AD-A150 595 ORGANOPHOSPHAZENES 18 FRIEDEL-CRAFTS PHENYLATION REACTIONS OF ALKYL AND N.. (U) VERMONT UNIV BURLINGTON DEPT OF CHEMISTRY C W ALLEN ET AL. 15 NOV 84 TR-16 1/1

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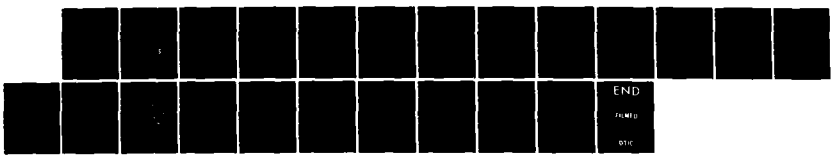

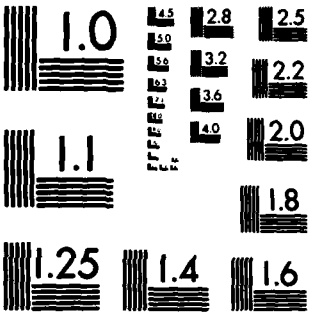

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The monodimethylamino and monobutylpentafluorocyclotriphosphazenes, $N_3P_3F_5R$(R = $N(CH_3)_2$, n-C_4H_9, t-C_4H_9) undergo the Friedel-Crafts phenylation reaction to yield $N_3P_3F_4(C_6H_5)R$. A geminal configuration was assigned to each of the phenyl derivatives based on the NMR (^1H, ^{13}C, ^{31}P) spectroscopic data. The crystal and molecular structures of $N_3P_3F_4(C_6H_5)$(t-C_4H_9) have been determined; the crystals are orthorhombic, space group P_{nma}, with a = 7.264(1)Å,

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20. Abstract (Continued)

b = 12.204(2)Å, c = 17.028(3)Å, V = 1509.5(7)Å^3, and Z = 4. The final refinement gave R = 0.042 and R_w = 0.054 for 1087 observed reflections. The molecule, which has a crystallographically-imposed mirror plane of symmetry, has the predicted geminal arrangement of organic groups and P-N distances of 1.618(1), 1.527(2), and 1.565(1)Å. The central P-N ring is planar within 0.007Å. Bond angles are: C-P-C, 108.9(1)°; F-P-F, 96.36(9)°; N-P-N, 114.1(1); 120.91(9)°; P-N-P, 122.58(9); 118.9(1)°. The observation of Friedel-Crafts phenylation of the alkylphosphazenes demonstrates that π donation from an exocyclic substituent is not a necessary prerequisite for this reaction.

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by

Christopher W. Allen, Scott Bedell, William T. Pennington and A. Wallace Condes

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University of Vermont
Department of Chemistry
Burlington, Vermont 05405

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Organophosphazenes. 18. Friedel-Crafts Phenylation Reactions of Alkyl and Dimethylamino Fluorocyclotriphosphazenes.[1]

Christopher W. Allen*[2a], Scott Bedell[2a], William T. Pennington[2b] and A. Wallace Cordes[2b]

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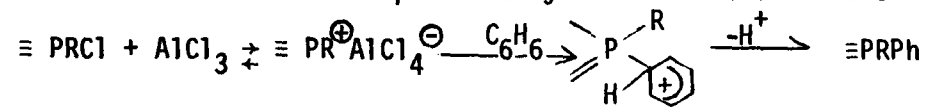
Abstract

