THE MECHANISM OF THE REACTION OF ORGANIC PHOSPHITES WITH TRIALKYLSILYL IODIDE. IODOANHYDRIDES OF P^{III} ACIDS AS INTERMEDIATES

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Summary

The reaction of a trialkyl phosphite with trimethylsilyl iodide, which leads to *O*-trimethylsilyl esters of alkylphosphonic acid, has been shown to involve several steps, all of which are defined. The first step is the formation of the iodo-phosphite, involving a four centre mechanism in which P^{III} plays the role of electrophile. This is in contrast to the analogous reaction of phosphites with alkyl halides (the Arbuzov reaction) which begins with nucleophilic attack of the phosphorus.
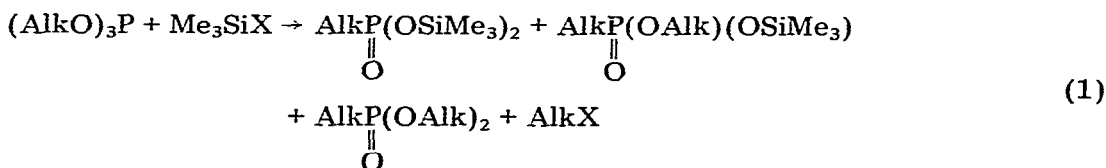
Introduction

Arbuzov reactions between alkyl halides and oxy-esters of tricoordinated phosphorus are among the commonest processes in organophosphorus chemistry and their mechanism, involving the formation of a phosphonium ion intermediate followed by its dealkylation with the counter-ion, serves as a model for a large number of reactions of esters of tricoordinated phosphorus with electrophilic reagents. It was thus natural that some of the earliest studies in silicon-phosphoroorganic chemistry were directed towards analogues of the Arbuzov reaction, i.e. the reaction between phosphites and trimethylsilyl halides [1,2]. However, contrary to an earlier report by Arbuzov and Pudovik [1], Malatesta found that the reaction of triethyl phosphite with triethylsilyl halides did not follow from the Arbuzov route [2], and gave the *O*-triethylsilyl ester of ethylphosphonic acid instead of the expected ester of silylphosphonic acid. Two alternative mechanisms were proposed; one involved an Arbuzov isomerization of triethyl phosphite to the ethylphosphonate followed by introduction of the silyl group in place of the ethyl ester group, and the other involved the introduction of silyl in place of ethyl in the phosphite followed by Arbuzov rearrangement induced by the ethyl halide formed in the first step. The second mechanism received strong support from the observation that com-

pounds of the general formula $P(OEt)_{3-n}(OSiEt_3)_n$, $n = 1, 2, 3$, were formed as final products when triethylsilyl bromide was treated with triethyl phosphite under conditions in which EtBr was rapidly removed from the system [3].

We recently observed that the reaction between alkyl phosphites and trimethyl silyl iodide, though leading to O-silyl esters of alkylphosphonic acid, involved various intermediates stable enough at lower temperatures to be identified. We thought that studies of these intermediates would give a deeper insight into the mechanism of the reaction of silyl halides with oxy-esters of tricoordinated phosphorus. Elucidation of this mechanism might extend the scope of the synthetic applications of silyl halides, in particular of trimethylsilyl iodide, in the chemistry of phosphorus.

Results and discussion

The reaction of trialkyl phosphites with trimethylsilyl halides gives phosphonates according to eq. 1.

$$(AlkO)_3P + Me_3SiX \rightarrow Alk\overset{\parallel}{\underset{O}{P}}(OSiMe_3)_2 + Alk\overset{\parallel}{\underset{O}{P}}(OAlk)(OSiMe_3)$$

$$+ Alk\overset{\parallel}{\underset{O}{P}}(OAlk)_2 + AlkX \tag{1}$$

$X = Br, I$

Study of the reaction of Me_3SiBr and Me_3SiI with trimethyl phosphite over a large temperature range by means of ^{31}P NMR spectroscopy revealed the presence of P^{III} ester intermediates. Two separate stages can be distinguished in the reaction of Me_3SiI. In the first, which can be conveniently followed in the temperature range -20 to $0°C$, only products of tricoordinated phosphorus are formed. The second stage, which involves transformation of these P^{III} intermediates to tetracoordinated phosphorus products, does not occur at a significant rate in this temperature range.

