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INTERACTION OF P(III) COMPOUNDS WITH SILYL HALIDES. THE MECHANISM OF THE REACTION OF PHOSPHOROUS TRIAMIDES WITH TRIALKYLSILYL HALIDES

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Abstract - The reaction of phosphorous hexamethyltriamide with trimethylsilyl iodide or bromide provides a convenient synthesis of the corresponding N,N,N',N'-tetramethylphosphorodiamidous halide. Low temperature ^{31}P NMR studies gave evidence of the formation of a phosphonium salt-type complex with direct P-Si bonding in the first step of the reaction. Analogous phosphonium salt intermediates involving P-P bonds were found to be involved in some ligand exchange process between two tricoordinated phosphorus species.

INTRODUCTION

So far little is known about the interaction of tricoordinate phosphorus with silicon compounds. Tertiary phosphines have been reported to form complexes of phosphonium salt structure with trialkylsilyl halides.[1] On the other hand it is known that the reaction of silyl halides with trialkyl phosphites, and presumably with other esters of tricoordinate phosphorus oxyacids, departs considerably from the classical Arbuzov scheme typical for the analogous processes involving alkyl halides.[2] We have shown recently[3] that the first step in the reaction of trimethylsilyl iodide or bromide with trimethyl phosphite is the formation of the dialkyl halophosphite. The elucidation of the detailed mechanism of this step could have more general significance since analogous ligand exchange processes between silicon and tricoordinate phosphorus are well-known and used in synthesis.[4,5] They also resemble exchange processes between two tricoordinate phosphorus species, which are very common in phosphorus chemistry[6,7] and analogous reactions between P(III) compounds and acyl halides.[8-10] No Arbuzov-type intermediates involving P-Si interaction have so far been detected in such processes involving silicon species, nor is the Arbuzov-type mechanism in agreement with some kinetic features of the reaction of phosphite with silyl halides.[3] Consequently, a four-centre mechanism was suggested, similar to the mechanism considered in analogous ligand exchange between some P(III) compounds.[6] However, the Arbuzov intermediate pathway remains attractive because of the generality of this concept in organophosphorus chemistry and we feel that the four-centre mechanism may be not an exceptional route in the discussed processes.

We thought that more light could be shed on the question of the Arbuzov intermediate in the ligand exchange between Si and P (tricoordinate) if we studied a model reaction involving species with a highly nucleophilic P(III) centre and a silicon reagent having a strongly nucleofugal group. Looking for such a model we found that $(Me_2N)_3P$ readily exchanges one amine group for halogen by the action of Me_3SiI or Me_3SiBr, and the course of this process may be conveniently followed by low temperature ^{31}P NMR.

This reaction seems also to provide a convenient synthetic route to $(Me_2N)_2PI$, which is a very useful and interesting chemical intermediate.

Finally, $(Me_2N)_2PI$ offers the possibility to extend the ligand exchange study to analogous reactions involving two P(III) species, processes which are of considerable importance in organophosphorus chemistry.

RESULTS AND DISCUSSION

The reaction of phosphorous hexamethyltriamide with trimethylsilyl halides leads to tetramethylphosphorodiamidous halide according to Eq. 1.

$$(Me_2N)_3P + Me_3SiX \rightleftharpoons (Me_2N)_2PX + Me_2NSiMe_3 \qquad (1)$$

$$X = Br, I$$

At ambient temperature reaction (1) occurs immediately in methylene chloride solution. At -50°C no formation of the phosphorodiamidous halide was observed; however, the ^{31}P NMR signals of $P(NMe_2)_3$ were drastically changed in result of the addition of trimethylsilyl halide, indicating a strong interaction of both species.

1. The interaction at temperatures below -50°

^{31}P NMR spectra as well as conductometric measurements prove the equilibrium of Eq. 2.

$$(Me_2N)_3P + Me_3SiX \rightleftharpoons [(Me_2N)_3\overset{+}{P}SiMe_3]X^- \qquad (2)$$

$$X = Br, I$$

The conductivity of the solution of the triamide in methylene chloride increases markedly with the addition of trimethylsilyl iodide up to a value typical for a strong electrolyte when one molar equivalent of the silane is introduced. Further addition of the silane does not change the conductivity. The shape of the conductometric curve (Fig. 1a) is characteristic of 1:1 stoichiometry of the complex and gives evidence that the equilibrium (2) lies well to the side of the complex.