The monodimethylamino and monobutylpentafluorocyclotriphosphazenes, $N_3P_3F_5R$(R = $N(CH_3)_2$, n-C_4H_9, t-C_4H_9) undergo the Friedel-Crafts phenylation reaction to yield $N_3P_3F_4(C_6H_5)R$. A geminal configuration was assigned to each of the phenyl derivatives based on the NMR (^1H, ^{13}C, ^{31}P) spectroscopic data. The crystal and molecular structures of $N_3P_3F_4(C_6H_5)$(t-C_4H_9) have been determined; the crystals are orthorhombic, space group P_{nma}, with a = 7.264(1)Å, b = 12.204(2)Å, c = 17.028(3)Å, V = 1509.5(7)Å^3, and Z = 4. The final refinement gave R = 0.042 and R_w = 0.054 for 1087 observed reflections. The molecule, which has a crystallographically-imposed mirror plane of symmetry, has the predicted geminal arrangement of organic groups and P-N distances of 1.618(1), 1.527(2), and 1.565(1)Å. The central P-N ring is planar within 0.007Å. Bond angles are: C-P-C, 108.9(1)°; F-P-F, 96.36(9)°; N-P-N, 114.1(1); 120.91(9)°; P-N-P, 122.58(9); 118.9(1)°. The observation of Friedel-Crafts phenylation of the alkylphosphazenes demonstrates that π donation from an exocyclic substituent is not a necessary prerequisite for this reaction.

Introduction

The Friedel-Crafts arylation is a classic reaction[3] in phosphazene chemistry dating back to the pioneering studies of Bode and Bach.[4] This reaction can be used to produce geminal aryl groups in chloro[4,5] and fluorocyclophosphazenes.[6] The observation that the reaction usually yields geminal di,tetra and hexaaryl phosphazenes but not mono, tri or penta substituted derivatives leads to the suggestion that a ≡ PPhCl center is more reactive than a ≡ PCl_2 center. Aminochlorophosphazenes also undergo the Friedel-Crafts arylation reaction which allows for conversion of a ≡ $PClNR_2$ center to a ≡ $P(Ph)NR_2$ center.[7] Depending on the nature of the amine, side reactions may also become significant. The phosphazo substituent also promotes Friedel-Crafts arylation at a ≡ $PCl(N{=}PPh_3)$ center.[8]

In spite of the synthetic utility of this reaction, little is known about the mechanism. The first step of the reaction is often assumed to be the formation of a phosphonium ion arising from halide abstraction by the Lewis acid. Definitive evidence on this point has yet to become available. The



addition compounds of aluminum tribromide with halocyclotriphosphazenes, $N_3P_3X_6.nAlBr_3$ (X - Cl, n = 1; X = Br, n = 1,2) are believed to have the aluminum tribromide entity coordinated to an endocyclic nitrogen atom.[9] Related addition compounds of $SbCl_5$ and $TiCl_4$ apparently involve the phosphonium ion.[10] Once the phosphonium ion forms attack on the solvent (benzene) could lead to a cationic sigma complex and deprotonation to the observed arylphosphazene. The assumption is implicity or explicitly made that the facility of the Friedel-Crafts reaction at a ≡PXR (R = aryl, amino; X = F, Cl) center is due to stabilization of the positive charge (or

developing positive charge) on the phosphorus atom by the electron donating substituent, R.

In this paper, we wish to explore the nature of some of the electronic factors involved in the proposed stabilization of the positive charge on the phosphorus center and expand the range of systems which undergo the Friedel-Crafts arylation reactions.

Experimental Section

Materials and Methods. Hexachorocyclotriphosphazene, $N_3P_3Cl_6$ (Firestone Corp.), was converted to hexafluorocyclotriphosphazene[11], which was in turn converted to (dimethylamino)pentafluorocyclotriphosphazene, $N_3P_3F_5N(CH_3)_2$[12], (n-butyl)pentafluorocyclotriphosphazene, $N_3P_3F_5CH_2(CH_2)_2CH_3$[13], and (t-butyl) pentafluorocyclotriphosphazene, $N_3P_3F_5C(CH_3)_3$[13], by previously reported procedures. Diethylether was distilled from sodium/benzophone. Benzene[14] was distilled from sodium. Other reagents and solvents were obtained from standard sources and used without further purification. Nmr spectra (in $CDCl_3$) were reported on a Bruker WM250 spectrometer operating at 250.1 (^1H), 62.9(^{13}C), 235.2(^{19}F), and 101.2 MHz (^{31}P). Tetramethylsilane, Me_4Si (for ^1H and ^{13}C), and fluorotrichloromethane, $CFCl_3$ (for ^{19}F), were used as internal references. For ^{31}P nmr, 85% H_3PO_4 was used as an external reference. Chemical shifts upfield to the reference are assigned a negative sign. ^{13}C, ^{19}F, and ^{31}P nmr spectra were recorded under conditions of broad-band decoupling. Mass spectra were recorded on a Finnegan 4610 mass spectrometer operating at 80eV. Elemental analyses were performed by Integral Microanalytical Laboratories.