Formation of P^{III} intermediates

The reaction of trimethyl phosphite (Ia) with trimethylsilyl iodide (IIa) in methylene chloride at $-10°C$ was monitored by ^{31}P NMR spectroscopy, with the results as described below. Initially, there is almost exclusive formation of dimethyl iodophosphite according to eq. 2:

$$P(OMe)_3 + Me_3SiI \rightarrow (MeO)_2PI + Me_3SiOMe \tag{2}$$

$\quad$ (Ia) $\qquad$ (IIa) $\qquad$ (IIIa) $\qquad$ (IV)
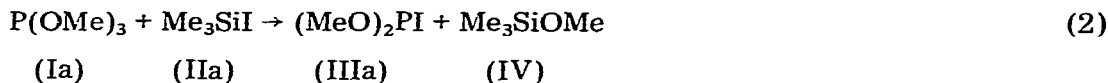
Although iodophosphites are known compounds [4,5], IIIa cannot be isolated from this system. It was identified by its ^{31}P NMR signal, showing the characteristic splitting pattern caused by $P^{31}OCH^1$ spin—spin coupling (Table 1), and also by trapping with diethylamine, in which IIIa was converted almost quantitatively to dimethyl N,N-diethylphosphoramidite, according to eq. 3 (as shown by the ^{31}P NMR spectrum).
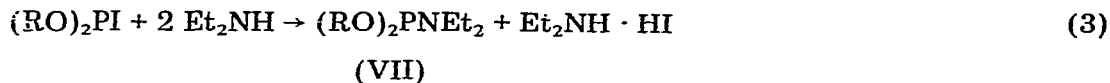
$$(RO)_2PI + 2 Et_2NH \rightarrow (RO)_2PNEt_2 + Et_2NH \cdot HI \tag{3}$$

$\qquad\qquad$ (VII)

TABLE 1

^{31}P NMR DATA

Compound	No.	δ (ppm)	J(POCH) (Hz)
(MeO)$_2$POSiMe$_3$	Ib	126.7 (sept)	11
MeOP(OSiMe$_3$)$_2$	Ic	115.9 (q)	10
P(OSiMe$_3$)$_3$	Id	113.2 (s)	—
(MeO)$_2$PI	IIIa	213.8 (sept)	10
MeO(Me$_3$SiO)PI	IIIb	192.7 (q)	10
(Me$_3$SiO)$_2$PI	IIIc	190.1 (s)	—
(PhO)$_2$POMe	Vc	127.7 (q)	8
(PhO)$_2$P(OSiMe$_3$)	Vb	123.3 (s)	—
(PhO)$_2$PI	VIa	201.2 (s)	—
(PhO)$_2$PBr	VIb	174.6 (s)	—

Following these observations, a compound of series VII, having R = Ph, was synthesized and isolated.

The concentration of the iodophosphite IIIa in the reaction system of Ia + IIa increases rapidly and reaches a maximum at about 50% of the initial concentration of phosphite when 1 : 1 molar substrate ratio is used and then slowly falls (Fig. 1). However, a large amount of the substrate Ia is present, along with trace amounts of other products at the point of maximum concentration of IIIa, which suggests that reaction 2 leads to an equilibrium lying roughly midway between substrates Ia, IIa and products IIIa and IV. It is noteworthy that reactions which are the reverse of those in eq. 2 were found to occur for the chloro- and bromo-analogues of IIIa [6].

