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PHOSPHA-S-TRIAZINES. VI. POLYMERIC SYSTEMS

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SUMMARY

Two types of polymeric phospha-s-triazines were synthesized: poly(perfluoroalkylethermonophospha-s-triazines) and monocyclic mono- and diphospha-s-triazines substituted on the carbon ring atoms by poly-(perfluoroalkylether) chains. The polymeric system consisting of the monophospha-s-triazine rings joined by perfluoroalkylether chains were found to have lower thermal oxidative stability than that shown by the corresponding dumbbell compounds. The monocyclic materials substituted by poly(perfluoroalkylether) chains possessed good thermal and oxidative stability, in addition to exhibiting anticorrosive and antioxidative properties.

INTRODUCTION

One of the potential uses for phospha-s-triazines is as high temperature fluids and additives. In actual applications, high molecular weight materials offer many advantages as compared to the relatively low molecular weight compounds synthesized to date [1,2]. One can visualize phospha-s-triazine polymers as arrangements of rings joined by chains or as single ring compounds substituted by very long side chains. The former concept is the more conventional and one which could lead to high molecular weight compositions. On the other hand, this approach requires that the ring closing process proceeds to completion and that the polymers are capped

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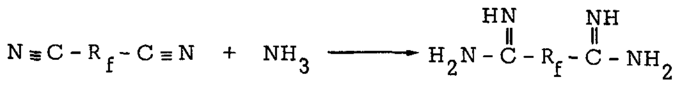
by unreactive end groups. The second system contains a very low percent of the phospha-s-triazine units and in its synthesis necessitates conducting all the reactions on polymeric materials. Consequently, in both instances incompleteness of any given process would be expected to result in low stability of the final product. To assess the advantages of either one of these arrangements, it appeared best to synthesize the representative materials and determine their thermal and oxidative stabilities in addition to their applicability as antioxidants and anticorrosion additives for perfluoroalkylether fluids.

RESULTS AND DISCUSSION

Poly(perfluoroalkylethermonophospha-s-triazines)

Poly(perfluoroalkylimidoylamidines) were successfully prepared and transformed into polytriazines by Dorfman and Emerson [3]. A parallel approach was followed in the synthesis of poly(perfluoroalkylethermonophospha-s-triazines). In the preparation of the poly(imidoylamidine) precursor, both the addition of the bis-amidine to the dinitrile and the addition of dinitrile to the bis-amidine were employed; the products were essentially identical. To ensure amidine termination, the polymer was treated with ammonia, followed by addition of a monofunctional perfluoroalkylether nitrile to provide the unreactive end-groups. The final step was the ring closure via reaction with diphenyltrichlorophosphorane. The overall reaction sequence employed is presented below:

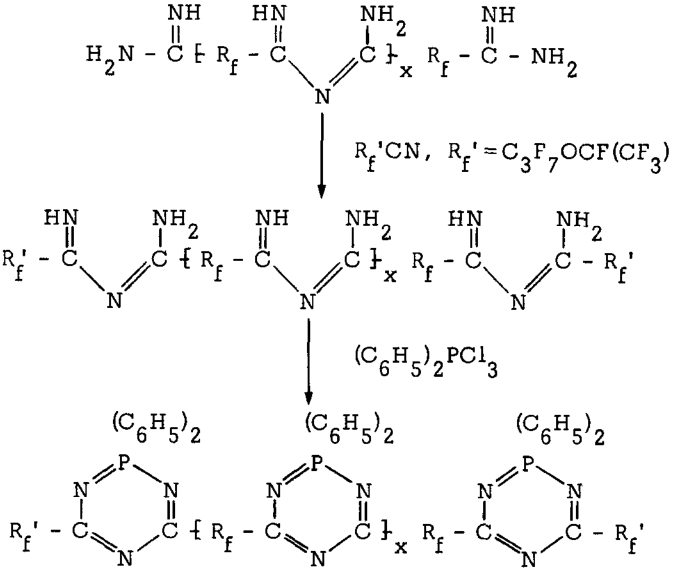


$$N\equiv C-R_f-C\equiv N \;+\; NH_3 \longrightarrow H_2N-\overset{\overset{HN}{\|}}{C}-R_f-\overset{\overset{NH}{\|}}{C}-NH_2$$