Preparations of $N_3P_3F_4[N(CH_3)_2]C_6H_5$ (1). A solution of 2.65g (.01 mol) of $N_3P_3F_5N(CH_3)_2$ in 12 mL of benzene was added to a solution of triethylamine (2.8g, 0.03 mol) and anhydrous aluminum chloride (9.3g,0.07 mol) in 50 mL of benzene.[14]. The reaction mixture was allowed to stir at reflux for five days. The mixture was hydrolyzed over acidified ice water and the layers were separated. The benzene layer was washed sequentially with saturated sodium bicarbonate solution and distilled water then dried over anhydous sodium

sulfate and decolorized. After removal of the solvent, the yellow liquid was distilled at reduced pressure (0.02 mmHg) to yield 1.0g (31.3% of theory) of a colorless oil, bp 60°C (.02 mmHg). Anal. Calcd. for $N_4P_3F_4C_8H_{11}$: C, 28.92; H, 3.31; mol wt 332. Found: C, 28.93; H, 3.49; mol wt 332 (mass spectrum[15]). ^{1}H NMR: [16]$\delta_{N(CH_3)_2}$ = 2.6 (d, 6H), $^3J_{PH}$ = 12.9; $\delta_{C_6H_5\text{-m,p}}$ = 7.5 (m, 3H); $\delta_{C_6H_5\text{-o}}$ = 7.8 (q, 2H), $^3J_{PH}$ = 13.8 ^{13}C NMR: $\delta_{N(CH_3)_2}$ = 35.6 (d), $^2J_{PC}$ = 3.5; δ_{ArC_2} = 131.2 (d); $^2J_{PC}$ = 10.7; δ_{ArC_3} = 128.8 (d), $^3J_{PC}$ = 14.9; δ_{ArC_4} = 132.7 (d), $^4J_{PC}$ = 3.1. ^{31}P NMR: $\delta_{\equiv PF_2}$ = 10.2 (m, 2P), $^1J_{PF}$ = 916.0[17]; $\delta_{\equiv PN(CH_3)_2C_6H_5}$ = 29.5 (m, 1P), $^2J_{pp}$ = 63.7.

Preparation of $N_3P_3F_4[CH_2(CH_2)_2CH_3]C_6H_5$ (2). The procedure was identical with that described for the preparation of 1 except that $N_3P_3F_5CH_2(CH_2)_2CH_3$ was used in place of $N_3P_3F_5N(CH_3)_2$. In a typical experiment, the following quantities were allowed to react for five days: $N_3P_3F_5CH_2(CH_2)_2CH_3$ (2.00g, 0.007 mol), triethylamine (2.25g, .02 mol) and aluminum chloride (7.45g, .06 mol). Distillation yielded a colorless oil whose ^{31}P nmr spectrum consisting of largely the unreacted starting material, small amounts of $N_3P_3F_4[CH_2(CH_2)_2CH_3]C_6H_5$ (less than 10%) and traces of other unidentified materials. Careful fractionation (35° at 0.02 mmHg) yielded an impure sample of the desired product. Anal. Calcd. for $N_3P_3F_4C_{10}H_{14}$: C, 34.78; H, 4.06; mol wt 345. Found: C, 29.43; H, 4.50; mol wt 345 (mass spectrum[15]). ^{1}H NMR: [16]$\delta_{\underline{CH_2}(CH_2)_2CH_3}$ = 1.9 (m, 2H); $\delta_{CH_2\underline{CH_2}\underline{CH_2}CH_3}$ = 1.5 (m, 4H); $\delta_{(CH_2)_3\underline{CH_3}}$ = 0.9 (m, 3H); $\delta_{C_6H_5\text{-m,p}}$ = 7.5 (m, 3H); $\delta_{C_6H_5\text{-o}}$ = 7.8 (q, 2H), $^3J_{PH}$ = 13.3. ^{13}C NMR: $\delta_{C\alpha}$ = 33.6 (m), $^1J_{PC}$ = 98.1; $\delta_{C_{\beta,\gamma}}$ = 23.2 (m, 2C), δ_{C_Δ} = 13.5 (s); δ_{ArC_2} = 130.0 (d), $^2J_{PC}$ = 11.0; δ_{ArC_3} = 128.8 (d), $^3J_{PC}$ = 12.9; δ_{ArC_4} = 132.6 (d), $^4J_{PC}$ = 3.4. ^{31}P NMR (approximate): $\delta_{\equiv PF_2}$ = 7.1, $^1J_{PF}$ = 883[17]; $\delta_{\equiv P(C_4H_9)C_6H_5}$ = 37.8, $^2J_{PP}$ = 36.

