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Reactions of tetracyclic tetraaminophosphoranes with dichloroperfluoro cyclic and acyclic alkenes and halo compounds

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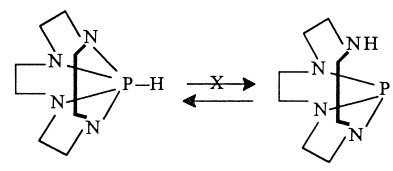
Abstract

Cyclenphosphorane was reacted with 1,2-dichlorotetrafluorocyclobutene-1, 1,2-dichlorohexafluorocyclopentene-1, 1,2-dichlorooctafluorocyclohexene-1, iodopentafluorobenzene, 2-iodo-1,1,1-trifluoroethane, and 2,3-dichlorohexafluorobutene-2 in the presence of triethylamine at room temperature in tetrahydrofuran to form monochlorotetrafluorocyclobutenylcyclenphosphorane, monochlorohexafluorocyclopentenylcyclenphosphorane, monochlorooctafluorocyclohexenylcyclenphosphorane, pentafluorophenylcyclenphosphorane, 1,1,1-trifluoroethylcyclenphosphorane, and 2-chlorohexafluorobut-2-enylcyclenphosphorane in 70–80% yields, respectively. Cyclamphosphine oxide reacted with dichloroperfluorocyclic alkenes in the presence of triethylamine in chloroform to form N-(chlorotetrafluorocyclobutenyl)cyclamphosphine oxide, N-(chlorohexafluorocyclo-pentyl)cyclamphosphine oxide and N-(chlorooctafluorocyclohexenyl)-cyclamphosphine oxide in ~50% yields, respectively. These moisture sensitive cyclamphosphine oxide derivatives are stable and are soluble in $CHCl_3$, CH_3CN and DMSO.

1. Introduction

Molecules that contain pentacoordinate phosphorus bonded to cyclic tetraamino moieties were first reported in 1977 [1–3], followed somewhat later by methoxy, thiomethoxy [4] and cyclenphosphonium salts [5]. The reactivity of these molecules should exhibit novel patterns because of (1) the presence of lone pairs on the nitrogen atoms, (2) the likelihood of the presence of a labile P–X bond (and potentially labile P–N bonds), and (3) the fact that an equilibrium can exist in solution between the phosphorane and phosphine forms when at least two of the N–N links are propyl or higher. This behavior was attributed to bonding configuration changes due to ring size [1]. Each of these patterns has been observed. CyclenPH (**2A**), when irradiated, gives the tetra-coordinate phosphoranyl radical, cyclenP● ([6–9]. The lithiated form of **2A** with cyclenPF yields cyclenP-Pcyclen [10,11]. **2A** reacts with diborane to afford cyclenphosphorane-bis(borane) which suggests unusually strong basicity of the P-bonded apical nitrogen atoms in the polycyclic structure [12–17]. In the case of **2A** only the phosphorane tautomer is observed [12]. This is in contrast to the bis(borane) adducts formed by cyclamphosphorane (**2B**) where both tautomers, phosphorane and phosphine, are formed. It appears that the initial equilibria observed by Atkins and Richman [2,3] play an important role in determining the chemistry of the cyclic tetraaminophosphoranes but subsequent reaction conditions can override the equilibria that are a function of ring size. However, unlike the larger macrocyclicamine phosphoranes, **2A** exists only as the closed tautomer in solution as well as in solid and gas phases.



2A **2A$_1$**

Attempts to isolate the open form **2A$_1$** by coordination to transition metals have thus far been unsuccessful except in rare cases where a bidentate structure was formed. Even with

