2 with release of H_2, facts that fall naturally into place given the difference in structure.

The stereochemistry shown in **4** is the most likely one for complexes **1** and **2**, because in all cases studied to date, an H_2 ligand has been trans to the ligand of highest trans effect in the molecule.



4

We can now see that MH_4L_3 complexes of the iron triad are really M(II) for Fe and Ru but **3** is genuinely Os(IV). The higher M–H bond energies of third-row metals may be responsible for the change in structure. A nonclassical dihydrogen complex can be seen as a way for a metal to gain the M–H_2 binding energy (ca. 3–10 kcal/mole in cases[4c] shere this has been measured) without losing much of the H–H binding energy. In other words we believe thermodynamics rather than kinetics decides the structural outcome. The rapid proton exchange between M(H) and M(H_2), which lead to the fluxionality, may go via the classical tetrahydrides.

Acknowledgment. We thank ARO for funding and Dr. G. Hlatky for discussions.

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Stable Trifluoromethylated Allyl and Heteroallyl Anions: Structure and Dynamic Properties

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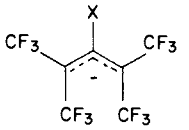
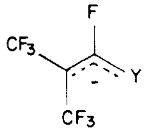
The tris(dimethylamino)sulfonium (TAS) cation uniquely stabilizes fluorinated anions. Unprecendented, isolable salts of perfluorinated tertiary carbanions[1a] and alkoxides[1b] have been described, and we report here the synthesis and characterization of isolable salts of fluorinated allyl and heteroallyl anions. The dynamic properties of these anions in solution are discussed and X-ray structural evidence for remarkably distorted 1,1,3,3-tetrakis(trifluoromethyl)allyl anion geometries (C_2 symmetry) is presented.

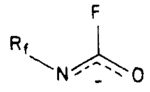
The salts **1a**, **2a**, **2b**, and **3** were prepared in nearly quantitative yields by simply adding the unsaturated fluoride acceptors $(CF_3)_2C{=}C{=}C(CF_3)_2$,[2] $(CF_3)_2C{=}C{=}O$,[3] $(CF_3)_2C{=}C{=}S$,[4] and $R_fN{=}C{=}O$,[5] respectively, to $TAS^+(CH_3)_3SiF_2^-$ in THF at low temperatures. Similarly, $(C_2H_5)_4N^+$ CN^- and 4-(dimethylamino)pyridine (DMAP) with $(CF_3)_2C{=}C{=}C(CF_3)_2$ gave **1b** and **1c**, respectively. Although some of these anions have been reported previously,[6] the cations employed here provide well-characterized, analytically pure solids of high stability that are suitable for spectroscopic and structural analyses[7,8] (Table I).



1a, X = F
b, X = CN
c, X = DMAP$^+$



2a, Y = O
b, Y = S



3a, R_f = CF_3
b, R_f = n-C_3F_7

The single-crystal X-ray diffraction analysis of zwitterion **1c** reveals an unusual twisted structure[9] (Figure 1). Features of this

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(1) (a) Smart, B. E.; Middleton, W. J.; Farnham, W. B. *J. Am. Chem. Soc.*, in press. (b) Farnham, W. B.; Smart, B. E.; Middleton, W. J.; Calabrese, J. C.; Dixon, D. A. *J. Am. Chem. Soc.* **1985**, *107*, 4565.

(2) Aronov, Y. E.; Cheburkov, Y. A.; Knunyants, I. L. *Bull. Acad. Sci. USSR, Ser. Khim.* **1967**, 1689.

(3) England, D. C.; Krespan, C. G. *J. Am. Chem. Soc.* **1966**, *88*, 5582.

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(5) Middleton, W. J. *J. Org. Chem.* **1984**, *49*, 4541.