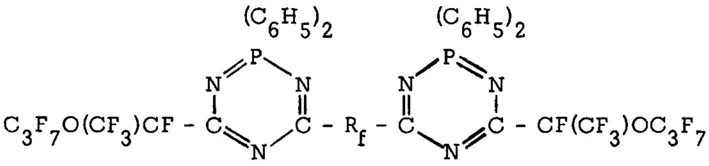
$R_f = CF(CF_3)[OCF_2CF(CF_3)]_mO(CF_2)_5O[CF(CF_3)CF_2O]_nCF(CF_3)$, $m + n = 3$

(The abbreviation used for this segment is 5-EDAF.)

$$\Big\downarrow\; N\equiv C-R_f-C\equiv N,\; NH_3$$



As evident from Table 1, both materials exhibited lower thermal and thermal oxidative stability than that found for the corresponding monomeric analogues [4]. The observed discrepancy in the thermal behavior could be caused by the possible presence of a CF_2 group adjacent to the ring derived from the potential 5-EDAF isomer, $-CF_2(CF_2)_3O[CF(CF_3)CF_2O]_4CF(CF_3)-$. No easy tests are available [5] to determine the presence and relative quantity of this isomer in, e.g., 5-EDAF-dinitrile and furthermore, even if this isomer was found to be present, it cannot be unequivocally assumed that the CF_2 linkage adjacent to the phospha-s-triazine ring is the cause of the lowered thermal stability. To prove whether indeed the lowered thermal stability is due to the 5-EDAF group, the dumbbell compound



R_f = 5-EDAF

TABLE 1

Degradation of poly(monophospha-s-triazines)[a]

Test	Temp	Atm	Starting Material		Residue		Oxygen Consumed		Volatiles	
No.	°C		mg	MW	%[b]	MW	mg	%[c]	mg	%[b]
1[d]	235	Air	527.7	7200	96.9	4800	1.42	22.5	15.2	2.9
2[d]	316	Air	488.6	7200	92.5	1600	4.61	72.1	47.5	9.7
3[d]	316	N_2	491.8	7200	94.2	1900	-	-	29.8	6.1
4[d]	235	Air	459.9	6800	98.5	4800	1.08	16.6	5.2	1.1
5[d]	316	Air	476.1	6800	91.8	1600	5.31	84.0	45.4	9.5
6[d]	316	N_2	532.7	6800	93.9	2400	-	-	28.7	5.4
7[e]	235	Air	553.5	2200	99.4	2210	0.0	0.0	2.8	0.5
9[e]	235	Air	559.2	2050	99.6	2040	0.0	0.0	2.1	0.4

a) All tests were performed over a period of 24 hr. b) Weight percent of starting material. c) Percent of oxygen available. d) In Tests 1-3 the polymer was prepared from polyimidoylamidine obtained by adding 5-EDAF-bisamidine to the dinitrile; the polyimidoylamidine in Tests 4-6 was formed by the reverse addition. e) Tests 7 and 9 were carried out on dumbbell materials; the compound in 7 used imidoylamidine prepared from 5-EDAF-bisamidine and $C_3F_7OCF(CF_3)CN$; in 9 the imidoylamidine was from reaction of $C_3F_7OCF(CF_3)$-$C(=NH)NH_2$ with 5-EDAF-dinitrile.

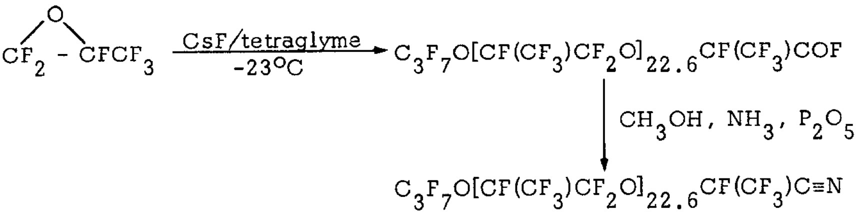
wherein the two rings are joined by the 5-EDAF link was prepared by the two different methods. The low temperature cyclization in the presence of the triethylamine acid acceptor utilized in the last step was found to result in a better yield and a purer product than the procedure originally described [6].