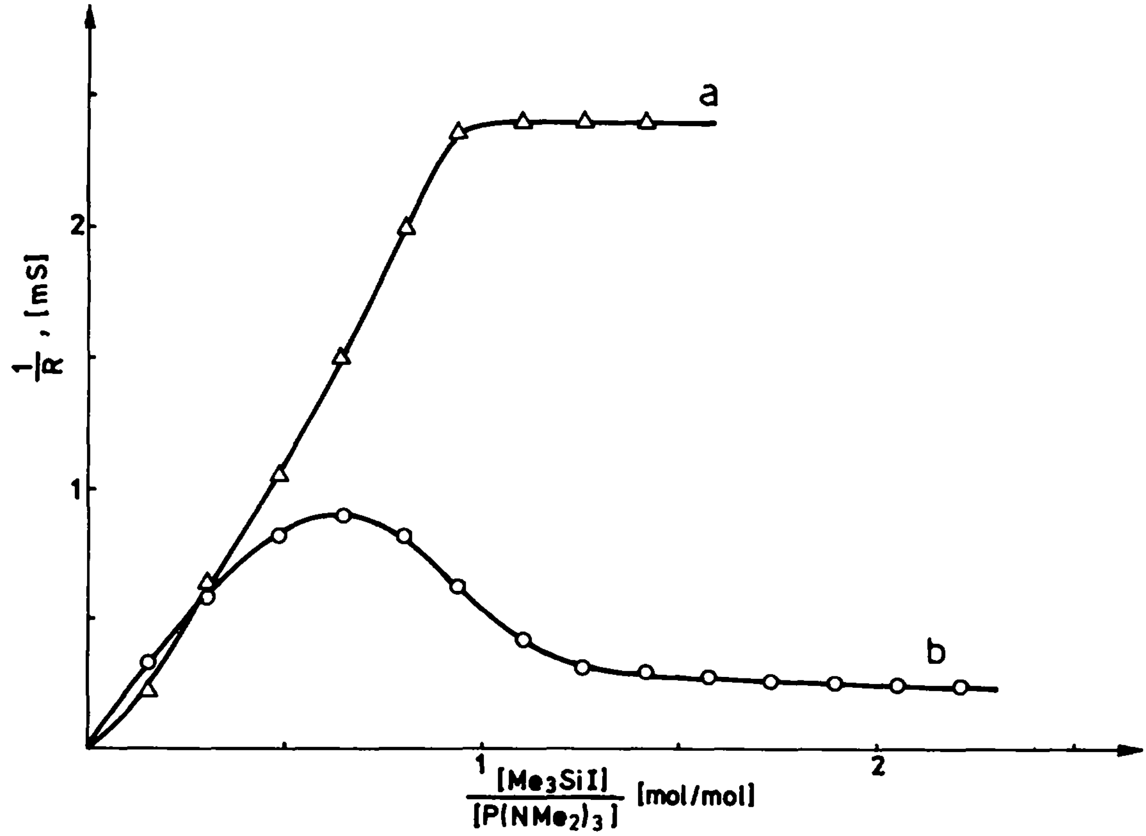
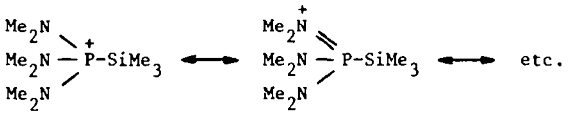


Figure 1. Conductometric titration of $(Me_2N)_3P$ with Me_3SiI in CH_2Cl_2, $[(Me_2N)_3P]_o = 0.165\ mol\cdot dm^{-3}$; a) at -50°C; b) at 25°C.