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transition metals, reactions of (cyclen)PH usually gave pentacoordinate structures arising from the typical trigonal bipyramidal (tbp) geometry associated with phosphorus(V) [6–9,16,17]. Lattman et al. have reported the synthesis and X-ray crystal structure of the first monodentate P-bound transition metal complex of **2A$_1$** which revealed a P–N transannular interaction – a unique geometry for a phosphine ligand. This complex provided the first structural confirmation of the tbp-constraining 'bite' of the cyclen ring at phosphorus [18]. Although a few metal derivatives of **2A** were reported, the chemistry of cyclenphosphorane is not well explored. To the best of our knowledge, there are no literature reports that describe the chemistry of cyclenphosphorane with fluorine-containing compounds. In this paper, we report the first examples of derivatives of cyclenPH that contain fluorine and/or fluorine and chlorine substituents. We also report evidence for the open form of cyclenphosphorane (**2A$_1$**) in the presence of organic compounds. In our efforts to compare the reaction chemistry of **2A** with that of cyclamphosphorane (**2B**), we were only able to isolate products that were the result of reaction of the oxidized form of the phosphine tautomer. Ligand substitution occurs at nitrogen and not at phosphorus.

2. Experimental section

2.1. Materials

1,4,7,10-tetraazacyclododecane (cyclen) (**1A**), 1,4,8,11-tetraazacyclotetradecane (cyclam) (**1B**), cyclenphosphorane (**2A**), cyclamphosphorane (**2B**) and cyclamphosphine oxide (**2C**) were prepared either by the method of Richman [1] or obtained as a gift from Dr. J. E. Richman. 1,2-dichlorotetrafluorocyclobutene-1 (**3**), 1,2-dichlorohexafluorocyclopentene-1 (**4**), 1,2-dichlorooctafluorocyclohexene-1 (**5**), iodopentafluorobenzene (**6**), 2-iodo-1,1,1-trifluoroethane (**7**) and 2,3-dichlorohexafluorobutene-2 (**8**) were obtained from PCR Inc. and used as received. Triethylamine and THF were obtained from Aldrich and used after drying with 4 Å molecular sieve.

2.2. General procedure

A conventional Pyrex glass vacuum system equipped with a Heise–Bourdon tube gauge and a Televac thermocouple gauge was used to handle gases and volatile liquids. Volatile compounds were measured quantitatively by using PVT techniques. All reactions and manipulations were carried out in an atmosphere of prepurified dinitrogen or under vacuum unless otherwise indicated. Solvents were rigorously dried over appropriate drying agents and distilled and deoxygenated prior to use. Mass spectra were recorded with a VG -7070 HS mass spectrometer operating at 10 eV. Correct chlorine isotopic ratios were observed in each case. ^{19}F NMR spectra were obtained on a JEOL FX-90Q FT NMR spectrometer with $CFCl_3$ as internal reference. ^{1}H NMR spectra were run in 5 mm NMR tubes in $CDCl_3$ and the peak positions were measured relative to residual $CHCl_3$ and reported relative to Me_4Si. ^{31}P NMR spectra were obtained on this instrument at 36.2 MHz and phosphorus spectra were calibrated using external 85% phosphoric acid as the reference with an internal locking mode. Infrared spectra were obtained on a Perkin-Elmer 1700 FT-IR spectrometer using KBr discs for involatile liquids and KBr pellets for solids.

2.3. Method

A stoichiometric amount of the starting material (50 mg; 0.25 mmol) was dissolved in 10 ml of THF and was placed in a 100 ml round-bottomed flask equipped with a 10/14 ground glass standard taper joint and a stirring bar. A stoichiometric amount of triethylamine was added to the solution. An equivalent stoichiometric amount of haloalkenyl/halophenyl was weighed, dissolved in 2 ml of THF, added to the flask (for volatile materials vacuum line transfer was used) and the components were well mixed. The mixture was allowed to stir at 25°C for 24 h. The solid that separated was removed by filtration. The filtrate was concentrated. The residue was taken up in THF and 10% benzene (by volume) was added. After filtration, the filtrate was concentrated and the pure compound was obtained.

2.4. Synthesis

2.4.1. Cyclen(chlorotetrafluorocyclobutenyl)phosphorane (**A**)

The triethylammonium salt was removed by extraction with chloroform : hexane (30 : 70) solvent mixture to give a thick oily yellow compound in 30% yield. Spectral data obtained: ^{19}F NMR: $\delta = -110.69$ (2F), -113.77 (2F); ^{1}H NMR: $\delta = 2.8$–3.5 (m, 16H); ^{31}P NMR: $\delta = -46.28$. MS (CI^+): $m/z = 358$ (M^+). IR (KBr plates): 2942 (s), 2890 (s), 1684 (s) 1682 (ms), 1563 (w), 1545 (w), 1461 (s), 1362 (ms), 1290 (s), 1224 (s), 1080 (s) cm^{-1}.