(6) (a) Gervits, L. L.; Rozov, L. A.; Mirzabekyants, N. S.; Makarov, K. N.; Zeifman, Yu. V.; Cheburkov, Y. A.; Knunyants, I. L. *Bull. Akad. Sci. SSSR, Div. Chem. Sci.* (*Engl. Transl.*) **1976**, *7*, 1592. (b) Knunyants, I. L.; Rokhlin, E. M. *Soviet Scientific Rev., Sect. B* **1984**, *5*, 97.

(7) Except for **1c**, these salts are sensitive to water. Their preparation and manipulation were routinely carried out in a drybox.

(8) ^{19}F NMR (solvent, T, °C): **1a** (THF-d_8, 24) δ −52.50 (d, 12F, J = 18.5 Hz), −74.02 (13-line m, 1 F, J = 18.5 Hz); **1b** (THF-d_8, −20) δ −49.75 (m), −55.91 (m); **1c** (o-xylene-d_{10}/EtCN, 24) δ −50.11 (m), −56.66 (m); **2a** (DMF-d_7, 50) δ 5.03 (qq, 1 F, J = 19.5, 11.8 Hz), −47.92 (dq, 3 F, J = 19.5, 11.8 Hz), −49.41 (dq (quintet), 3 F, J = 11.8, 11.8 Hz); **2b** (CD_3CN, 25) δ 4.41 (qq, 1 F, J = 15, 23 Hz), −52.9 (dq, 3 F, J = 23, 10 Hz), −54.0 (dq, 3 F, J = 15, 10 Hz); **3a** (DMF-d_7, −50) major isomer δ 1.00 (q, 1 F, J = 11.3 Hz), −45.30 (d, 3 F, J = 11.3 Hz); minor isomer δ 11.4 (q, 1 F, J = 11 Hz), −40.7 (d, 3 F, J = 11 Hz); **3a** (DMF-d_7, 50) δ 1.55 (s, 1 F, $w_{1/2}$ = 105 Hz), −45.75 (s, 3 F, $w_{1/2}$ = 14 Hz); **3b** (THF, −50 °C) major isomer δ 5.5 (br s, 1 F), −79.04 (t, J = 7.6 Hz, 3 F), −79.4 (br s, 2 F), −127.53 (t, J = 10 Hz, 2 F); **3b** (DMF-d_7, 23 °C) δ 7.05 (br s, $w_{1/2}$ = 30 Hz, 1 F), −77.75 (br s, $w_{1/2}$ = 30 Hz, 2 F), −79.48 (t, J = 7.5 Hz, 3 F), −127.06 (t, J = 10 Hz, 2 F). UV (CH_3CN) λ_{max} (nm): **1a** 270 (ϵ 25 900); **1b** 352 (ϵ 15 200), 224 (ϵ 5400); **1c** 298 (ϵ 29 600), 216 (ϵ 7600).

(9) Crystal structure information for **1c**: orthorhombic; space group *Pbca* (No. 61); a = 11.262 (1) Å, b = 17.022 (2) Å, c = 17.167 (2) Å, V = 3291 Å^3, Z = 8; T = −100 °C. Data collected on Syntex R3 diffractometer, graphite monochromator, Mo Kα, λ = 0.71069 Å; 3787 reflections, 4.0 < 2θ < 55.0°; 1653 unique reflections with $I > 3.0\sigma(I)$. Structure solved by direct methods (MULTAN); full-matrix, least-squares refinement. All hydrogens refined isotropically; other atoms, anisotropically. Final R = 0.043, R_w = 0.042 for 293 independent variables.

0002-7863/86/1508-3125$01.50/0

Table I. Physical and Dynamic Properties of Fluorinated Allyl and Heteroallyl Anions

struct[a]	mp, °C[b]	recryst solvent	$\Delta G^{\ddagger}_{rot}$ (T, °C), kcal/mol
1a	gum		11.6 (−10)[c]
1b	219–220 dec	THF/Et_2O	14.0 (48)[c]
1c	162–164 dec	toluene	17.5 (130)[d]
2a	201–202	THF/Et_2O	>19[e] (75)
2b	65–70		
3a	129–130	THF/Et_2O	[f]
3b	35–36	THF/Et_2O	[f]