^{31}P NMR spectra made for the mixtures of substrates at different molar ratio of the amide to the silane confirm these conclusions. The spectrum of $(Me_2N)_3P + Me_3SiI$, 2:1 molar ratio shows two signals with the same intensity: 120 ppm (substrate) and 64 ppm (complex), while the spectrum of a mixture at 1:1 molar ratio shows only one signal at 64 ppm. Moreover, it is evident that the equilibrium (2) is slow in comparison to the NMR timescale. The signal of the complex is accompanied by the satellites arising from ^{31}P-^{29}Si spin-spin coupling, at the expected relative intensity. The possibility of these signals being accidental side-bands was excluded by recording the spectra of a series of experiments on two different spectrometers: Jeol JNM FX-60 (at 24.2 MHz) and Bruker AX-90 (at 36.3 MHz). In all cases both the relative intensity and the coupling constant remained the same. The relatively high value of the coupling constant (115 Hz) suggests direct P-Si bonding in the complex. The chemical shift observed for the complex corresponds well with the values reported for the analogous phosphonium salts $(R_2N)_3\overset{+}{P}R\ X^-$.[11]
Large chemical shift change due to the complex formation ($\Delta\delta$ = 56 ppm) and relatively high stability of the complex is interpreted in terms of contribution from mesomeric structures:



Low temperature ^{1}H NMR spectra of $P(NMe_2)_3 + Me_3SiI$ gave no further information about the structure of the complex. Below -50°C one broadened signal is observed in the range of the phosphoramide protons (2.6 ppm). The silyl protons appear at 0.55 ppm, while there is no free Me_3SiI (0.93 ppm) in the sample. This observation confirms that all the iodosilane is involved in the complex.

In the case where X = Br, low-temperature ^{31}P NMR spectra prove that the reaction takes an analogous course. The equilibrium (2), in contrast, lies on the side of the free substrates, although the signal of the complex is still present in the spectrum. Its chemical shift is the same as in the case where X = I. This difference in reactivity between bromosilane and iodosilane is connected with the higher energy of ionisation of the Si-Br bond compared with the Si-I bond.

2. The interaction above -50°C

When the temperature is slowly raised above -50°, the formation of $(Me_2N)_2PX$ is observed according to Eq. 1. The ^{31}P NMR signal of the complex disappears and the signal of the product or the signals of its complex with $(Me_2N)_3P$ (see next section) appear. The triamide is converted completely when X = I; however, the equilibrium constant is close to 1 when X = Br, and it is known to be much smaller than 1 for X = Cl. The reverse reaction has been used for the synthesis of some phosphorus triamides[12] (Eq. 3).

$$(Me_2N)_2PCl + R_2NSiMe_3 \longrightarrow (Me_2N)_2PNR_2 + Me_3SiCl \qquad (3)$$

Reaction (1) leads exclusively to the substitution of one amine group by halogen. The thermodynamics are unfavourable for the substitution of other groups since the reverse reaction readily occurs[13,14] (Eq. 4).

$$PI_3 + R_2NSiMe_3 \longrightarrow R_2NPI_2 + (R_2N)_2PI + Me_3SiI \qquad (4)$$

Reaction (1) is fast at room temperature for both the silyl iodide and bromide. A broadening of the ^{1}H and ^{31}P NMR signals is observed for $Me_3SiBr + P(NMe_2)_3$ as a result of the rapid reversibility of the reaction. The other reason for the broadening of the ^{31}P NMR signals is the interaction of $(Me_2N)_2PX$ with $P(NMe_2)_3$, which is discussed in the following section.

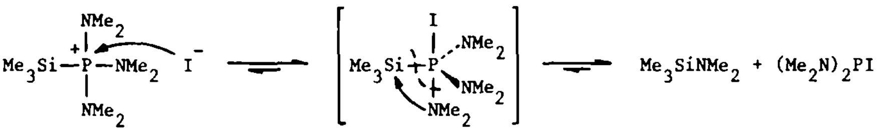
Reaction (1) is analogous to the reaction of a phosphite triester with Me_3SiI[3] (Eq. 5).

$$(RO)_3P + Me_3SiI \longrightarrow (RO)_2PI + Me_3SiOR \qquad (5)$$

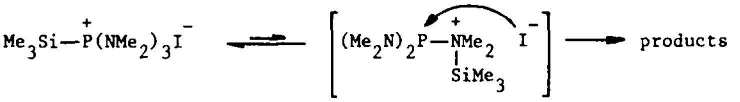
R = Alkyl, Ph

However, in that case we did not observe any P-Si interaction using low-temperature ^{31}P NMR spectroscopy. In the case of triamides the P-Si interaction is evident and it results from the much higher nucleophilicity of the triamides in comparison with that of the phosphites.