Subsequently bismethyl(trimethylsilyl) phosphite (Ib) appears. The rate of its formation increases as reaction 2 proceeds (see Fig. 1), indicating that it must be produced in a subsequent reaction. As the process continues the sequence of consecutive reactions leads to two series of compounds: (MeO)$_{2-n}$-(Me$_3$SiO)$_n$PI, n = 0, 1, 2, (IIIa, b, c) and (MeO)$_{3-m}$(Me$_3$SiO)$_m$P, m = 0, 1, 2, 3, (Ia, b, c, d); the other products of this reaction are believed to be Me$_3$SiOMe
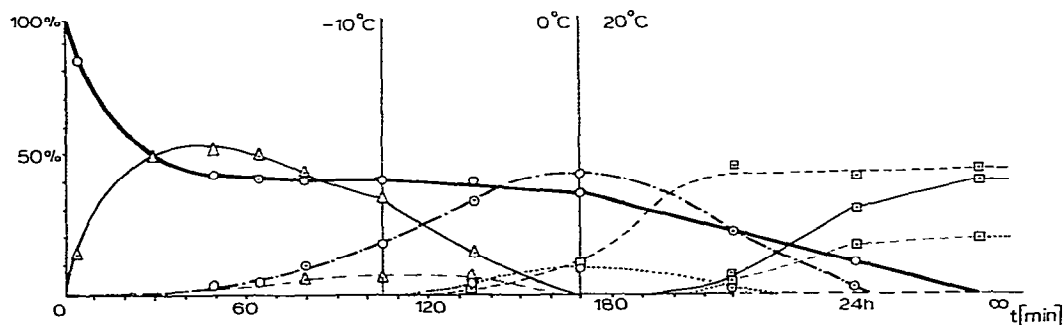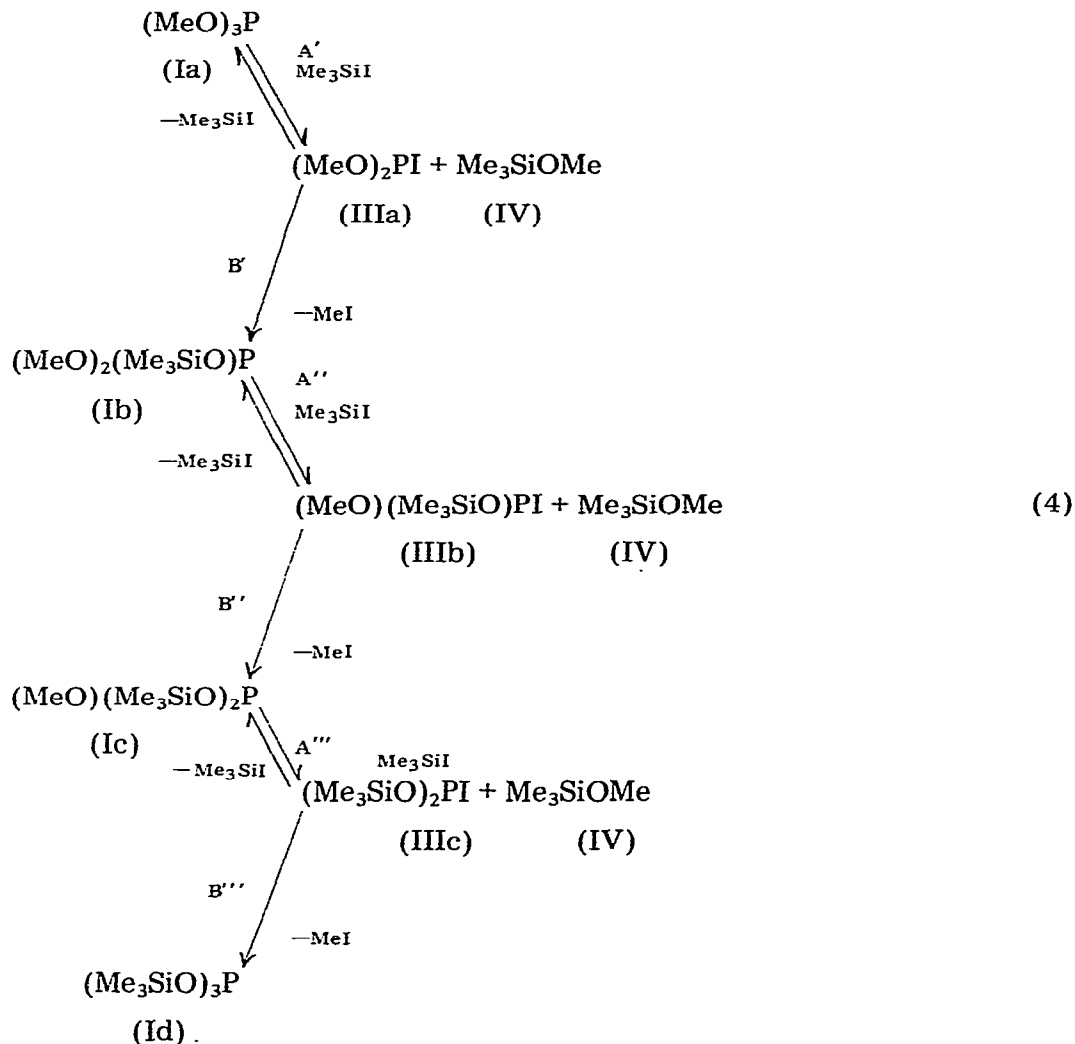

Fig. 1. The course of the reaction of trimethyl phosphite with trimethylsilyl iodide in methylene chloride as monitored by ^{31}P NMR spectroscopy. The initial concentrations were: (MeO)$_3$P 1.25 mol dm^{-3}; Me$_3$SiI 1.25 mol dm^{-3}. The reaction mixture was kept for 105 min at −10°C, then 65 min at 0°C, and was finally raised to 20°C.