Preparation of $N_3P_3F_4[C(CH_3)_3]C_6H_5$ (3). The procedure was identical with that described above except that $N_3P_3F_5C(CH_3)_3$ was used as the phosphazene starting material. In a typical experiment, the following quantities were allowed to react for five days: $N_3P_3F_5C(CH_3)_2$ (3.80g, 0.013 mol), triethylamine (4.10g, 0.04 mol), and aluminum chloride

(13.5g, 0.10 mol). The yellow solid remaining after removal of the benzene was sublimed to yield 1.78g (39.0% of theory) of a milk-white solid, mp 102-103°. Anal. Calcd. for $N_3P_3F_4C_{10}H_{14}$: C, 34.78; H, 4.06; mol wt 345. Found: C, 35.05; H, 3.68; mol wt 345 (mass spectrum[15]).

^1H NMR[16]: $\delta_{C(CH_3)_3}$ 1.1 (d, 9H), $^3J_{PH}$ = 17.7; $\delta_{C_6H_5\text{-}m,p}$ = 7.5 (m, 3H); $\delta_{C_6H_5\text{-}o}$ = 7.8 (q, 2H), $^3J_{PH}$ = 12.0. ^{13}CNMR: $\delta_{C(CH_3)}$ = 33.9 (m); δ_{ArC_2} = 131.9 (d), $^2J_{PC}$ = 10.1; δ_{ArC_3} = 128.2 (d), $^3J_{PC}$ = 12.9; δ_{ArC_4} = 132.5 (d), $^4J_{PC}$ = 3.4. ^{31}P NMR: $\delta_{\equiv PF_2}$ = 9.2, $^1J_{PF}$ = 885.1[17]: $\delta_{\equiv PPhC(CH_3)_3}$ = 46.1, $^3J_{PP}$ = 36.1.

Attempted Friedel-Crafts reaction of $N_3P_3F_5OCH_2CH_3$. The procedure was identical with that described above except that $N_3P_3F_5OCH_2CH_3$ (prepared from $N_3P_3F_6$ and $LiOCH_2CH_3$) was used as the phosphazene starting material. No phosphazene containing products were recovered from the benzene layer after hydrolysis.

X-ray Analysis of $N_3P_3F_4(C_6H_5)(t\text{-}C_4H_9)$. Crystals of 3 suitable for x-ray work were obtained by recrystallization from a petroleum ether-CH_2Cl_2 mixture. A colorless crystal of approximate dimensions 0.30 X 0.30 X 0.35 mm, mounted on a glass fiber, was used for data collection on an Enraf-Nonius CAD-4 diffractometer employing graphite-monochromated Mo Kα (γ =0.71073 Å) radiation. A least-squares fit of the diffractometer setting angles for 25 carefully centered reflections (24° < 2θ < 37°) gave the unit cell parameters listed in Table I.

