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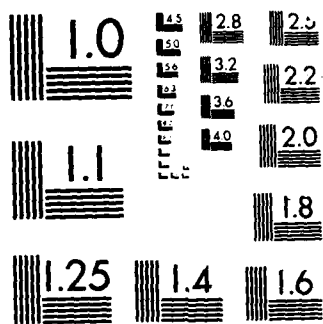
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ORGANOPHOSPHAZENES. 20. CARBONYL FUNCTIONALIZED
ARYL FLUOROCYCLOTRIPHOSPHAZENES.¹

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ABSTRACT

The reactions of 2-(3-lithiophenyl)-1,3-dioxane and 2-(4-lithiophenyl)-2-methyl-1,3-dioxane with hexafluorocyclotriphosphazene have been examined. Subsequent deacetalization to give the unprotected carbonyl compounds has been achieved. The new carbonyl functionalized aryl phosphazenes were characterized by mass spectrometry and infrared and nmr (¹H, ¹³C, ³¹P) spectroscopy.

INTRODUCTION

Reactions of an organofunctional substituent on a cyclophosphazene ring have been the subject of several recent investigations. The conversions of olefinic,² para-lithiophenoxy³ and para-aminophenoxy⁴ phosphazenes to novel cyclophosphazenes and phosphazene polymers have been reported. Since the aryl group can act as a carrier for a wide range of functional groups, arylphosphazenes are a particularly well

suited class of compounds for these studies. While certain functionalized arylphosphazenes are directly available from the reactions of halophosphazenes with aryllithium⁵ or Grignard^{6,7} reagents, systems with the broadest spectrum of reactivity are usually sensitive to organometallic reagents and hence must be prepared by indirect routes. In this paper, we present the preparation of arylphosphazenes with carbonyl moieties as substituents on the arene. Acetonyl-⁸ and p-(formyl)phenoxyphosphazenes⁹ have previously been reported.

EXPERIMENTAL

Hexafluorocyclotriphosphazene¹⁰, $N_3P_3F_6$ (1), 2-(3-bromophenyl)-1,3-dioxane and 2-(4-bromophenyl)-2-methyl-1,3-dioxane¹¹ were prepared by previously reported procedures. Diethylether was distilled from sodium benzophenone. Petroleum ether (bp 30-60°) was distilled from sodium ribbon. Methyl triphenylphosphonium bromide, n-butyl lithium (1.55 M in hexanes), and lithium aluminum hydride were purchased from Aldrich and used as received. Nmr spectra (in $CDCl_3$) were recorded on a Brüker WM 250 spectrometer operating at 250.1 (1H), 62.9 (^{13}C) and 101.2 (^{31}P) MHz. Tetramethylsilane was used as an internal reference for 1H and ^{13}C while 85% H_3PO_4 was used as an external standard for ^{31}P spectra. Chemical shifts upfield from the reference were assigned a negative value and ^{13}C and ^{31}P spectra were recorded under conditions of broad band decoupling. Infrared spectra were obtained as thin films on NaCl or KBr disks on a Nicolet 6000 spectrometer. Mass spectra were recorded on a Finnigan 4610 instrument operating at 70 eV.

Preparation of (3-Formylphenyl)pentafluorocyclotriphosphazene propylene acetal, $N_3P_3F_5(3-C_6H_4CHOCH_2CH_2CH_2O)$, 2.

A previously described air sensitive reagent reaction vessel¹² equipped with a magnetic stirrer, nitrogen inlet, oil bubbler and pressure equalizing dropping funnel was charged with 0.35 g (0.05 mol) of lithium wire in 25 mL of diethyl ether. After flushing the system with nitrogen, a solution of 4.86 g (0.02 mol) of 2-(3-bromophenyl)-1,3-dioxane in 75 mL of diethylether was slowly added and the solution left to stir overnight. The lithiated dioxane was transferred¹² to a solution of 5.00 g (0.02 mol) of 1 in 100 ml of diethylether at 0°. When the transfer was complete, the solution was allowed to warm to room temperature and stirred overnight. The solvent was removed and petroleum ether added to precipitate the lithium salts. The solution was filtered and the solvent removed to afford a yellow oil which was distilled under reduced pressure to give 3.31 g (42.3% of theory) of a clear liquid, b.p. 70-73° (0.03 mm Hg).