△, (MeO)$_{2-n}$(Me$_3$SiO)$_n$PI; o, (MeO)$_{3-n}$(Me$_3$SiO)$_n$P; ———, n = 0; ·—·—·, n = 1; · · · · · ·, n = 2; o o o o, n = 3.
◻, (MeO)$_{2-n}$(Me$_3$SiO)$_n$PMe; ———, n = 0; · · · · · ·, n = 1; ------, n = 2.

(IV) and MeI. The addition of a large excess of IV to the system greatly reduces the concentration of products III and causes a corresponding increase in the concentration of compounds I, which provides evidence that compounds Ia—Id are formed from IIIa—IIIc and IV.

Bismethyl(trimethylsilyl) phosphite (Ib) was also obtained by an independent route and its reaction with trimethylsilyl iodide was studied. In the early stages of the process the main product was IIIb, but Ic subsequently appeared and this was followed by the completion of the cycle with the appearance of IIIc and IV. The main course of reaction between Ia and IIa at temperatures below $-10°C$ may thus be depicted as in the following scheme:

$$(MeO)_3P$$

(Ia)   A′   Me$_3$SiI

$-Me_3SiI$

$$(MeO)_2PI + Me_3SiOMe$$

(IIIa)   (IV)

B′ $-MeI$

$$(MeO)_2(Me_3SiO)P$$

(Ib)   A″   Me$_3$SiI

$-Me_3SiI$

$$(MeO)(Me_3SiO)PI + Me_3SiOMe$$ (4)

(IIIb)   (IV)

B″ $-MeI$

$$(MeO)(Me_3SiO)_2P$$

(Ic)   A‴   Me$_3$SiI

$-Me_3SiI$

$$(Me_3SiO)_2PI + Me_3SiOMe$$

(IIIc)   (IV)

B‴ $-MeI$

$$(Me_3SiO)_3P$$

(Id) .

The results shown in Figs. 1, 2 and 3 indicate that in addition to the reactions depicted in eq. 4 there must be other processes. Although the reactions designated B were shown to proceed practically irreversibly, small amounts of Ia and IIIa were observed in the reaction between Ib and IIa (Fig. 3), probably
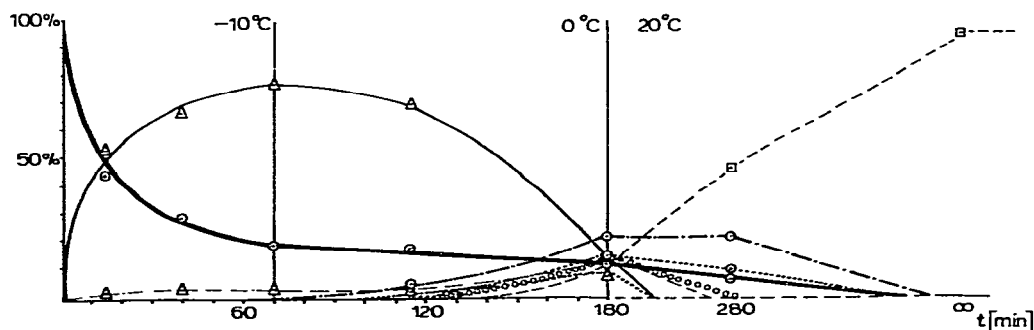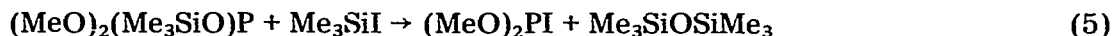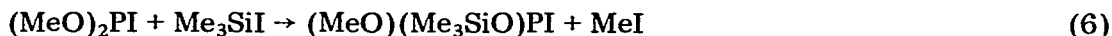
Fig. 2. The course of the reaction of trimethyl phosphite with an excess of trimethylsilyl iodide in methylene chloride as monitored by ^{31}P NMR spectroscopy. The initial concentrations were: $(MeO)_3P$ 1.05 mol dm^{-3}; Me_3SiI 2.1 mol dm^{-3}. The temperature was —10°C for the first 70 min, 0°C for a further 110 min, and was finally raised to 20°C. For an explanation of the symbols see Fig. 1.

as a result of the reaction shown in eq. 5.

$$(MeO)_2(Me_3SiO)P + Me_3SiI \rightarrow (MeO)_2PI + Me_3SiOSiMe_3 \tag{5}$$

The direct formation of IIIb from IIIa and IIa according to eq. 6 also accounts for the observation that small amounts of IIIb is formed in advance of the formation of Ib in the presence of a large excess of silyl iodide (see Fig. 2).