Systematic absences of 0kl (k+l, odd) and hk0 (h odd) indicate the space group $Pna2_1$ (No. 33) or Pnma (No. 62); the centrosymmetric space group Pnma was verified by the refinement. Reflections were measured using ω-2θ scans for 2θ from 2 to 50° (h = 0 to 8, k = 14 to 0, l = 0 to 20). The scan range was (1.00 + 0.35 tan θ)° and the scan speed varied from 20 to 4°/min. Of the 1529 reflections measured, 1087 had $I > 3\sigma(I)$ and were used in the refinement. Data were corrected for Lorentz and polarization effects, but due to the low linear absorption coefficient and regular shape of the crystal an absorption correction was not made. Periodic measurement of the intensities of three reflections (2 9 0, 4 0 4, 0 5 11) indicated a 28% decline during the 13.2 hours of data collection, and intensities were

corrected accordingly.

The structure was solved by direct methods (MULTAN80)[18] and refined by full-matrix least-squares techniques. The molecule has a crystallographically imposed mirror symmetry. Hydrogen atoms were located using difference Fourier techniques and were included in the refinement. The final cycle of refinement based on $(|F_o|-|F_o|)^2$ included positional and anisotropic thermal parameters for all non-hydrogen atoms and positional parameters for the hydrogen atoms. The isotropic thermal parameters of each hydrogen atom was constrained at a value 20% greater than the B_{eq} of the carbon atom to which it was attached. The final R[19] = 0.042 and R_w[20] = 0.054. The weighting scheme used was $w = [\sigma^2(F) + 0.03F^2]^{-1}$ where $\sigma(F)$ was derived from counting statistics. The goodness of fit[21] was 2.36. In the final cycle of refinement the maximum shift/error was 0.09. The final difference map has a maximum value of 0.36 $e^-/Å^3$. No secondary extinction correction was made. The atomic scattering factors for neutral atoms were those of Cromer and Waber[22] and the real and imaginary dispersion corrections were those of Cromer.[23] The computer programs used were those provided by the Enraf-Nonius Structure Determination Package.

Results and Discussion

The synthetic results show that a fluorine atom in both monoalkyl and mono(N,N-dimethylamino)pentafluorocyclotriphosphazenes can be conveniently replaced by a phenyl group under Friedel-Crafts conditions. Although dialkylaminochlorophosphazenes have previously been shown to undergo Friedel-Crafts phenylation[7], we report the first example of the corresponding reaction in the fluorophosphazene series and the first example of Friedel-Crafts phenylation of an alkylphosphazene. The new compounds (1, 2, 3) have all been characterized by elemental analyses, mass spectrometry and nmr (^1H, ^{13}C, ^{31}P) spectroscopy. Observations of the stereochemical course of the Friedel-Crafts phenylation of other cyclophosphazenes[4-7] leads to the suggestion of a geminal substitution pattern being followed in the synthesis of 1, 2 and 3. The observed values of the $^2J_{PCC}$[24] and $^3J_{PCCH}$[25] coupling constants in 1, 2 and 3 are consistent

with geminal rather than non-geminal configurations. The ^{31}P nmr spectra of each of the new compounds shows a large, complex, triplet ($J_{PF} \sim$ 883 - 916 Hz) due to the $\equiv PF_2$ centers and a small ($J_{PP} \sim$ 36 Hz) triplet due to the $\equiv PR(Ph)$ center. The lack of a doublet due to a $\equiv PFR$ (or $\equiv PFPh$) center confirms the absence of non-geminal products.

The crystal and molecular structures of 3 were determined in order to verify the proposed geminal configuration and to explore the structural consequences of the sterically crowded environment arising from substitution of both a phenyl and tert-butyl group on the same phosphorus atom. The final atomic coordinates of all unique atoms are given in Table II. Selected bond lengths and bond angles may be found in Table III and an ORTEP drawing of the molecule, together with the atom numbering scheme, is shown in Figure 1. The observed molecular structure of 3 confirms the geminal configuration of organic functions on the phosphazene. The solid state structure of 3 is such that the phosphazene ring is bisected by a mirror plane containing N_1, P_1, C_1, C_4, H_4, C_5, C_7 and H_{71}. The conformations adopted by the phenyl and tert-butyl groups lead to the minimum amount of steric interaction between the two groups. A comparison of the structure of 3 with the structure of the previously reported geminal diphenyltetrafluorocyclotriphosphazene, 2,2-$N_3P_3F_4(C_6H_5)_2$ (4)[26] is of interest. In 4 five of the six phosphazene ring atoms are close to coplanarity with the phenyl substituted phosphorus atom 0.20Å out of this plane. In 3 the central ring is more nearly planar (within 0.007Å): the P_3N_2 segment which excludes the unique nitrogen atom is planar within 0.001Å and the N 1 atom is displaced only 0.014(3)Å from this plane. In comparing 3 to 4 a small effect of the increased steric demand of the tert-butyl group over the phenyl group is seen in the bond angles at the organosubstituted phosphorus atom (<CPC = 108.9(1) for 3 and 107.9(3) for 4; <NPN = 114.1(1) for 3 and