2.4.2. Cyclen(chlorotetrafluorocyclopentenyl)phosphorane (**B**)

A thick oily compound was obtained in 90% yield. Spectral data obtained: ^{19}F NMR: $\delta = -108.10$ (2F), -113.39 (2F), -130.63 (2F); ^{1}H NMR: $\delta = 2.75$ (m, 16H); ^{31}P NMR: $\delta = -45.83$. MS (CI^+): $m/z = 408$ (M^+). HRMS(FAB^+) Calcd. for $C_{13}H_{16}N_4PF_6{\cdot}HCl$: 409.0785; Found: 409.0785. IR (KBr plates): 2921 (s), 2851 (s), 1772 (ms), 1577 (bs), 1337 (s), 1276 (ms), 875 (s) 648 (ms) cm^{-1}.

*2.4.3. Cyclen(chlorooctafluorocyclohexenyl)phosphorane (**C**).*

A liquid, yellow, thick, oily compound was obtained in 70% yield. Spectral data obtained: ^{19}F NMR: $\delta = -106.21$ (2F), -108.91 (2F), -133.42 (2F), -135.05 (2F); ^{1}H NMR: $\delta = 2.7$–3.0 (m, 16H); ^{31}P NMR: $\delta = -47.71$. MS (CI^+): $m/z = 458$ (M^+). IR (KBr plates): 2937 (vs), 2873 (vs), 1655 (bm), 1544 (s), 1466 (m), 1388 (m), 1249 (s), 1226 (m), 1190 (m), 1146 (m), 1128 (m), 1086 (s), 870 (w), 672 (ms) cm^{-1}.

*2.4.4. Cyclen(pentafluorophenyl)phosphorane (**D**)*

50 mg (0.25 mmol) cyclenphosphorane was taken in a 100 ml round bottomed Pyrex flask equipped with a 14/20 ground glass standard joint and magnetic stirring bar and dissolved in 2 ml dry chloroform. Iodopentafluorobenzene (74 mg, 0.25 mmol) was added and the mixture was stirred slowly for 12 h. A white solid was formed. It was separated by filtration and washed with 10 ml dry chloroform and dried under vacuum. **D** was obtained in 90% yield. Spectral data obtained: ^{19}F NMR: $\delta = -120.85$ (ortho, 2F), -153.39 (p, 1F), -159.57 (m, 2F); ^{1}H NMR: $\delta = 2.9$–3.1 (m, 16H); ^{31}P NMR: $\delta = -42.28$. MS (CI^+): $m/z = 366$ (M^+); HRMS(CI^+) Calcd. for $C_{14}H_{16}N_4PF_5$: 366.1032; Found: 366.1040. IR (KBr plates): 3019 (m), 2947 (s), 2879 (vs), 1685 (s), 1511 (m), 1482 (m), 1456 (s), 1392 (s), 1237 (vs), 968 (ms), 849 (s), 710 (s) cm^{-1}.

*2.4.5. Cyclen(trifluoroethyl)phosphorane (**E**)*

The oily, yellowish liquid was obtained in 60% yield. Spectral data obtained: ^{19}F NMR: $\delta = -58.32$ (3F); ^{1}H NMR: $\delta = 4.04$ (q, 2H), $\delta = 2.8$–3.2 (m, 16H); ^{31}P NMR: $\delta = -42.54$. MS (CI^+): $m/z = 283$ ($M^+ + 1$). IR (KBr plates): 2945 (s), 2911 (s), 1636 (w), 1588 (vs), 1490 (s), 1348 (m), 1300 (m), 1224 (s), 948 (m), 930 (m), 850 (bm), 730 (s), 672 (s) cm^{-1}.