$$(MeO)_2PI + Me_3SiI \rightarrow (MeO)(Me_3SiO)PI + MeI \tag{6}$$

To verify reaction 4 and to obtain information on the mechanisms of the component processes we also studied the reaction between IIa and triphenyl phosphite. The reaction occurs in a much simpler fashion as only the first step,
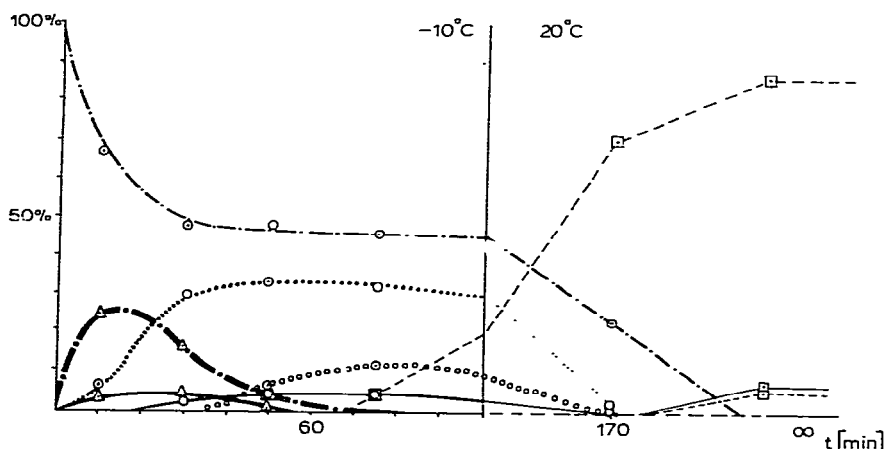

Fig. 3. The course of the reaction of bismethyl(trimethylsilyl) phosphite with trimethylsilyl iodide in methylene chloride as followed by ^{31}P NMR spectroscopy. The initial concentrations were: $(MeO)_2$-POSiMe$_3$ 1.06 mol dm^{-3}; Me_3SiI 1.06 mol dm^{-3}. The temperature was —10°C for the first 100 min and was then raised to 20°C. For an explanation of the symbols see Fig. 1.

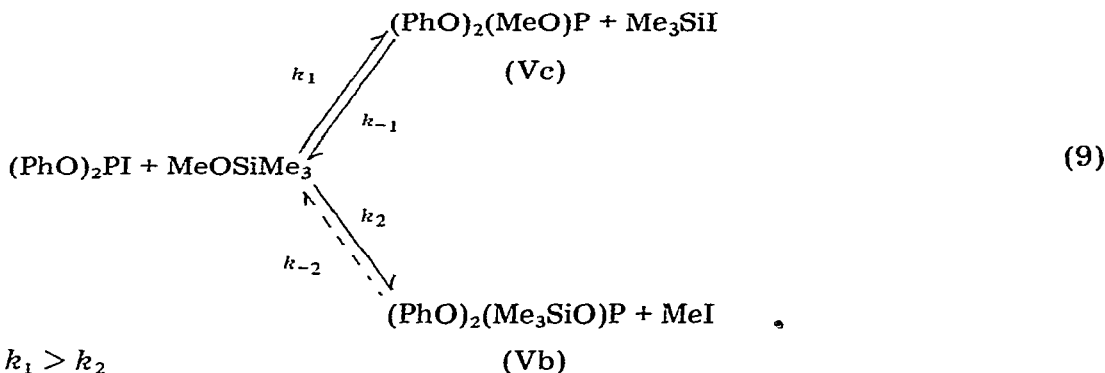i.e. the analogue of reaction A', takes place, leading to equilibrium 7.

$$(PhO)_3P + Me_3SiI \overset{K_1}{\rightleftharpoons} (PhO)_2PI + Me_3SiOPh \tag{7}$$

   (Va)   (VIa)
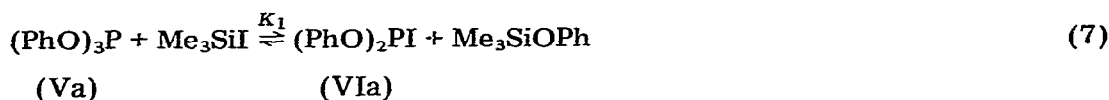
No reaction involving $C(Ar)$—O bond cleavage was observed, and so the silyl esters of phosphorus cannot be formed by route B (eq. 4) nor by a reaction analogous to that depicted in eq. 6.