Thus we propose as the mechanism of formation of $(Me_2N)_2PI$ the nucleophilic attack of iodine on phosphorus in the complex (Eq. 6), although we cannot reject the stepwise mechanism (Eq. 7), in which the attack of iodine takes place at an ammonium-type complex existing at low concentration in the solution, undetected by ^{31}P NMR.



(6)



(7)

3. The interaction of product with substrate

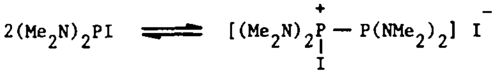
Reaction (1) is accompanied by the Arbuzov-type intermediate formation (Eq. 8). A similar adduct was observed in the reaction of $P(NMe_2)_3$ with $(Me_2N)_2P^+\ AlCl_4^-$.[15,16]

$$(Me_2N)_3P + (Me_2N)_2PI \rightleftharpoons [(Me_2N)_3\overset{+}{P}\text{-}P(NMe_2)_2]\ I^- \qquad (8)$$

The ^{31}P NMR spectrum of the products of reaction of $(Me_2N)_3P + Me_3SiI$, 2:1, cooled to -60°, shows two doublets: $\delta_1 = 85$ ppm, $\delta_2 = 48$ ppm, $J_{P-P} = 320$ Hz. At this temperature the equilibrium (8) lies well to the side of the complex. These doublets undergo coalescence with increase of temperature and only two broad signals are observed at ambient temperature: $\delta_1 = 150$ ppm, $\delta_2 = 90$ ppm, reflecting two features of the equilibrium (8): a shift of the equilibrium position towards the free components as the temperature is raised, and a high rate of formation and decomposition of the complex, leading to a short lifetime of all species involved in the equilibrium compared with the NMR time-scale. The thermodynamic parameters of the complex formation can readily be estimated from the position of the signals at different temperatures: $\Delta H = -10.7$ Kcal·mol^{-1}, $\Delta S = -33.8$ cal mol^{-1}deg^{-1}, equilibrium constant $K(25^oC) = 3$ dm^3 mol^{-1}, $K(-30^oC) = 180$ dm^3·mol^{-1}.

The same qualitative features of the ^{31}P NMR spectra are observed for the system $P(NMe_2)_3 + Me_3SiBr$, 1:1. The spectra of the complexes in the case of iodine and bromine are identical (Table).

The formation of the complex is also reflected in the conductometric titration curve (Fig. 1b). The conductivity of a 0.32 M solution of $(Me_2N)_2PI$ in the presence of an equimolar amount of $P(NMe_2)_3$, (1/R = 4 mS), is much higher than the conductivity of a 0.32 M solution of $(Me_2N)_2PI$ itself (1/R = 0.2 mS). The fairly high conductivity of pure $(Me_2N)_2PI$ in CH_2Cl_2 is probably due to the self-ionization reaction (Eq. 9).



$$2(Me_2N)_2PI \rightleftharpoons [(Me_2N)_2\underset{I}{\overset{+}{P}} - P(NMe_2)_2]\ I^- \qquad (9)$$

The conductometric titration curve illustrates the change of the concentration of $(Me_2N)_3\overset{+}{P}\text{-}P(NMe_2)_2I^-$. When the reaction of $(Me_2N)_3P$ with Me_3SiI is carried out at room temperature, $(Me_2N)_2PI$ is formed immediately. It interacts with the excess of $P(NMe_2)_3$ still present in the solution. Hence the maximum of the conductivity is reached when the $[(Me_2N)_3P]$ to (Me_3SiI) ratio is about 2:1 (i.e. $[(Me_2N)_3P]/[(Me_2N)_2PI] = 1:1$). Further titration results in decrease of conductivity until all the triamide is consumed and then stabilization of the conductivity is observed on the level of $(Me_2N)_2PI$.