The dumbbell compounds exhibited thermal oxidative stability, Table 1, at least at 235°C, comparable to that of the monomeric compounds [4]. Consequently, it must be deduced that the lower stability of the polymonophospha-s-triazines must be due to some aspect of the polymerization process resulting in the formation of weak links.

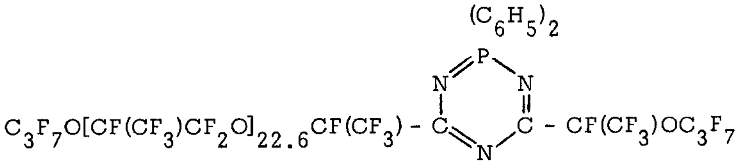
Monomeric phospha-s-triazines substituted by polymeric perfluoroalkylether chains

Monophospha- and diphospha-s-triazines substituted on the ring carbon atoms by poly(hexafluoropropene oxide) chains of the general formula $C_3F_7O[CF(CF_3)CF_2O]_{1,2}CF(CF_3)$- were found to exhibit good thermal and oxidative stabilities in addition to providing an antioxidative and anticorrosive protection when utilized as additives to Krytox fluids in the presence of M-50 alloy [1,2]. However, in view of their relatively low molecular weight, these materials were not ideally suited as high temperature fluids. Furthermore, it was of interest to determine whether phospha-s-triazines, wherein the ring itself constitutes only a very small portion of the molecule, will still possess the anticorrosive and antioxidative properties. In view of the development of the low temperature, high yield synthesis process, the purification via distillation became unnecessary; thus permitting the synthesis of materials where the number of the repeating hexafluoropropene oxide units was of the order of 20.

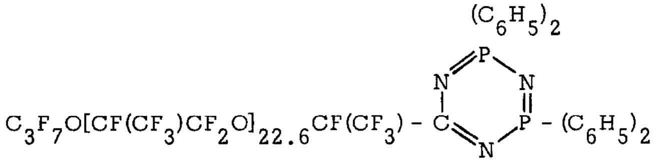
The preparation of the long chain acid fluorides directly from hexafluoropropene oxide, followed by transformation to the nitrile, was carried out utilizing the previously-described procedures [7,8]. The reaction scheme is given below:



To obtain the monophospha-s-triazine substituted by the long chain perfluoroalkylether on one carbon and the short chain ether, $C_3F_7OCF(CF_3)$- on the other, the above nitrile was transformed into an amidine which was then treated with the short chain nitrile, $C_3F_7OCF(CF_3)CN$. The ring closure was effected by reaction with diphenyltrichlorophosphorane giving the desired product:



The diphospha-s-triazine was synthesized in a parallel fashion, resulting in:



The molecular weights and elemental analyses of the above-discussed mono- and diphospha-s-triazines were found to be in good agreement with the theoretical values. Both of these materials exhibited very good thermal and oxidative stabilities as evident from the data given in Table 2. The antioxidative and anticorrosive action of these phospha-s-triazine compositions, Table 3, when utilized as additives in Krytox fluids (Du Pont trade name, $F\text{-}[CF(CF_3)CF_2O]_nC_2F_5$) was significantly better than that observed for the short chain perfluoroalkylether-substituted analogues prepared

TABLE 2

Degradation of poly(hexafluoropropene oxide) substituted mono- and diphospha-s-triazines[a]

Material	Temp	Atm	Starting Material		Residue		Oxygen Consumed		Volatiles	
	°C		mg	MW	%[b]	MW	mg	%[c]	mg	%[b]
$[(\text{poly HFPO})\text{CN}][(\text{HFPO})_2\text{CN}][\Phi_2\text{PN}]$[d]	235	Air	563.2	4700	99.9	4500	0.0	0.0	0.5	0.1
-ditto-	316	Air	394.3	4700	98.6	4400	0.3	0.8	5.0	1.3
-ditto-	316	N_2	378.3	4700	99.7	4500	-	-	1.2	0.3
$[(\text{poly HFPO})\text{CN}][\Phi_2\text{PN}]_2$[e]	235	Air	637.7	4700	99.9	4900	0.0	0.0	0.6	0.1
-ditto-	316	Air	292.5	4700	98.5	4400	2.3	7.3	4.1	1.4
-ditto-	316	N_2	299.8	4700	99.1	4800	-	-	2.2	0.7