*2.4.6. 2-Chlorohexafluorobut-2-enylcyclenphosphorane (**F**)*

The reaction was carried out on a 50 mg, (0.25 mmol) scale, using the stoichiometric ratio 1 : 1 of the reactants. The yellow, thick, oil was obtained in 20% yield. Spectral data obtained: ^{19}F NMR: $\delta = -61.53$ (m, 3F), -59.24 (m, 3F); ^{1}H NMR: $\delta = 2.8$–3.2 (m, 16H). ^{31}P NMR: $\delta = -45.83$. MS (CI^+); $m/z = 396$ (M^+). IR (KBr plates): 2937 (s), 2868 (vs), 1688 (m), 1664 (w), 1455 (ms), 1310 (s), 1244 (s), 1176 (vs), 1121 (vs), 1066 (m), 954 (ms), 910 (s), 765 (w), 721 (s), 644 (m) cm^{-1}.

*2.4.7. N-(chlorotetrafluorocyclobutenyl)cyclamphosphine oxide (**G**)*

The reaction was carried out on a 0.25 mmol scale. The salt that formed was separated, and the filtrate was concentrated to leave compound **G** as a thick oil in 70% yield. Spectral data obtained: ^{19}F NMR: $\delta = -109.52$ (2F), -111.76 (2F); ^{1}H NMR: $\delta = 2.8$–4.2 (bm, 16H), 1.9–2.2 (bm, 4H); ^{31}P NMR: $\delta = +28.04$. MS (CI^+): $m/z = 402$ (M^+), 383 ($M^+ - F$), 243 ($M^+ - C_4F_4Cl$). IR (KBr plates): 2922 (s), 2853 (s), 1658 (s), 1462 (s), 1411 (w), 1264 (s), 1180 (s), 978 (m), 866 (w), 687 (bm) cm^{-1}.

*2.4.8. N-(chlorohexafluorocyclopentenyl)cyclamphosphine oxide (**H**)*

The reaction was performed on a 0.25 mmol scale. The salt formed was separated, and the filtrate was concentrated to give compound **H** as a thick oil in 60% yield. Spectral data obtained: ^{19}F NMR: $\delta = -108.50$ (2F), -110.05 (2F), -127.28 (2F); ^{1}H NMR: $\delta = 1.65$ (m, 4H), 2.6–3.2 (m, 16H); ^{31}P NMR: $\delta = +30.98$. MS (CI^+): $m/z = 452$ (M^+), 433 ($M^+ - F$), 413 ($M^+ - 2HF_2$), 243 ($M^+ - C_5F_6Cl$). IR (KBr plates): 2959 (s), 2858 (s), 1630 (bs), 1587 (m), 1487 (w), 1473 (m), 1457 (m), 1391 (w), 1261 (s), 1171 (vs), 982 (s), 731 (m) cm^{-1}.

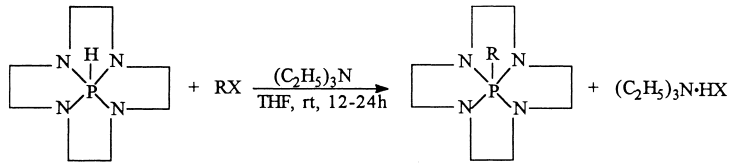
*2.4.9. N-(chlorooctafluorocyclohexenyl)cyclamphosphine oxide (**I**)*

Compound **I** was obtained in 50% yield as a thick oil. Spectral data obtained: ^{19}F NMR: $\delta = 106.02$ (2F), 108.69 (2F), 133.22 (2F), 135.15 (2F); ^{1}H NMR: $\delta = 1.63$ (m, 4H), 2.63-−3.18 (m, 16H); ^{31}P NMR: $\delta = 31.50$. MS (CI^+): $m/z = 503$ ($M^+ + 1$). IR (KBr plates): 2960 (s), 2848 (s), 1640 (m), 1472 (m), 1450 (m), 1385 (w), 1259 (s), 1184 (vs), 975 (s), 725 (m) cm^{-1}.

3. Results and discussion

The cyclicphosphoranes, **2A** and **2B**, were reacted via three routes with a variety of cyclic and acyclic compounds that contained labile halogen substitutents: (a) with the lithiated derivatives of **2A** and **2B** in tetrahydrofuran (THF) at reflux for 12 h; (b) with **2A** and **2B** in $CHCl_3$ at 25°C for 12–14 h; (c) with **2A** and **2B** in the presence of triethylamine in THF at 25°C for 12–24 h.