115.3 (3) for 4). The phosphorus-nitrogen bond lengths are virtually identical in 3 and 4 and vary in a manner which is typical of assymetrically substituted phosphazenes and can be correlated with the relative ability of each phosphorus center to attract nitrogen lone pair electron density.[26] The small differences in P-N distances of P2 are consistent with the effect of replacing an sp^2 carbon atom (in 4) by a sp^3 carbon atom (in 3) resulting in a decrease of electron attracting power of the organosubstituted phosphorus atom. Consequently, the lone pair of electrons at N(2) is more strongly attracted to P(2) in 3 than in 4. The increased charge density on P(2) in 3 results in less charge delocalization from N(1) and hence a longer P(2)N(1) distance in 3 vs 4 (1.565(1) vs 1.555(4)).

One of the goals of this study was to probe substituent electronic effects which may be operative in the Friedel-Crafts phenylation reaction. Since previous investigations have employed exocyclic groups which are potential π donors to the phosphorus center, monoalkylpentafluorocyclotriphosphazenes were examined in order to clarify differences between σ and π donor substituent effects. The fact that the alkylphosphazenes undergo the phenylation reaction unambiguously demonstrates that π donation from the exocyclic function is not a necessary prerequisite for this reaction to be effective. The significantly higher yield in the reaction of tert-butyl versus the n-butyl phosphazene suggests the operation of a mechanism wherein the phosphorus atom undergoing substitution goes to a lower coordinate intermediate (or transition state). This process would be most favorable for the tert-butyl derivative where the maximum relief of steric strain can be obtained. A reasonable model for the Friedel-Crafts phenylation reaction of substituted phosphazenes is one in which the substituted phosphorus atom goes through a three coordinate phosphorus (v) intermediate which is stabilized by the σ electron releasing nature of the substituent. It is possible that in the phenyl and dialkylamino systems, the σ electron releasing effect may be supplemented by a π effect but conclusive evidence of this has yet to be presented. The reluctance of the

monoethoxypentafluorocyclotriphosphazene to enter into the Friedel-Crafts reaction is consistent with these proposals since the high electronegativity of the oxygen atom makes it a poor electron releasing substituent and the small size does not allow for significant relief of steric strain on going to a lower coordinate transition state intermediate.

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Supplementary Material Available. Table 5.1. showing major mass spectral fragments and their intensities (pages), Table 5.2 showing structure factor amplitudes for $N_3P_3F_4(C_6H_5)(C_4H_9)$, and Table 5.3 showing anisotropic thermal parameters for $N_3P_3F_4(C_6H_5)(C_4H_9)$. Ordering information is given on any current masthead page.

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13. Ramachandran, K.; Allen, C. W. J. Am. Chem. Soc. 1982, 104, 2396.
14. Benzene is a known carcinogen and hence should be only used in a well ventilated hood while wearing appropriate protective gloves.

15. Mass spectral data are available as supplementary material.

16. All nmr (^1H, ^{31}P) chemical shifts in ppm and coupling constants in Hz.

17. Due to considerable second order character, the $^1J_{PF}$ values are only approximate.

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19. $R = \Sigma(|F_o| - |F_c|)/\Sigma|F_o|$

20. $R_w = [\Sigma w(|F_o| - |F_c|)^2/\Sigma w|F_o|^2]^{1/2}$

21. $G.O.F. = [\Sigma w(|F_o| - |F_c|)^2/N_o - N_v)]^{1/2}$, where N_o = no. of observations, and N_v = no. of variables.