a) All the tests were performed over a period of 24 hr. b) Weight percent of starting material. c) Percent of oxygen available. d) $[(\text{poly HFPO})\text{CN}][(\text{HFPO})_2\text{CN}][\Phi_2\text{PN}]$ represents 1-diphenylphospha-3-($C_3F_7O[CF(CF_3)CF_2O]_x$-$CF(CF_3)$)-5-($C_3F_7OCF(CF_3)$)-2,4,6-triazine. e) $[(\text{poly HFPO})\text{CN}][\Phi_2\text{PN}]_2$ represents 1,3-bis(diphenylphospha)-5-($C_3F_7O[CF(CF_3)CF_2O]_xCF(CF_3)$)-2,4,6-triazine.

TABLE 3

Degradation of Krytox fluid in the presence of M-50 alloy coupon at 316°C in oxygen over a period of 24 hr[a]

Fluid Used g	Additive	Oxygen Consumed Total mg	Oxygen Consumed %[b]	Oxygen Consumed mg/g[c]	Total Products Formed mg	Total Products Formed mg/g[d]
12.13	none	70.8	24.6	5.84	576.7	47.54
12.91	1%[e] [(poly HFPO)CN][(HFPO)$_2$CN][Φ_2PN][f]	0.0	0.0	0.0	1.7	0.13
13.38	1% [(poly HFPO)CN][Φ_2PN]$_2$[g]	0.0	0.0	0.0	2.4	0.18

a) The apparatus consisted of a sealed glass tube wherein the metal coupon was suspended in the fluid; the test was conducted in pure oxygen; at the conclusion of the test, the oxygen was measured and the products were collected and measured. b) Percent of oxygen available. c) Oxygen consumed in mg/g Krytox employed. d) Products formed in mg/g Krytox employed. e) The percent is weight percent of additive per weight of Krytox fluid. f) [(poly HFPO)CN][(HFPO)$_2$CN][Φ_2PN] represents 1-diphenylphospha-3-($C_3F_7O[CF(CF_3)CF_2O]_xCF(CF_3)$)-5-($C_3F_7OCF(CF_3)$)-2,4,6-triazine. g) [(poly HFPO)CN][Φ_2PN]$_2$ represents 1,3-bis(diphenylphospha)-5-($C_3F_7O[CF(CF_3)CF_2O]_xCF(CF_3)$)-2,4,6-triazine.

earlier [1,2]. It would thus appear that poly(hexafluoropropene oxide) units capped by phospha-s-triazine rings offer much better candidates for high temperature fluids and fluid additives than poly(monophospha-s-triazines).

EXPERIMENTAL

General

All solvents used were reagent grade and were dried and distilled prior to use. Operations involving moisture or air sensitive materials were carried out either in an inert atmosphere enclosure (Vacuum Atmospheres Model HE-93B), under nitrogen by-pass, or in vacuo. The commercially available starting materials were usually purified by distillation, crystallization, or other appropriate means.

Infrared spectra were recorded either neat (on liquids) or as double mulls (Kel-F oil No. 10 and Nujol) using a Perkin-Elmer Corporation Infrared Spectrophotometer Model 21. The molecular weights were determined in hexafluorobenzene using a Mechrolab Model 302 vapor pressure osmometer. The mass spectrometric analyses were obtained employing a Du Pont 21-491B double focusing mass spectrometer attached to a Varian gas chromatograph Model 2700, equipped with a flame ionization detector, and a Du Pont 21-094 data acquisition and processing system. The elemental analyses were performed by Schwarzkopf Microanalytical Laboratory, Woodside, New York.