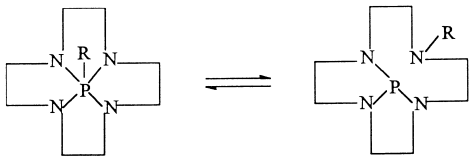
With lithiated phosphoranes, a variety of products were formed in very low yields and purification was difficult. When the phosphoranes were reacted in $CHCl_3$, the products were obtained in very good yields and their isolation and purification were straightforward. Because of the small scale on which the reactions were carried out, it was not possible to measure the quantity of HX obtained. However, when an organic base was present, the trialkylammonium salt that formed provided indirect evidence that the reaction proceeded as desired. Tetrahydrofuran (THF) was the solvent of choice for these reactions since the phosphorane products were soluble while the salts that formed precipitated in essentially quantitative amounts, indicating that the reactions occurred as follows:



R (X= Cl)	reactant	product	R (X= I)	reactant	product
c-C_5F_4Cl	**3**	**A**	C_6F_5	**6**	**D**
c-C_5F_6Cl	**4**	**B**	$C_2H_2F_3$	**7**	**E**
c-C_6F_8Cl	**5**	**C**			
C_4F_6Cl	**8**	**F**			

3.1. Reactions with **2A**

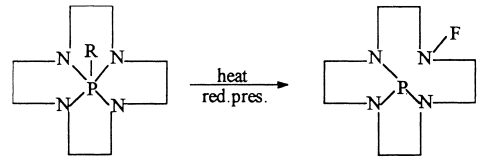
As stated in the Section 1, **2A** exists only as the 'closed' tautomer and does not show the 'open' tautomer form either in solution or in solid or gas phase. The first monodentate P-bound transition metal complex of **2A** was prepared and its X-ray crystal structure was obtained by Lattman [6–9]. In our studies, cyclenphosphorane was reacted with chlorofluoro or iodofluoro compounds to form products that contained pentacoordinated phosphorus. Interestingly, when dichlorotetrafluorocyclobutene (**3**) was reacted with **2A** in the presence of $(C_2H_5)_3N$, to give **A**, the reaction occurred more rapidly than with the dichloroperfluorocyclic olefins **4** and **5**. The ^{31}P NMR spectrum showed a signal at $\delta = +110$ ppm which is a marked shift from the region characteristic for P(V) at $\delta = -46.28$ ppm. This suggested that a P(III) derivative may be present based on the following equilibrium:



R = c-C_4F_4Cl

All attempts to isolate the proposed P(III) derivative failed.

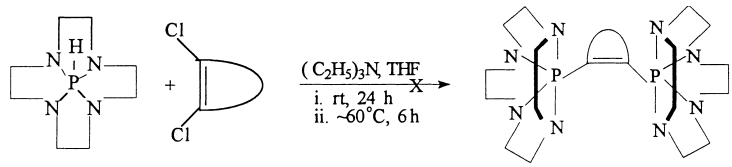
In reactions of **2A** with **4** and **5**, similar shifts in the ^{31}P NMR signals were not observed. When **4** and **5** were reacted with **2A** in the presence of a base, the reaction rate was slower than with **3**. Similar reaction rates were also observed in our earlier studies with tetraazamacrocyclic amines [19]. The amine salts were recovered from the reaction mixtures in essentially quantitative yields supporting the formation of products **B** and **C**, respectively. However, when purification was attempted by using bulb-to-bulb distillation, decomposition occurred to give the fluorocyclenphosphorane (**J**) which was confirmed by comparing the ^{31}P and ^{19}F NMR spectra and mass spectroscopy (MS) with the previously reported literature values [20]. Cyclohexene **5** with **2A** in the presence of triethylamine in THF gave **J** in 25% yield in addition to product **C**.



R = c-C_5F_6Cl, c-C_6F_8Cl **J**

Although the reaction of **5** with cyclenphosphorane was slower than with the four- and five-membered cyclic olefins, the reaction rate was much faster than with tetraazamacrocyclic amines where many days were required for the reaction to reach completion even at higher temperature. However, the reactions of **3** with the latter amines were also much faster than the reactions of the amines with **4** and **5** [18–21].