[a] Counterion for all anions is TAS^+, except for **1b** (Et_4N^+) and **1c** (zwitterion). [b] Satisfactory elemental analyses (C, H, N, F, S) were obtained for each compound. [c] THF-d_8 solvent. [d] o-Xylene/propionitrile solvent. [e] In DMF at 75 °C, k <10 s^{-1} since the CF_3–CF_3 coupling patterns (J = 11.8 Hz) at 75 and 24 °C are identical. Lower limit for $\Delta G^{\ddagger}$ was estimated by using the Eyring equation. [f] Not measureable because of fluoride ion dissociation, see text.

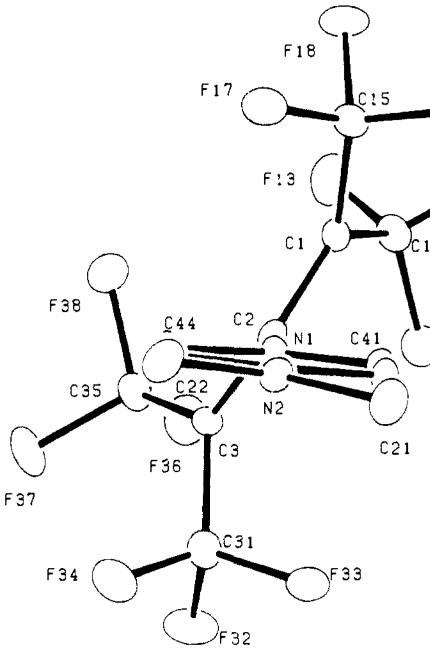

Figure 1. X-ray crystal structure of zwitterion **1c** viewed from the end of the pyridine ring along a pseudo C_2 axis (atoms are shown at the 25% probability level; hydrogen atoms removed for clarity). Featured dihedral angles (deg) between indicated planes, bond angles (deg), and bond lengths (Å): C_{11}–C_1–C_{15}/C_1–C_2–C_3 = 30.0; C_{31}–C_3–C_{35}/C_1–C_2–C_3 = 36.5; C_{45}–N_1–C_{41}/C_1–C_2–C_3 = 60.5; $\angle C_1$–C_2–C_3 = 129.5 (4); $\angle C_{11}$–C_1–C_{15} = 114.6 (3); $\angle C_{15}$–C_1–C_2 = 123.2 (4); $\angle C_{11}$–C_1–C_2 = 122.4 (4); C_1–C_2 = 1.381 (5); C_1–C_{11} = 1.475 (5); C_2–N_1 = 1.473 (4).

propeller-shaped molecule (approximate C_2 symmetry with the idealized axis passing through C_2, N_1, and N_2) include (a) local planar geometry at the allyl termini (C_1, C_3) and (b) 30° and 36° dihedral angles between the "CF_3-substituted blades" and the $C_1C_2C_3$ allyl carbon plane. Surprisingly, the $\angle C_1C_2C_3$ (129.5°) and the C_1–C_2 bond length in this highly distorted molecule are nearly the same as the corresponding bond angle and bond length claimed for the allyl anion itself.[10]

The ^{19}F NMR spectrum of **1a** in THF-d_8 at 24° C shows only two resonances in the ratio of 12:1, which indicates fast C–C bond rotation.[11] The variable-temperature ^{19}F NMR spectra of **1a** are shown in Figure 2. Barriers to rotation about the C_1–C_2 bonds in **1a–c**, which all exhibit dynamic behavior, were estimated from variable-temperature NMR spectra, using a two-site model that neglects F–F couplings.[12] The $\Delta G^{\ddagger}$ values are listed in Table I.