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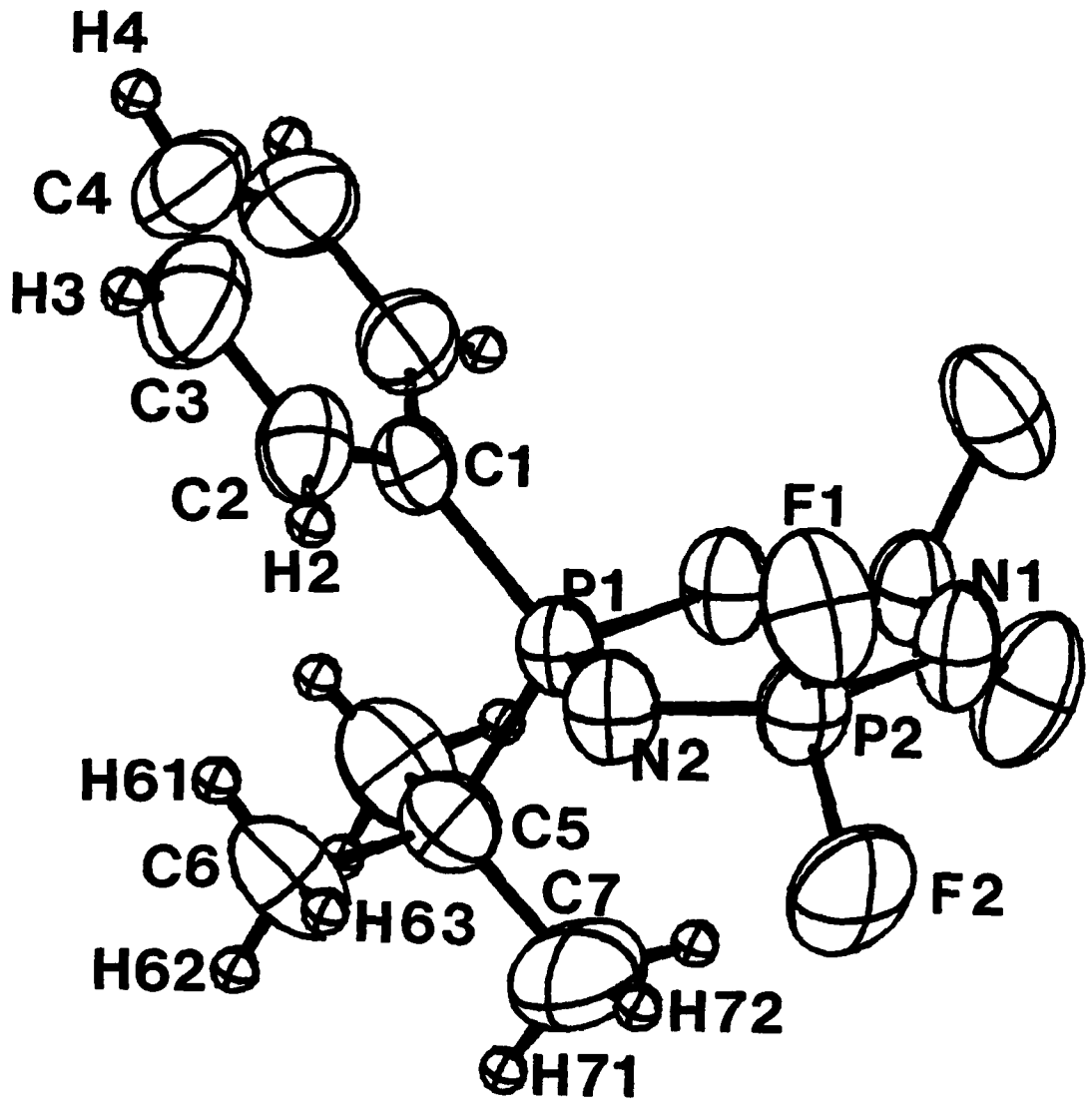
H4
C4
H3
C3
C2
H2
C1
F1
N1
P1
N2
P2
H61
C5
C6
C7
F2
H63
H62
H72
H71

Figure 1. ORTEP drawing (50% probability elipsoids) of 2,2-$N_3P_3F_4(C_6H_5)(C_4H_9)$ showing the atom numbering scheme.