Diphenyl iodophosphite was used as a model reactant in investigating the reaction of iodophosphites with trimethylmethoxysilane and was obtained from the reaction of diphenyl chlorophosphite with trimethyliodosilane (eq. 8).

$$(PhO)_2PCl + Me_3SiI \rightarrow (PhO)_2PI + Me_3SiCl \tag{8}$$

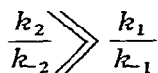(VIc)   (VIa)
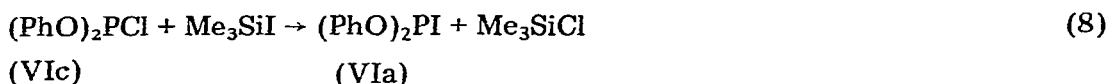
The reaction proceeds rapidly at ambient temperatures and gives the iodophosphite in almost 100% yield. Trimethylsilyl chloride may be removed by vacuum distillation. The bromophosphite (VIb) was obtained similarly from trimethylsilyl bromide, and this type of reaction may offer a satisfactory alternative to those used for the synthesis of reactive halogenophosphites [5,6]. Diphenyl iodophosphite is only moderately stable at ambient temperatures, since it slowly disproportionates.

The course of the reaction between $(PhO)_2PI$ and trimethylmethoxysilane is depicted in Fig. 4. The results are in full agreement with the mechanism presented in eq. 4. The sequence leads to two products, diphenylmethyl phosphite and bisphenyl(trimethylsilyl) phosphite as shown in eq. 9. The former is formed more rapidly, and it is initially produced in a considerable excess as the kinetic product. However, the proportion of the products is reversed as the reaction proceeds the silyl ester and methyl iodide being thermodynamically preferred to the methyl ester and silyl iodide.

$$(PhO)_2PI + MeOSiMe_3 \quad \begin{array}{c} \overset{k_1}{\underset{k_{-1}}{\rightleftharpoons}} (PhO)_2(MeO)P + Me_3SiI \quad (Vc) \\ \\ \overset{k_2}{\underset{k_{-2}}{\rightleftharpoons}} (PhO)_2(Me_3SiO)P + MeI \quad (Vb) \end{array} \tag{9}$$

$$k_1 > k_2$$

$$\frac{k_2}{k_{-2}} \gg \frac{k_1}{k_{-1}}$$

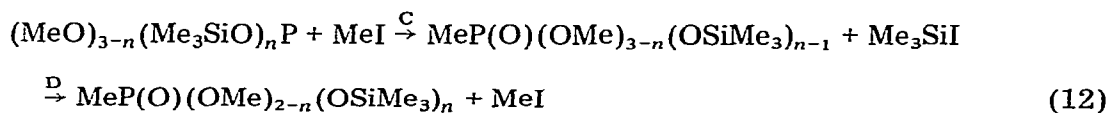*The formation mechanism of P^{III} intermediates*

It is appropriate at this point to consider the mechanism of the replacement of the methoxy group by iodine, and of the conversion of the iodophosphite to

Fig. 4. The course of the reaction of diphenyl iodophosphite with trimethylmethoxysilane in methylene chloride at $-10°$C. The initial concentrations were: $(PhO)_2PI$, 0.82 mol dm^{-3} and Me_3SiOMe, 0.82 mol dm^{-3}. a, $(PhO)_2PI$; b, $(PhO)_2POMe$; c, $(PhO)_2POSiMe_3$; d, $MeP(OPh)_2$.

the silyl phosphite. Two reaction paths can be considered, one involving nucleophilic substitution at 3-coordinate phosphorus (eq. 10) and the other involving nucleophilic attack of phosphorus on silicon to give an intermediate having a higher coordination number at phosphorus (eq. 11).

(10)

(11)

The route shown in eq. 11 seems attractive since it includes a fragment of the Arbuzov-type mechanism common in phosphorus chemistry. Moreover, the interaction of tricoordinated phosphorus with trimethyliodosilane can lead to the formation of a phosphonium salt [7]. However, this mechanism is not consistent with results shown in Figs. 1—4 and must be rejected. Since OR^- is a much poorer leaving group than I^-, the phosphorane 1 should react to form the