¹H Nmr:¹³ δ H(aromatic) 7.70-8.02 m (4H); δ $OCH_2CH_2CH_2OCH-$ 4.12 m (4H); δ $OCH_2CHHCH-CH-$ 2.12 m (1H); δ OCH_2CHHCH_2OCH 1.41 m (1H); δ $O(CH_2)_3OCH-$ 5.53 s (1H).

Preparation of (3-Formylphenyl)pentafluorocyclotriphosphazene, $N_3P_3F_5(3-C_6H_4CHO)$, 3.

Thirty drops of a 50% (v,v) aqueous H_2SO_4 solution were added to a slurry of 10 g of silica gel (70-230 mesh) in 50 mL of methylene

chloride and stirred until the aqueous phase had absorbed onto the silica.¹⁴ Then, 3.00 g (0.0076 mol) of 2 were added and the slurry stirred for five days. After neutralization with NaHCO_3 , the slurry was filtered and washed with diethyl ether. The filtrates were combined and the solvents removed to give 1.89 g (74.7% of theory) of a clear liquid, b.p. 68-71° (.03 mm Hg). Anal. Calcd for $\text{N}_3\text{P}_3\text{F}_5\text{C}_7\text{H}_5\text{O}$, mol. wt. 335. Found: m 335 (mass spectrum).

^1H Nmr: δ H(aromatic) 7.75-8.09 m (4H), δ CHO 9.99 s (1H). ^{31}P Nmr δ = PRF 35.14 m (1P), $^1J_{\text{PF}} = 988$, $^2J_{\text{PP}} = 77.4$, $^3J_{\text{PFtrans}} = 19.3$, $^3J_{\text{PFcis}} = 3.5$; δ = PF₂ 9.15 m (2P), $^1J_{\text{PF}} = 895$. ^{13}C Nmr:¹⁵ δ C₂ 129.74, δ C₃ 130.74, δ C₄ 131.88, δ C₅ 141.09, δ C₆ 127.58; δ C₇ 192.41. IR:¹⁶ 1695 (s, $\nu_{\text{C=O}}$), 1589 (m, $\nu_{\text{C=C}}$ aromatic), 1274 (s, ν_{PN}), 945 (m, ν_{PF} asym), 838 (m, ν_{PF} sym).

Attempted Wittig Reaction of 3. The reaction of triphenylphosphine methylene (prepared from methyl triphenylphosphonium bromide and butyl lithium) with 3 yielded a poorly characterized oil. The nmr spectrum of this material contained no aldehydic or styryl proton resonances.

Preparation of (4-Acetylphenyl)pentafluorocyclotriphosphazene propylene acetal, $\text{N}_3\text{P}_3\text{F}_5\text{-4-C}_6\text{H}_4\text{C(CH}_3\text{)OCH}_2\text{CH}_2\text{CH}_2\text{O}$, 4. The preparation is similar to that of 2 except that 2-(4-bromophenyl)-2-methyl-1,3-dioxane (10.63 g, 0.0413 mol) in 125 mL of ether was used in place of 2-(3-bromophenyl)-1,3-dioxane. The lithiated dioxane was allowed to

react with a solution of 10.33 g (0.0413 mol) of 1 in 200 mL of ether. The resulting oil was distilled at reduced pressure to give 11.21 g (66.7 % of theory) of a clear liquid, b.p. 82-85° (0.02 mm Hg).

$^1\text{H Nmr}$: $^{17}\delta_{\text{HO}}$ 7.95 m (2H); δ_{Hm} 7.69 m (2H); $\delta_{\text{OCH}_2\text{CH}_2\text{CH}_2\text{OC}(\text{CH}_3)-}$ 3.81 m (4H); $\delta_{\text{OCH}_2\text{CHHCH}_2\text{OC}(\text{CH}_3)-}$ 2.13 m (1H); $\delta_{\text{OCH}_2\text{CHHCH}_2\text{OC}(\text{CH}_3)-}$ 1.28 m (1H); δ_{CH_3} 1.51 s (3H).