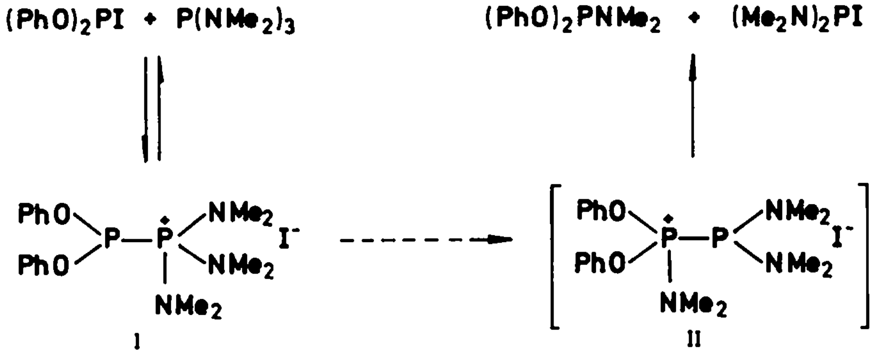
4. Arbuzov intermediate in a ligand exchange reaction between two tricoordinate phosphorus centres

We noticed that analogous interaction to those described in preceding section arose when the triamide was mixed at -60° with diphenyl iodophosphite (Eq. 10).

$$P(NMe_2)_3 + (PhO)_2PI \rightleftharpoons [(PhO)_2P\text{-}\overset{+}{P}(NMe)_3]\ I^- \qquad (10)$$

The ^{31}P NMR spectrum shows two doublets: δ_1 = 171 ppm [$(PhO)_2P-$] and δ_2 = 51 ppm [$(Me_2N)_3\overset{+}{P}-$], J_{P-P} = 210 Hz. Small signals of both substrates are also present, since the equilibrium is not totally shifted to the side of the complex, and the rate of complex formation and decomposition at this temperature is slow enough so that the lifetimes of all species of equilibrium (10) are longer than those of the spinstates of the P nuclei. The chemical shift of $(Me_2N)_3\overset{+}{P}-$ assumes approximately the same value as in the product formed according to equation (8) (see Table), which confirms the structure of the complex.

When the temperature is raised ligand exchange takes place according to Scheme



Scheme 1

The intermediate I may decompose directly, or more probably first undergoes the ligand migration step. It is reasonable that the intermediate II, if formed, must behave differently from I, decomposing almost irreversibly to components, because the nucleophilicity of the monoamide is low compared with that of the triamide.

Thus there is a close analogy in the mechanism of ligand exchange of the triamide with the phosphorus iodide to that with the silyl iodide. Both these systems involve the transient formation of an Arbuzov-type intermediate which decomposes stepwise or directly with the formation of the P(III) ligand exchange product.

These results demonstrate that an Arbuzov-type intermediate mechanism may operate in ligand exchange processes at tricoordinate phosphorus, although the scope of these reactions is not quite clear. It is evident that the high nucleophilicity of $P(NMe_2)_3$ plays an essential role in the formation of this intermediate. On the other hand, small concentrations of the intermediate formed by species of lower P(III) nucleophilicity, not detectable by ^{31}P NMR, may still be of kinetic significance.

Table. ^{31}P NMR data of some complexes of $P(NMe_2)_3$ with Si- and P(III)-halides

Compound	Temp.[°C]	δ substr.[ppm]	δ prod.[ppm]	$\Delta\delta$ [ppm]	J [Hz]
$(Me_2N)_3\overset{+}{P}SiMe_3I^-$	-60	120	64	-56	115 ($^{31}P-^{29}Si$)
$(Me_2N)_3\overset{+}{P}SiMe_3Br^-$	-60	120	64	-56	
$(Me_2N)_3\overset{+}{P}{}^1P^2(NMe_2)_2I^-$	-60	120(P^1) 208(P^2)	48(P^1) 85(P^2)	-72 -123	320 (P-P)
$(Me_2N)_3\overset{+}{P}{}^1P^2(NMe_2)_2Br^-$	-60	120(P^1) 172(P^2)^a	48(P^1) 85(P^2)	-72 -87	320 (P-P)
$(Me_2N)_3\overset{+}{P}{}^1P^2(OPh)_2I^-$	-60	120(P^1) 202(P^2)	51(P^1) 171(P^2)	-69 -31	210 (P-P)