phosphonium ion 3 or to re-form the original reactants. Formation of the phosphonium salt 2 could eventually take place in a rate-limiting step (r.l.s.) of the process to give IIIa, while dealkylation of 3 would be rate-limiting in the formation of Ib. Compounds IIIa and Ib are formed via the same intermediate, which is produced from the substrates in a fast preequilibrium step and is present at very low concentrations (the intermediates 1, 2 and 3 were not detected). The rates of formation of IIIa and Ib would be proportional to the stationary concentration of this intermediate, which is highest in the beginning of the process. Thus the observed acceleration in the formation of Ib (Fig. 1) is not explained. On the basis of mechanism 11 it is also difficult to account for the reverse process from IIIa to Ia, as it would require the ionization of the $\equiv$Si—OR bond of low polarizability as a result of the attack of a soft nucleophile on the hard silicon electrophilic centre. Thus an alternative four-centre mechanism, 10, is postulated. Although the P^{III} atom is a strong nucleophilic centre, stronger than the ester oxygen, the latter is harder and thus more silicophilic. Thus a mechanism which involves nucleophilic attack of the oxygen at silicon assisted by nucleophilic attack of iodine to phosphorus seems feasible. O-Trimethylsilyl phosphite Ib is formed as a result of a subsequent secondary reaction between the iodophosphite IIIa and trimethylmethoxysilane IV involving an analogous four-centre mechanism. Both, IIIa and IV arise from the reaction between Ia and IIa.

The formation of phosphonates

Phosphonate products $MeP(O)(OMe)_{2-n}(OSiMe_3)_n$ are not formed at temperatures below $-10°C$ *. If the reaction mixture is kept for a long time at this temperature, the main products are compounds of series I and methyl iodide. The amounts of the thermodynamically less stable Me_3SiI, $MeOSiMe_3$ and compounds of series III are low. There is a tendency towards formation of P^{III} esters with a maximum number of the silyl groups.

When the temperature is raised to $0-20°C$, a slow but complete conversion of P^{III} compounds to the phosphonates is observed. Therefore, the products in eq. 4 necessarily lie on the reaction path leading to the phosphonates, and are true intermediates in the process studied. This conversion must include two processes, which are known to occur under these conditions. The first is a classical Arbuzov reaction [8] of methyl iodide with phosphites, and the second the reaction between iodosilane and phosphonate involving reintroduction of a silyl group in place of an alkyl group in the ester (eq. 12).

$$(MeO)_{3-n}(Me_3SiO)_nP + MeI \xrightarrow{C} MeP(O)(OMe)_{3-n}(OSiMe_3)_{n-1} + Me_3SiI$$

$$\xrightarrow{D} MeP(O)(OMe)_{2-n}(OSiMe_3)_n + MeI \qquad (12)$$

$n = 1, 2, 3$

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* The reactions involving the highly nucleophilic $P(OSiMe_3)_3$ may constitute an exception to this rule.

If $n = 0$ simple Arbuzov isomerization occurs

$$P(OMe)_3 \xrightarrow{MeI} MeP(O)(OMe)_2$$

(VIIIa)

Results previously reported from this laboratory [9] show that the Arbuzov reaction with a mixed alkyl silyl phosphite involves desilylation with the formation of silyl halides, and not dealkylation. The sequence of decreasing rates in the four consecutive reactions in the overall process is D > A > B > C, i.e. the Arbuzov reaction (C) is the slowest step.

The subsequent introduction of the silyl group in place of an alkyl group in the phosphite is expected to increase the nucleophilicity of the P^{III} centre, and so the reactivity in the Arbuzov reaction should increase in the following sequence.

$$P(OR)_3 < P(OR)_2(OSiMe_3) < P(OR)(OSiMe_3)_2 < P(OSiMe_3)_3$$

This sequence is confirmed by the course the reaction takes when the temperature is increased to 20°C, as set out in Figs. 1—3.

Since the rates of both steps A and C increase with an increasing degree of replacement of the alkyl by the silyl groups, the process initially gives the bis-(trimethylsilyl) ester of the phosphonic acid. This is virtually the only product when the initial ratio of silane to phosphite is higher than 2. At a smaller ratio the bissilyl ester is formed initially, the amount depending on the amount of ≡Si—I added. In the absence of Me$_3$SiI the remaining unchanged trialkyl phosphite is transformed into the dialkyl ester of the alkylphosphonic acid by reaction with the MeI liberated in the first step of the reaction. Thus the mixed silyl alkyl ester (VIIIb) is always formed in poor yields. The same thing is observed during the reaction of trimethylsilyl bromide with trimethyl phosphite and we believe that this process has an analogous mechanism.