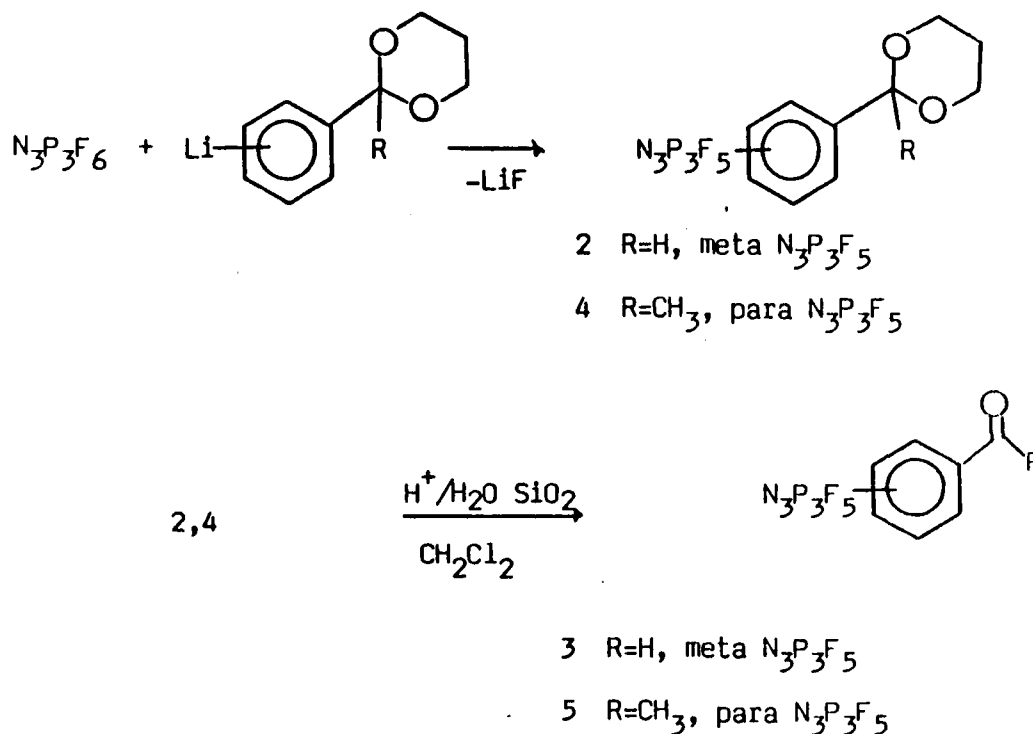
Preparation of 4-Acetylphenylpentafluorocyclotriphosphazene,

$\text{N}_3\text{P}_3\text{F}_5-4-\text{C}_6\text{H}_4\text{C}(\text{O})\text{CH}_3$, 5. The procedure is similar to that used to prepare 3. In a typical experiment, 1.00 g (0.0025 mol) of 4 were added to the acidic silica gel slurry and stirred for two days. The resulting oil was distilled to give 0.73 g (85.0% of theory) of a clear liquid, b.p. 72-75° (0.03 mm Hg). Anal. Calcd. for $\text{N}_3\text{P}_3\text{F}_5\text{C}_8\text{H}_7\text{O}$ mol. wt. 359. Found: 359 (mass spectrum). $^1\text{H Nmr}$: $\delta_{\text{H}(\text{aromatic})}$ 7.96-8.10 m (4H); δ_{CH_3} 2.65 s (3H). $^{31}\text{P Nmr}$: δ_{PFR} 33.78 m (1P), $^1J_{\text{PF}} = 989$, $^2J_{\text{PP}} = 79.3$, $^3J_{\text{PFtrans}} = 19.6$, $^3J_{\text{PF cis}} = 4.3$; δ_{PF_2} 8.91 m (2P), $^1J_{\text{PF}} = 902$. $^{13}\text{C Nmr}$: $^{18}\delta_{\text{C}_2}$ 128.89; δ_{C_3} 131.5; δ_{C_4} 141.82, δ_{C_5} 197.45, δ_{C_6} 26.89. IR: 16 1698 (s, $\nu_{\text{C=O}}$), 1587 (m $\nu_{\text{C=C}}$ aromatic), 1272 (s, $\nu_{\text{P=N}}$), 943 (m, ν_{PFasym}), 839 (ν_{PF} , sym).

Attempted Reduction of 5. Although the reaction of 5 with lithium aluminum hydride in diethyl ether was accompanied by the disappearance of the carbonyl band in the IR, no isolable product was obtained.