[a] chemical shift of $(Et_2N)_2PBr^5$

5. Preparative aspects

Phosphorodiamidous iodides have been previously obtained by 3 methods:

(i) halogen exchange in phosphorodiamidous chlorides with magnesium iodide:[17]

$$(R_2N)_2PCl + MgI_2 \longrightarrow (R_2N)_2PI + MgICl$$

(ii) reaction of silylamines with PI_3, mentioned earlier in this paper:[13,14]

$$2R_2NSiMe_3 + PI_3 \longrightarrow (R_2N)_2PI + 2Me_3SiI$$

(iii) halogen exchange between phosphorodiamidous chlorides and trimethyliodosilane:[14]

$$(R_2N)_2PCl + Me_3SiI \longrightarrow (R_2N)_2PI + Me_3SiCl$$

The reaction (1) has some advantages as it is simple, starts from the available triamide, and leads to a high yield of the product (100% as measured by ^{31}P NMR). Tetramethylphosphorodiamidous iodide is a yellow liquid, but from some solvents, e.g. pentane, it forms a crystalline precipitate, presumably $(Me_2N)_2\overset{+}{P}(I)\text{-}P(NMe_2)_2I^-$. Physicochemical data of $(Me_2N)_2PI$: b.p. 50°/1 mmHg; ^1H NMR: δ = 2.65 ppm (d, $PNCH_3$), J_{PNCH} = 13 Hz; ^{31}P NMR see table; MS: m/e 202 $(Me_2N\overset{+}{P}I)$, 119 $[(Me_2N)_2\overset{+}{P}]$, 76 $(Me_2N\overset{+}{P}H)$, 60 $(Me_2N{=}\overset{+}{P})$, 44 $(Me_2\overset{+}{N})$; anal.: C 19.25 (calc. 19.5), H 5.8 (4.9), N 10.9 (11.3), P 12.0 (12.6), I 45.5 (51.6) % (some deviations from calculated values caused by the extreme sensivity of the product towards atmosphere moisture).

The iodoamide reacts vigorously with water giving a mixture of products produced by the cleavage of P-N bonds by the HI formed in the reaction. Hydrolysis in the presence of Et_3N leads to $(Me_2N)_2P(O)H$ with 100% yield. Reaction with secondary amines gives mixed triamides $(Me_2N)_2PNR_2$ identified by ^{31}P NMR: the diethylamino derivative was obtained in this way. The reaction with absolute ethanol in the presence of Et_3N gives $(Me_2N)_2POEt$ (identified by ^{31}P NMR). The complex of iodoamide with $AlCl_3$ in CH_2Cl_2 was observed in ^{31}P NMR, which gives broad signal at 253 ppm. This result is in accordance with the data for $(Me_2N)_2PCl\cdot AlCl_3$.[16]

EXPERIMENTAL

General

All manipulations, including NMR sample preparation, were carried out under an inert atmosphere of dry nitrogen. Methylene chloride was purified by shaking with oleum followed by distillation from $LiAlH_4$.

Phosphorous hexamethyltriamide was purified by distillation from CaH_2 under reduced pressure. Iodotrimethylsilane was distilled from aluminium powder directly before use. Diphenyl iodophosphite was obtained by a method described.[3]

NMR measurements

^{31}P NMR spectra were recorded on a Jeol JNM FX-60 FT spectrometer using sample tubes of 10 mm outside diameter. Chemical shifts are reported relative to external 85% H_3PO_4.

In low temperature experiments the tube containing the solution of $P(NMe_2)_3$ (0.50 mol dm^{-3}, 0.2 ml of $P(NMe_2)_3$ in 2 ml of CH_2Cl_2) closed with a rubber membrane was connected through a needle to a reservoir of dry nitrogen for levelling the pressure and it was frozen in liquid nitrogen. Then the appropriate amount of iodosilane was introduced with a microsyringe. The closed tube was warmed cautiously to about -100°C to give an uniform solution then put into the NMR probe.