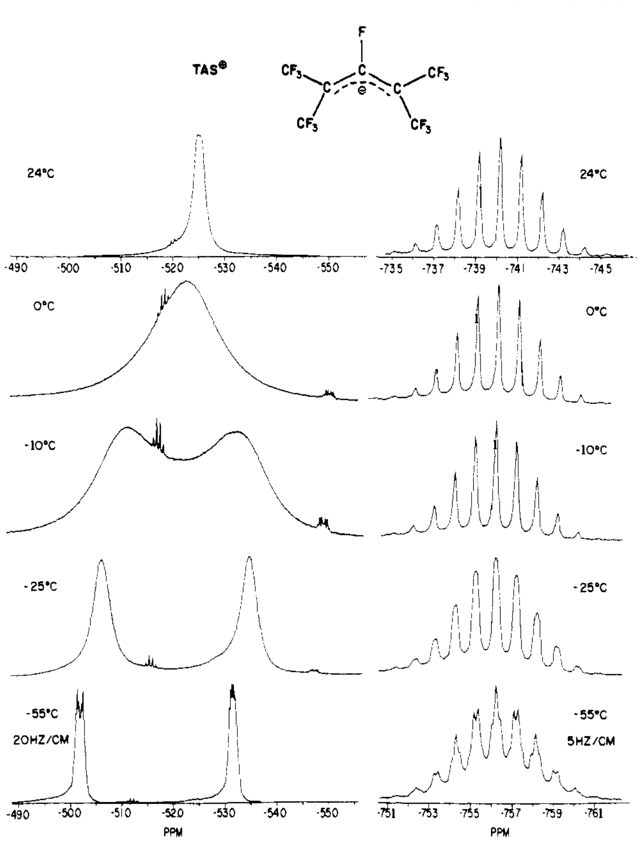

Figure 2. Variable-temperature ^{19}F NMR spectra of TAS^+ **1a** (THF-d_8).

In contrast to the line broadening reported for other salts of **2a**,[6] the TAS salt shows no loss of coupling up to 75 °C, which implies a barrier to rotation of a least 19 kcal/mol (Table I, footnote *e*). Anions **3a,b** exist in two isomeric forms (92:8 and 93:7, respectively) at low temperatures (DMF, −50 °C).[8] Unlike anion **1a**, their isomer interconversions are accompanied by loss of F–F couplings,[8] which indicates fluoride ion dissociation and prevents the measurement of rotational barriers for **3a,b**.

The $\Delta G^{\ddagger}$ for **1a–c** may be compared with the 18 kcal/mol barrier for allylcesium, which is regarded as a lower limit to the rotational barrier of the free allyl anion in solution.[13] We believe the substantially lower barriers for the CF_3-substituted anions can be attributed largely to the raised ground-state energies of their twisted forms relative to a transition state of C_s symmetry.[14]

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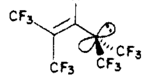
(10) Schleyer, P. v. R. *J. Am. Chem. Soc.* **1985**, *107*, 4793. Ab initio values for allyl anion: $\angle$CCC = 131.6–132.2°, r(C–C) = 1.387–1.388 Å.

(11) Exchange of the CF_3 groups via C–F bond dissociation to F^- and $(CF_3)_2C{=}C{=}C(CF_3)_2$ is ruled out because couplings between CF_3 and CF nuclei are maintained (Figure 1). The rate of CF_3 exchange at −10 °C is ca. 1.2×10^3 s^{-1}, whereas the four-bond F–F coupling constant is 18.5 Hz.

(12) Errors due to this approximation should be negligible since the coupling constants are very small ($J_{CF_3-CF_3}$ = 4.5–5.5 Hz, J_{CF_3-CF} = 18.5 Hz) relative to the CF_3 chemical shift differences (558 (**1a**), 1159 (**1b**), 1233 Hz (**1c**) at 188.23 MHz). See: Binsch, G.; Kessler, H. *Angew. Chem., Int. Ed. Engl.* **1980**, *19*, 411. Chemical shifts for structures **1a–c** are temperature-dependent. For each structure the CF_3 chemical shift difference at the coalescence temperature was obtained by linear extrapolation of data obtained at lower temperatures where site exchange is not fast enough to cause line broadening.

(13) Thompson, T. B.; Ford, W. T. *J. Am. Chem. Soc.* **1979**, *101*, 5459.