Experimental

Substrates and solvent

Trimethyl, triethyl and triphenyl phosphites were of reagent grade and were purified by distillation from sodium. Diphenyl chlorophosphite was synthesized by literature procedures [10]. Trimethylsilyl iodide was prepared from hexamethyldisiloxane, iodide and aluminum as described by Jung [11]. Trimethylsilyl bromide was made analogously. Trimethylmethoxysilane was made by a standard method [12] and was dried over and distilled from CaH$_2$.Bismethyl-(trimethylsilyl) phosphite was prepared by the reaction of dimethyl phosphite with Me$_3$SiCl in the presence of Et$_3$N [13].

Methylene chloride was carefully dried with LiAlH$_4$ and distilled.

Studies of the reactions of trimethylsilyl iodide (IIa) with trimethyl phosphite (Ia)

The reaction of Ia with IIa was carried out in a 10 mm cross-section glass NMR tube under an atmosphere of dry nitrogen with precautions to avoid any contact with the atmosphere. First, the tube was flushed with dry nitrogen intro-

duced through a drawn out tube. The nitrogen was slowly passed through the NMR tube while it was charged with 1 ml of methylene chloride: 0.24 g (0.002 mol) of Ia and the appropriate amount of IIa by means of gas-tight Hamilton syringe equipped with a long Teflon needle. The tube was cooled in a dry ice/acetone bath. The tube was tightly closed with a stopper and placed in the thermostated ^{31}P probe of a Jeol JNM FX-60 spectrometer equipped with Fourier-transform accessories, and the reaction was monitored by NMR. The solutions for studies of the reaction of IIa with bismethyl(trimethylsilyl) phosphite and the reaction of diphenyl iodophosphite with trimethylmethoxysilane were prepared analogously.

The signals of phosphites $(MeO)_n P(OSiMe_3)_{3-n}$, $n = 0$—3, were identified by the known chemical shifts of compounds with $n = 0, 1, 3$, and their characteristic splittings due to $^{31}POC^1H$ spin—spin coupling. The signals of iodophosphites $IP(OMe)_{2-n}(OSiMe_3)_n$, $n = 0$—2, were identified from the known chemical shift of the compound $n = 0$ [4] and splitting due to $^{31}POC^1H$ coupling by assuming that the variation on introduction of consecutive silyl groups in place of alkyl groups would be the same as those in series I.

Synthesis of diphenyl iodophosphite (VIa)

A solution of diphenyl chlorophosphite (VIc) (3.3 g, 0.013 mol) in methylene chloride (10 ml), was contained in a vessel with a reflux condenser and was protected from atmospheric moisture. Trimethylsilyl iodide (2.6 g, 0.013 mol) was added dropwise with stirring. The ^{31}P NMR spectrum taken immediately after the addition showed that VIc was wholly transformed into a single phosphorus-containing product. Methylene chloride and IIc were removed under vacuum to leave a viscous liquid coloured by traces of iodine. No substantial change of the ^{31}P NMR spectra was found when the product was kept for several hours, but, after a day considerable amounts of disproportionation products and some unidentified products appeared.

Synthesis of O,O-diphenyl-N,N-diethylphosphoroamidite (VIIb). Identification of the product of reaction of phosphites with trimethylsilyl iodide

To a solution of triphenyl phosphite Va (4.1 g, 0.013 mol) in 10 ml of methylene chloride contained in the apparatus used for the synthesis of VIa, trimethylsilyl iodide IIa (4 g, 0.02 mol) was added dropwise during 5 min. The mixture was stirred for another 20 minutes, then the glass inlet tube was inserted and purified nitrogen was passed through the apparatus while it was cooled to −20°C. A solution of diethylamine (3 g, 0.04 mol) in 5 ml of methylene chloride was then added dropwise. The precipitate was filtered off, and after distillation of the volatile components the filtrate was subjected to vacuum distillation. 3 g of VIIb (b.p. 120°C/0.05 mmHg) was obtained (yield 80%). The compound had the same properties as those obtained by Fluck [14]. In order to determine the ^{31}P NMR signal of $IP(OMe)_2$, Ia was treated with a stoichiometric amount of IIa in a 10 mm cross-section NMR tube at −20°C. After 1 hour the ^{31}P NMR spectrum was taken, and showed, in addition to the signal from Ia, a signal at 213 ppm which was attributed to $IP(OMe)_2$ (IIIa). When an excess of Et_2NH was introduced, the signal at 213 ppm entirely disappeared while the signal from Ia remained. The main product under these con-

ditions showed a signal at 148 ppm which corresponds well with known values for dialkyl phosphoramidites $(RO)_2PNR'_2$ [15].

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