RESULTS AND DISCUSSION

Carbonyl functionalized arylphosphazenes may be produced through the action of lithiated benzaldehyde or acetophenone derivatives on $\text{N}_3\text{P}_3\text{F}_6$ (1), provided that the carbonyl moiety is protected as an acetal. Removal of the protecting group may be effected through the use of diluted H_2SO_4 on silica gel. The new carbonyl derivatives were



characterized by infrared (IR) and nmr (^1H , ^{13}C , ^{31}P) spectroscopy as well as mass spectrometry.

The proton nmr spectra of the dioxane derivatives 2 and 4 resemble those of the parent organic compounds with additional phosphorus-proton coupling in the phenyl region. All of the aryl and benzylic

protons resonances are shifted downfield relative to the parent compounds due to the strong electron withdrawing effect of the $\text{N}_3\text{P}_3\text{F}_5$ residue.¹⁹

The lower yield of 2 relative to 4 may be related to the fact that the benzylic proton of 2 is acidic and could be removed by the organolithium reagent. The resulting carbanion may then enter into undesirable side reactions. By way of contrast, 4 has no benzylic protons thus this potential for side reactions is obviated.

The acetal protecting groups were removed using dilute H_2SO_4 adsorbed onto silica gel. More conventional methods of acetal cleavage²⁰ failed, as the $\text{N}_3\text{P}_3\text{F}_5$ group is sensitive to the aqueous acidic conditions usually employed. An additional benefit of this procedure is that the 1,3 propane diol liberated in the reaction, which could react further with the phosphazene,²¹ is adsorbed onto the silica gel and is easily removed from the reaction mixture.

The ^1H nmr spectra of compounds 3 and 5 also resemble those of benzaldehyde and acetophenone with additional phosphorus-proton coupling being evident in the aryl proton region. A comparison of the carbonyl carbon chemical shifts between the parent organic compounds and the phosphazene derivatives reveals a downfield shift of about 1.5 ppm due to the electron withdrawing effect of the phosphazene. The ^{31}P nmr data are consistent with a monosubstituted phosphazene. The downfield doublet due to the ^mPFR center is interpretable on a first

order basis and the values of $^1J_{PF}$, $^2J_{PP}$, $^3J_{PF}$ (cis and trans) are easily obtained. The resonance for the $=PF_2$ centers is second order in nature. The IR spectra of 3 and 5 contain the characteristic bands for a monoarylpentafluorocyclotriphosphazene²² as well as the $\nu_{C=O}$ and $\nu_{C=C}$ bands associated with an aromatic carbonyl compound. It is interesting to note that value for $\nu_{C=O}$ in 5 is identical to that of p-nitroacetophenone. Other measurements indicate that the electron withdrawing ability of the $N_3P_3F_5$ group on a phenyl ring is approximately equivalent to that of a nitro group.¹⁹

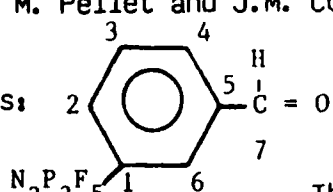
Limited attempts at further synthetic transformations of the carbonyl groups directed towards the preparation of olefins² were unsuccessful. As indicated by IR spectroscopy, the carbonyl group undergoes reaction but only unidentified degradation products were obtained. It is believed that oxyanions produced in the reaction undergo further reactions with the $N_3P_3F_5$ moiety. Thus, any synthetic applications of these materials must involve sequences which can not be interrupted by reactions at the phosphorus (v) center.

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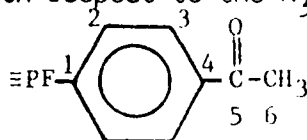
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13. Chemical shifts in ppm and coupling constants in Hz.
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15. Assignments are as follows:



The resonance for C_1 is not observed due to the reduced intensity of a quaternary carbon combined with extensive phosphorus and fluorine coupling.
16. In cm^{-1} .

17. Ho and Hm refer to position with respect to the $\text{N}_3\text{P}_3\text{F}_5$ group.

18. Assignments are as follows:



The resonance for C_1 is not observed due to the reduced intensity of quaternary carbon combined with extensive phosphorus and fluorine coupling.

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