(14) The lower barrier to rotation in **1a** vs. **1b** is consistent with the relative stabilizing interactions between the σ^* C–F vs. σ^* C–CN orbitals and the adjacent lone pair (hyperconjugation)[15] in transition state A. The higher



A

barrier in **1c** probably reflects the steric bulk of the 2-DMAP substituent. The similarly high barriers in **2a** (>19 kcal/mol) and oxallyl anion itself (>21 kcal/mol)[16] suggest that a single pair of *gem*-CF_3 groups is not sufficient to appreciably lower the barriers in allyl anions.

(15) (a) Apeloig, Y.; Rappoport, Z. *J. Am. Chem. Soc.* **1979**, *101*, 5095. (b) Apeloig, Y.; Karni, M.; Rappoport, Z. *J. Am. Chem. Soc.* **1983**, *105*, 2784.

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J. Am. Chem. Soc. **1986**, *108*, 3127-3128

Further studies to elucidate the electronic and steric effects of fluorination on the physicochemical properties of allyl and heteroallyl anions (and their corresponding free radicals) are in progress.

Supplementary Material Available: Tables of atomic coordinates, thermal parameters, and structure amplitudes (5 pages). Ordering information is given on any current masthead page.

Synthesis of Skeletally Stabilized Acyclic Triphosphazanes

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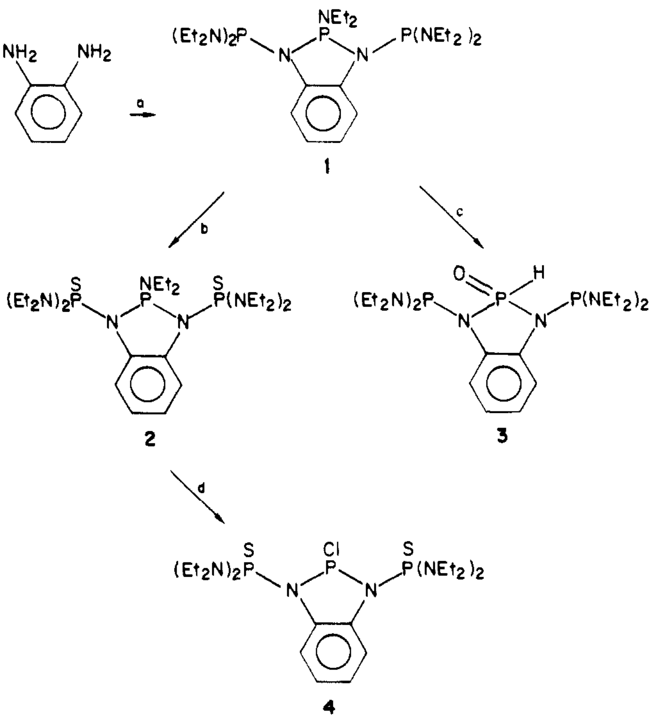
In contrast to the many studies of linear phosphazenes reported,[1] relatively few dealing with analogous phosphazanes $-(PRNR'-)_n$ containing more than two phosphorus atoms have appeared. The absence of acyclic phosphazanes (where $n > 2$) apparently results from the tendency, in their formation reactions, to form low molecular weight four-, six-, or eight-membered rings.[2] We now wish to report the preparation of a new class of skeletally stabilized triphosphazanes, which, because of their high stability, allow first studies of acyclic phosphazane chemistry and offer potential as novel synthons in higher oligomer/polymer formation reactions.

Reaction of 1,2-diaminobenzene with excess $P(NEt_2)_3$ for 10 h at 85 °C results in near quantitative formation of triphosphazane **1** (Scheme I). Excess $P(NEt_2)_3$ is removed in vacuo. Passage of **1** in toluene through a 5-cm silica gel column, followed by removal of toluene in vacuo, yields pure **1**. **1** has not been obtained crystalline. However, in 4 h with 2 equiv of S_8 at 25 °C in toluene it under goes oxidation selectively and quantitatively at the exo phosphorus atoms [P(1) and P(3)] to form **2**. **2** is obtained crystalline from CH_2Cl_2 (mp 115-118 °C) (figure 1).