Stability investigations

The degradations were performed in sealed ampoules of ~ 50 ml volume over a period of 24 hr at the specified temperatures. The atmospheres studied were nitrogen and air; the gas pressures used were ~ 350 mm Hg at room temperature. The samples were introduced into the ampoules as Freon-113 solutions. The solvent was removed in vacuo and the sample dried at 50°C or above. At the conclusion of the experiment, the ampoules were cooled in liquid nitrogen and opened into the vacuum system. The liquid nitrogen noncondensibles were measured and analyzed by gas chromatography. The liquid nitrogen condensibles were fractionated from a warming trap through -23, -78°C into a liquid nitrogen cooled trap. Each fraction was measured, weighed, and analyzed by infrared spectroscopy, batch mass spectrometry, and GC-MS. The involatile residues were handled in an inert atmosphere enclosure to avoid any interferences from subsequent hydrolysis or oxidations in the case of purely thermal degradations. The residue itself was weighed and subjected to infrared spectral analyses and molecular weight determination.

Preparation of 5-EDAF-bisamidine, 5-EDAF$[C(=NH)NH_2]_2$

To stirred liquid ammonia (70 ml) at -23°C over 50 min was added 5-EDAF-dinitrile, $N{\equiv}CCF(CF_3)[OCF_2CF(CF_3)]_mO(CF_2)_5O[CF(CF_3)CF_2O]_n$-$CF(CF_3)C{\equiv}N$ (m + n = 3), (15.11 g, 14.6 mmol). The resulting mixture was then stirred at ambient temperature and the residual ammonia was removed in vacuo to give a clear, viscous material (15.22 g; MW 1600). The infrared spectrum exhibited bands indicative of both amidine and imidoylamidine linkages.

Preparation of poly-5-EDAF amidine-imidoylamidine, $H_2NC(=NH)$-5-EDAF-$[C(=NH)N=C(NH_2)$5-EDAF$]_xC(=NH)NH_2$

To the above-prepared amidine (2.88 g, 1.88 mmol), with stirring, was added dropwise 5-EDAF-dinitrile (1.86 g, 1.80 mmol). On continued stirring, the solution became very viscous and, after standing for 7 days, the clear material had a molecular weight of 7000. To ensure amidine termination, ammonia was condensed onto a concentrated solution of the above material in Freon-113. The resulting mixture was stirred for 1 hr at -20°C, then ~ 2 hr at ambient temperature. Residual ammonia and Freon-113 were subsequently removed in vacuo to give a viscous material (4.68 g).

Adding the amidine to the nitrile, in an otherwise parallel process, resulted in 99.8% yield of the product, MW 7200.

Preparation of perfluoroalkylether-terminated poly-5-EDAF-monophospha-s-triazine, $C_3F_7OCF(CF_3)[C_2N_3P(C_6H_5)_2][5\text{-EDAF-}C_2N_3P(C_6H_5)_2]_xCF(CF_3)\text{-}OC_3F_7$

(a) Using polyimidoylamidine obtained by addition of 5-EDAF-bisamidine to 5-EDAF-dinitrile

A solution of poly(imidoylamidine), $C_3F_7OCF(CF_3)C(NH_2){=}N\text{-}C({=}NH)\text{-}[5\text{-EDAF-}C({=}NH)N{=}C(NH_2)]_xCF(CF_3)OC_3F_7$, (2.98 g, 0.48 mmol) and triethylamine (2.3 ml, 16.5 mmol) in Freon-113 (40 ml) was added over a period of 1.25 hr to a solution of diphenyltrichlorophosphorane (1.43 g, 4.9 mmol) in benzene (25 ml). The mixture was stirred and heated at 50°C for 110 hr. Upon cooling, the solids were removed by filtration and washed with benzene and Freon-113. The residue that remained following solvent removal was triturated with Freon-113. The extracts were concentrated, then filtered in Freon-113 (80 ml) through a 1.5 x 3 cm column of Woelm neutral alumina to give, after solvent removal, a viscous product (2.78 g, 79.7%); MW found, 7200. Anal. Calcd. for $C_{189.76}H_{61.80}F_{208.48}N_{18.54}O_{27.90}P_{6.18}$: C, 31.66; H, 0.87; F, 55.01; N, 3.61; O, 6.20; P, 2.66. Found: C, 31.49; H, 1.78; F, 53.02; N, 3.48; P, 2.49.