Table I. Crystallographic Data

formula	$C_{10}H_{14}F_4N_3P_3$
fw	345.16
cryst. syst.	orthorhombic
space group; molecules/cell	Pnma; 4
a, Å	7.264 (1)
b, Å	12.204 (2)
c, Å	17.028 (3)
V, $Å^3$	1509.5 (7)
reflcns used for unit cell; 2θ range, deg	25; 24 - 37
calcd density, g cm^{-3}	1.52
F(000)	704
reflcns scanned; obsd	1529; 1087
2θ range of reflcns scanned, deg	2 - 50
μ (Mo Kα), cm^{-1}	4.2
final R; R_w	0.042; 0.054
max shift in final cycle (σ)	0.09
max peak (final diff map), $eÅ^{-3}$	0.36
goodness of fit	2.36
parameters refined; parameter/reflcn ratio	122; 8.9

Table II. Positional Parameters

Atom	x/a	y/b	z/c	Beq
P1	0.9951(1)	0.250	0.52490(5)	3.57(2)
P2	0.8471(1)	0.13958(6)	0.65138(4)	4.48(2)
F1	0.6759(3)	0.0679(2)	0.6496(1)	7.04(4)
F2	0.9549(3)	0.0696(2)	0.7091(1)	7.84(5)
N1	0.7965(5)	0.250	0.6928(2)	5.21(8)
N2	0.9409(3)	0.1388(2)	0.5711(1)	4.32(5)
C1	0.8770(5)	0.250	0.4331(2)	3.80(8)
C2	0.8298(4)	0.1522(2)	0.3968(2)	5.11(7)
C3	0.7363(5)	0.1539(3)	0.3267(2)	7.05(9)
C4	0.6923(7)	0.250	0.2920(3)	7.4(1)
C5	1.2400(5)	0.250	0.5068(2)	4.75(9)
C6	1.2928(5)	0.1476(3)	0.4606(2)	6.62(8)
C7	1.3385(7)	0.250	0.5869(3)	9.6(2)
H2	0.854(4)	0.085(2)	0.421(2)	6*
H3	0.717(4)	0.103(3)	0.304(2)	8*
H4	0.624(8)	0.250	0.254(3)	11*
H61	1.233(4)	0.156(3)	0.412(2)	7*
H62	1.422(5)	0.150(3)	0.452(2)	7*
H63	1.251(4)	0.079(3)	0.491(2)	7*
H71	1.480(8)	0.250	0.571(4)	11*
H72	1.294(5)	0.194(3)	0.625(2)	11*

Starred atoms were refined isotropically.

Table III. Selected distances (Å) and Angles (deg) for $N_3P_3F_4(C_6H_5)(C_4H_9)$.[a]

P1 - N2	1.618(1)	N2 - P1 - N2	114.1(1)
P1 - C1	1.784(3)	N2 - P1 - C1	108.04(7)
P1 - C5	1.806(3)	N2 - P1 - C5	108.82(8)
P2 - F1	1.521(1)	C1 - P1 - C5	108.9(1)
P2 - F2	1.520(1)	F1 - P2 - F2	96.36(9)
P2 - N1	1.565(1)	F1 - P2 - N1	108.2(1)
P2 - N2	1.527(2)	F1 - P2 - N2	110.06(8)
C1 - C2	1.387(2)	F2 - P2 - N1	108.3(1)
C2 - C3	1.374(3)	F2 - P2 - N2	110.21(8)
C3 - C4	1.351(4)	N1 - P2 - N2	120.91(9)
C5 - C6	1.526(3)	P2 - N1 - P2	118.9(1)
C5 - C7	1.540(5)	P1 - N2 - P2	122.58(9)
C-H (ave)	0.93(9)	P1 - C1 - C2	120.6(1)
		P1 - C5 - C6	109.6(2)
		P1 - C5 - C7	107.9(2)

[a]Numbers in parentheses are estimated standard deviations in the least significant digits.

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