Scheme I[a]



[a] Conditions: (a) $3P(NEt_2)_3$, $-4Et_2NH$; (b) 1/4 S_8/toluene; (c) H_2O/CH_2Cl_2; (d) 2HCl (anhydrous), $-Et_2NH_2Cl$/toluene.

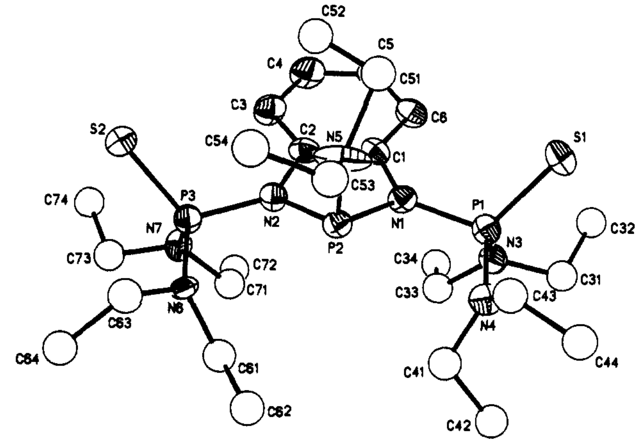


Figure 1. Structure and numbering scheme for $C_6H_4N_2[P(S)(NEt_2)_2]_2PNEt_2$ (**2**) showing 50% probability thermal ellipsoids. Hydrogen atoms omitted for clarity. Selected bond distances (Å) and angles (deg): P(1)–N(1), 1.672 (5); P(2)–N(1), 1.744 (5); P(2)–N(2), 1.762 (5); P(3)–N(2), 1.664 (5); P(1)–N(3), 1.645 (5); P(1)–N(4), 1.647 (5); P(1)–S(1), 1.940 (2); P(2)–N(5), 1.633 (6); P(3)–N(6), 1.649 (5); P(3)–N(7), 1.639 (5); P(3)–S(2), 1.936 (2); C(1)–N(1), 1.427 (7); C(2)–N(2), 1.426 (7); P(2)–N(1)–C(1), 112.6 (4); N(1)–P(2)–N(2), 87.5 (2); P(2)–N(2)–C(2), 111.5 (4); N(1)–C(1)–C(2), 110.9 (5); N(2)–C(2)–C(1), 112.0 (5); C(2)–N(2)–P(3), 125.3 (4); P(2)–N(2)–P(3), 123.0 (3); P(1)–N(1)–C(1), 125.8 (4); P(1)–N(1)–P(2), 121.6 (3); N(2)–P(2)–N(5), 107.8 (3); N(1)–P(2)–N(5), 103.8 (3); S(1)–P(1)–N(1), 114.8 (2); S(2)–P(3)–N(2), 114.7 (2).

Triphosphazane **1**[3,4] and the bis(sulfide) **2**[3,5] have been characterized by a combination of ^1H and ^{31}P NMR, IR, and mass spectral data. Both **1** and **2** contain symmetrically oriented triphosphazane units in solution, since both exhibit symmetrical AX_2 ^{31}P NMR spectral patterns. The $^2J_{PP}$ coupling constant in trans P–N–P diphosphazane conformations are known to be relatively small, 18–25 Hz, whereas for cis conformations they are larger, typically 200–732 Hz.[6] Since $^2J_{PP}$ for **1** varies only from 29.4 to 53.0 Hz over the temperature range −90 to +104 °C, it appears to be in or close to a trans–trans conformation in solution and is primarily in this conformation to 104 °C.