(b) Using polyimidoylamidine obtained by addition of 5-EDAF-dinitrile to 5-EDAF-bisamidine

Following the above procedure, a quantitative yield of product, MW 6800, was obtained. Anal. Calcd. for $C_{179.52}H_{58.60}F_{196.96}N_{17.58}O_{26.30}P_{5.86}$: C, 31.68; H, 0.87; F, 54.98; N, 3.62; O, 6.18; P, 2.67. Found: C, 32.11; H, 1.49; F, 52.62; N, 3.63; P, 2.73.

Preparation of $(C_3F_7OCF(CF_3)[C_2N_3P(C_6H_5)_2])_2\text{-5-EDAF}$

(a) From 5-EDAF-bisamidine

5-EDAF-Bisamidine (2.93 g) was treated with a perfluoroalkylether nitrile, $C_3F_7OCF(CF_3)CN$, (1.96 g, 6.3 mmol). After 24 hr, the excess nitrile was removed in vacuo giving a clear viscous liquid, 4.42 g. A solution of the imidoylamidine, $5\text{-EDAF-}[C({=}NH)N{=}C(NH_2)CF(CF_3)OC_3F_7]_2$, (4.29 g, 2.36 mmol) and triethylamine (2.8 ml, 20.08 mmol) in Freon-113 (5 ml) was added over a period of 21 min to a stirred solution of diphenyltrichlorophosphorane (1.78 g, 6.11 mmol) in benzene (10 ml) at 50°C. The mixture was then stirred and heated at 50°C for 92 hr. The cool solution was filtered through a 1.5 x 3 cm column of Woelm neutral alumina to afford, after solvent removal, a clear viscous product, 4.01 g (76.7% yield). Anal. Calcd. for $C_{56}H_{20}F_{58}N_6O_7P_2$: C, 32.77; H, 0.98; F, 53.68; N, 4.09; O, 5.46; P, 3.02; MW, 2052.64. Found: C, 33.34; H, 1.15; F, 54.39; N, 4.39; P, 2.74; MW, 2200.

(b) From mono-amidine, $C_3F_7OCF(CF_3)C(=NH)NH_2$

To stirred liquid ammonia (30 ml) at -28°C over 10 min was added $C_3F_7OCF(CF_3)CN$ (3.0 g, 9.64 mmol). The resulting mixture was then stirred at ambient temperature to allow the evaporation of ammonia. Residual volatiles were removed in vacuo to give a clear material (1.62 g, 51.3% yield) composed of 94% of the amidine and 6% of the corresponding imidoylamidine (determined by gas chromatography). This material (1.62 g, 4.94 mmol) was then treated with 5-EDAF-dinitrile (2.41 g, 2.33 mmol). Subsequently, a solution of the imidoylamidine, 5-EDAF[$C(NH_2)=NC(=NH)CF(CF_3)OC_3F_7]_2$, (4.01 g, 2.38 mmol) and triethylamine (2.2 ml, 15.77 mmol) in Freon-113 (5 ml) was introduced over a period of 20 min to a stirred solution of diphenyltrichlorophosphorane (1.58 g, 5.42 mmol) in benzene (10 ml) at 50°C. The mixture was then stirred and heated at 50°C for 72 hr. The cool solution, after filtration through a 1.5 x 3 cm column of Woelm neutral alumina, followed by solvent removal, gave a clear viscous product, 4.13 g (84% yield). Anal. Calcd. for $C_{56}H_{20}F_{58}N_6O_7P_2$: C, 32.77; H, 0.98; F, 53.68; N, 4.09; O, 5.46; P, 3.02; MW, 2052.64. Found: C, 33.35; H, 1.08; F, 54.56; N, 4.45; P, 2.30; MW, 2050.

Telomerization of hexafluoropropene oxide

Hexafluoropropene oxide (10.63 g, 64.03 mmol) was condensed at -196°C onto cesium fluoride (0.01 g, 0.07 mmol) and tetraglyme (0.016 ml) in a 25-ml carefully dried, evacuated round bottom flask. The flask was subsequently isolated from the remainder of the vacuum system, warmed to ~ -23°C and stirred (70 hr). At the end of the reaction, the volatiles were removed, initially at the reaction temperature then at ambient temperature. This procedure largely prevented the production of low molecular weight telomers. The residue (10.45 g) was an opaque viscous material.