In attempts to form the 2 : 1 products, the cyclic olefins **3**, **4** and **5** were reacted with **2A** in a 2 : 1 stoichiometric ratio in the presence of triethylamine at 25°C for 24 h and then at ~60°C for 6 h. In all cases, only 1 : 1 products were formed (**A**, **B** and **C**) and unreacted **2A** was recovered.



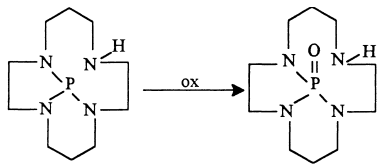
2A **3**, c-$C_4F_4Cl_2$; **4**, c-$C_5F_6Cl_2$; **5**, c-$C_6F_8Cl_2$

Iodopentafluorobenzene (**6**) reacted smoothly with **2A** in the presence of triethylamine in THF at 25°C to form **D** in 90% yield. Bromopentafluorobenzene also reacted under similar conditions with **2A** to give **D** in 70% yield. The reaction rate was slower than with **5**. When iodotrifluoro-

ethane (**7**) was allowed to react with **2A** under similar conditions, the reaction took place slowly with a concomitant color change from colorless to yellow. To compare with the reactivity of cyclic alkenes towards **2A**, $CF_3C(Cl)=C(Cl)CF_3$ (**8**) was also reacted to form **F**. Substitution of a single chlorine atom was observed but the yield was somewhat lower than with the cyclic olefins.

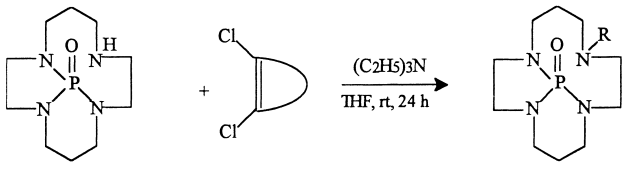
3.2. Reactions with 2B

In contrast to **2A**, cyclamphosphorane (**2B**) exists in tautomeric forms suggesting that it might exhibit different chemical behavior towards dichloroperfluorocyclic and acyclic alkenes. The reactions between **2B** and dichloroperfluorocyclic alkenes (**3**, **4** and **5**) were carried out under the same conditions used for the reactions with **2A**. While reactions occurred with **3**, **4** and **5**, the desired products could not be isolated due to the complex product mixtures. Apparently the P(III) tautomer was oxidized during the reaction to form the phosphine oxide.



2B **2C**

Because of the coexistence of P(III) and P(V) tautomeric forms and the ease of oxidation of one tautomer (the P(III) form), the studies were complicated. Some reactions of **3**, **4** and **5** were carried out with cyclamphosphine oxide (**2C**) in the presence of triethylamine. N-substituted derivatives of **2C** were formed in each case and the amine salt formed during the reaction was isolated in essentially quantitative yield. The reactions occurred as follows and are very similar to the reactions of **3**, **4** and **5** with tetraazamacrocyclic amines where N-substituted derivatives were prepared [19].



2C **3, 4, 5,**

G (R = c-C_4F_4Cl)

H (R = c-C_5F_6)

I (R = c-C_6F_8Cl)

The compounds **G**, **H**, and **I** have been isolated and characterized by ^{1}H, ^{31}P, and ^{19}F NMR spectroscopy and mass spectra were obtained that showed peaks assignable to molecular ions. These compounds are hygroscopic. They are slightly soluble in THF and methylene chloride and soluble in acetonitrile, dimethylformamide and dimethyl sulfoxide.

The infrared spectra of all of the products reported in this paper are consistent with their expected structures. Cyclic alkenyl bonds are observed as strong bands between 1600 and 1750 cm^{-1}. Ring protons of the substituted cyclenphosphorane or cyclamphosphorane are found at frequencies between 2950 and 2990 cm^{-1}. The double bonds between phosphorus and oxygen are seen as medium to strong vibrations between 1400 and 1480 cm^{-1} for **G**, **H**, and **I**.

Acknowledgements

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