The structure of **2**, and by inference **1**, has been confirmed by a single-crystal X-ray analysis.[7] Crystals of **2** contain eight

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(1) (a) Allcock, H. R.; Tollefson, N. M.; Arcus, R. A.; Whittle, R. R. *J. Am. Chem. Soc.* **1985**, *107*, 5166. (b) Allcock, H. R. *Chem. Eng. News* **1985**, *63*, 22. (c) Allcock, H. R. In *Rings, Clusters, and Polymers of the Main Group Elements*; Cowley, A. H., Ed.; ACS Symposium Series 232; American Chemical Society: Washington, DC, 1982; p 49.

(2) (a) Shaw, R. A. *Phosphorus Sulfur* **1978**, *4*, 101. (b) Keat, R. *Top. Curr. Chem.* **1983**, *102*, 89. (c) Thompson, M. L.; Tarassoli, A.; Haltiwanger, R. C.; Norman, A. D. *J. Am. Chem. Soc.* **1981**, *103*, 6770. (d) Niecke, E.; Ruger, R.; Schoeller, W. W. *Angew Chem., Int. Ed. Engl.* **1981**, *20*, 1034. (e) Zeiss, W.; Barlos, K. *Z. Naturforsch., B* **1979**, *34B*, 423. (f) Chen, H.-J.; Hartiwanger, R. C.; Hill, T. G.; Thompson, M. L.; Coons, D. E.; Norman, A. D. *Inorg. Chem.*, in press. (g) Thompson, M. L.; Haltiwanger, R. C.; Norman, A. D. *J. Chem. Soc., Chem. Commun.* **1979**, 647. (h) Zeiss, W.; Schwarz, W.; Hess, H. *Angew. Chem., Int. Ed. Engl.* **1977**, *16*, 407.

(3) Satisfactory elemental analyses were obtained for all compounds except **3**, which was thermally unstable and persistently contaminated by traces of thermolysis products.

(4) **1**: $^{31}P\{^1H\}$ NMR (C_6D_6) δ 106.5 (d, $^2J_{PP} = 43.5$ Hz, area 2), 100.5 (t, area 1); ^1H NMR (C_6D_6) δ 7.11–6.66 (complex, area 4, C_6H_4), 3.14–2.71 (complex, area 20, CH_2CH_3), 1.02 (complex, area 30, CH_2CH_3); MS, parent at m/e 557, $C_{26}H_{54}N_7P_3^+$.

(5) **2**; $^{31}P\{^1H\}$ NMR (C_6D_6) δ 98.3 (t, $^2J_{PP} = 63.9$ Hz, area 1), 65.1 (d, area 2); ^1H NMR (C_6D_6) δ 7.91 and 6.90 (multiplets, area, 4, C_6H_4), 3.62–2.75 (m, area 20, CH_2CH_3), 1.03 (m, area 30, CH_2CH_3); MS, parent at *m/e* 621, $C_{26}H_{54}N_7P_3S_2^+$; IR (KBr), characteristic absorption at 606 cm^{-1} (P=S).

(6) Keat, R.; Manojlovic-Muir, L.; Muir, K. W.; Rycroft, D. S. *J. Chem. Soc., Dalton Trans.* **1981**, 2192.

(7) Crystal data for **2**: $C_{26}H_{54}N_7P_3S_2$, formula weight 621.81, orthorhombic, *Pbca*, $a = 14.652$ (5) Å, $b = 18.911$ (9) Å, $c = 24.324$ (9) Å, $V = 6739$ (4) $Å^3$, $Z = 8$, $D_{obsd} = 1.25$ g/cm, $D_{calcd} = 1.23$ g/cm. Intensity data: Nicolet P3/F autodiffractometer equipped with low-temperature apparatus, data collected at −80 °C, Mo K_α ($\lambda = 0.71069$ Å), Wycoff ω scan mode, $3.0 < 2\theta < 45.0$, 5029 reflections collected, of which 2754 were observed $[(F_o) > 6\sigma(F_o)]$. The structure was solved by direct methods and refined by block cascade least-squares calculations; $R = 0.060$, $R_w = 0.075$. Hydrogen atoms were included in idealized geometries, riding on the atoms to which they are attached, with a single refined thermal parameter.

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