Preparation of 1-diphenylphospha-3-$(C_3F_7O[CF(CF_3)CF_2O]_xCF(CF_3)$-5-$(C_3F_7OCF(CF_3))$-2,4,6-triazine

Methanol (2.5 ml) was added dropwise to the above-prepared stirred telomer; stirring was continued for 24 hr. The mixture was then transferred to a separatory funnel with Freon-113 (10 ml), washed with water (3 x 15 ml), and dried over anhydrous magnesium sulfate. After removal of solvent in vacuo, the methyl ester, 10.05 g, MW 4100, was obtained. Onto the methyl ester (9.76 g, 2.4 mmol) was then condensed ammonia (5 ml) and the mixture was stirred for 30 min, then allowed to warm to ambient temperature. Final removal of all the volatiles was accomplished in vacuo at ~ 70°C to give the amide (9.7 g). The corresponding nitrile, 7.54 g (79.5% yield) was prepared by heating a mixture of the amide (9.49 g) and phosphorus pentoxide (15.26 g, 0.11 mol) at 175-202°C for 4 hr followed by cooling and extraction with Freon-113. Subsequently, to stirred ammonia (40 ml) at -26°C was added dropwise a solution of the nitrile (4.7 g) in Freon-113 (3 ml). Removal of ammonia and the residual volatiles gave the amidine (4.58 g); MW 4600.

To prepare the imidoylamidine, $C_3F_7OCF(CF_3)C(=NH)\text{-}N\text{-}C(NH_2)CF(CF_3)[CF_2CF(CF_3)]_{22.6}OC_3F_7$, a mixture of the amidine (2.51 g) and the nitrile $C_3F_7OCF(CF_3)CN$ (1.01 g) was stirred overnight, then the excess nitrile (0.83 g) was removed in vacuo to yield the desired product (2.73 g). Finally, a solution of the imidoylamidine (2.25 g) and triethylamine (0.25 ml, 1.79 mmol) in Freon-113 (10 ml) was added over a period of 30 min to a solution of diphenyltrichlorophosphorane (0.26 g, 0.89 mmol) in benzene (5 ml) at 50°C. The mixture was then stirred and heated at 50°C for 100 hr. After removal of solvents under reduced pressure, the residue was treated with Freon-113 (4 x 20 ml); each portion was filtered through a 1.5 x 3 cm column of neutral Woelm alumina. The solvent was removed in vacuo to give a viscous product, 2.13 g. Anal. Calcd. for $C_{91.86}H_{10}F_{157.72}N_3O_{24.62}P$: C, 24.10; H, 0.22; F, 65.47; N, 0.92; O, 8.61; P, 0.68; MW, 4576.92. Found: C, 24.28; H, 0.47; F, 66.78; N, 0.68; P, 0.44; MW, 4700.

Preparation of 1,3-bis(diphenylphospha)-5-($C_3F_7O[CF(CF_3)CF_2O]_xCF(CF_3)$)-2,4,6-triazine

A solution of the amidine, $C_3F_7O[CF(CF_3)CF_2O]_{22.6}CF(CF_3)C(=NH)-NH_2$, (2.07 g) and triethylamine (0.35 ml, 2.51 mmol) in Freon-113 (4 ml) was introduced over a period of 30 min to a solution of imido-tetraphenyldiphosphinic acid trichloride (0.95 g, 1.94 mmol) in acetonitrile (10 ml) at 50°C. The mixture was then stirred and heated at 50°C for 136 hr. After removal of the solvents under reduced pressure, the residue was treated with Freon-113 (5 x 15 ml); each portion was filtered through a 1.5 x 3 cm column of neutral Woelm alumina. The solvent was removed in vacuo to give a viscous product, 1.72 g. Anal. Calcd. for $C_{97.86}H_{20}F_{146.72}N_3O_{23.62}P_2$: C, 26.32; H, 0.45; F, 62.43; N, 0.94; O, 8.46; P, 1.39; MW, 4465.02. Found: C, 25.86; H, 0.65; F, 64.45; N, 0.88; P, 0.82; MW, 4700.

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