^1H NMR spectra were recorded on a Bruker AX-90 FT spectrometer using sample tubes of 5 mm o.d. Chemical shifts are reported relative to TMS. C_6D_6 was used as the internal lock.

Samples for low temperature ^1H NMR measurements were prepared in a 3 ml flask connected to a NMR tube and closed with rubber membrane. The concentration of $P(NMe_2)_3$ was 0.25 mol dm^{-3} (0.1 ml of $P(NMe_2)_3$ in 1.5 ml CH_2Cl_2 + 0.5 ml C_6D_6). After freezing with liquid nitrogen and adding the iodosilane, the apparatus was warmed cautiously until the reaction mixture became homogeneous. Then the solution was placed in the cooled NMR tube which was sealed off and put into the probe.

Conductometric measurements

Conductometric measurements were carried out with a Radelkis conductometer OK 102/1 equipped with a conductivity call OK-902.

Preparative experiments

Preparation of N,N,N',N'-tetramethylphosphorodiamidous iodide - Phosphorous hexamethyltriamide (4.5 g, 0.0275 mol) in CH_2Cl_2 (15 ml) were placed in 50 ml round-bottom flask equipped with a magnetic stirrer and reflux condenser. Iodotrimethylsilane (5.5 g, 0.0275 mol) was added with a syringe equipped with teflon needle through the reflux condenser. The flask was then connected for a vacuum distillation. After evaporating off the solvent and dimethyl(trimethylsilyl)amine, the product was distilled giving 4 g (60%) of $(Me_2N)_2PI$ (b.p. 50°/1 mmHg).

Reaction of $(Me_2N)_2PI$ with diethylamine - N,N,N',N'-tetramethylphosphorodiamidous iodide (0.50 g, 0.002 mol) in CH_2Cl_2 (5 ml) were placed in a 15 ml round-bottom flask. Diethylamine (0.29 g, 0.004 mol) was added by syringe. The ^{31}P NMR spectrum made after filtering off the amine hydroiodide showed a signal at 120.5 ppm (100%) which corresponded well to the reported values for $(Me_2N)_2PNEt_2$.[11] Distillation resulted in disproportionation of the product.

Reaction of $(Me_2N)_2PI$ with ethanol - To a solution of $(Me_2N)_2PI$ (0.50 g, 0.002 mol) and triethylamine (0.20 g, 0.002 mol) in CH_2Cl_2 (5 ml) anhydrous ethanol (0.092 g, 0.002 mol) was introduced. ^{31}P NMR spectrum made after filtering off the amine hydroiodide showed signals at 137.5 ppm [$EtOP(NMe_2)_2$, 70%], 143.7 ppm [$(EtO)_2PNMe_2$, 20%] and 22.8 ppm [$(Me_2N)_2P(O)H$, 10%]. On distillation the product underwent disproportionation to amidophosphites $(Me_2N)_nP(OEt)_{3-n}$.

Reaction of $(Me_2N)_2PI$ with water - Water (0.036 g, 0.002 mol) was added by microsyringe to a solution of $(Me_2N)_2PI$ (0.50 g, 0.002 mol) and Et_3N (0.20 g, 0.002 mol) in 5 ml of dioxan. The ammonium iodide was filtered off and the ^{31}P NMR spectrum of the residue showed a signal at 22.4 ppm (100%), J_{PH} = 581 Hz, corresponding to $(Me_2N)_2P(O)H$.[11]

Interaction of $(Me_2N)_2PI$ with $AlCl_3$ - $(Me_2N)_2PI$ (0.2 g, 0.8 mmol) was dissolved in CH_2Cl_2 (2 ml) in a 10 mm o.d. NMR tube. Freshly-sublimed $AlCl_3$ (0.12 g, 1 mmol) was added under a stream of nitrogen. The $AlCl_3$ easily dissolved. The ^{31}P NMR spectrum showed a broad signal at 253 ppm (ca 60%) which corresponds to the chemical shift of the acid-base adduct and two unidentified signals at 216 ppm (30%) and 24 ppm (10%).

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