Phosphorylation of *N*-trimethylsilyllactams

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Treatment of *N*-trimethylsilyllactams with phosphoryl chlorides results in mixtures of products, whose formation can be explained by competition between *N*- and *O*-phosphorylation.

Key words: *N*-trimethylsilyllactams, dual reactivity; *N*-phosphoryllactams, synthesis.

Silylated lactams have been successfully used in the synthesis of *N*-substituted lactams.[1] Some of them, *e.g.*, *N*-acyl- and *N*-sulfonyllactams, are used as anti-convulsants and stimulants of the central nervous system.[2–5] The properties of *N*-phosphoryllactams have not been studied until now due to the poor availability of these compounds.

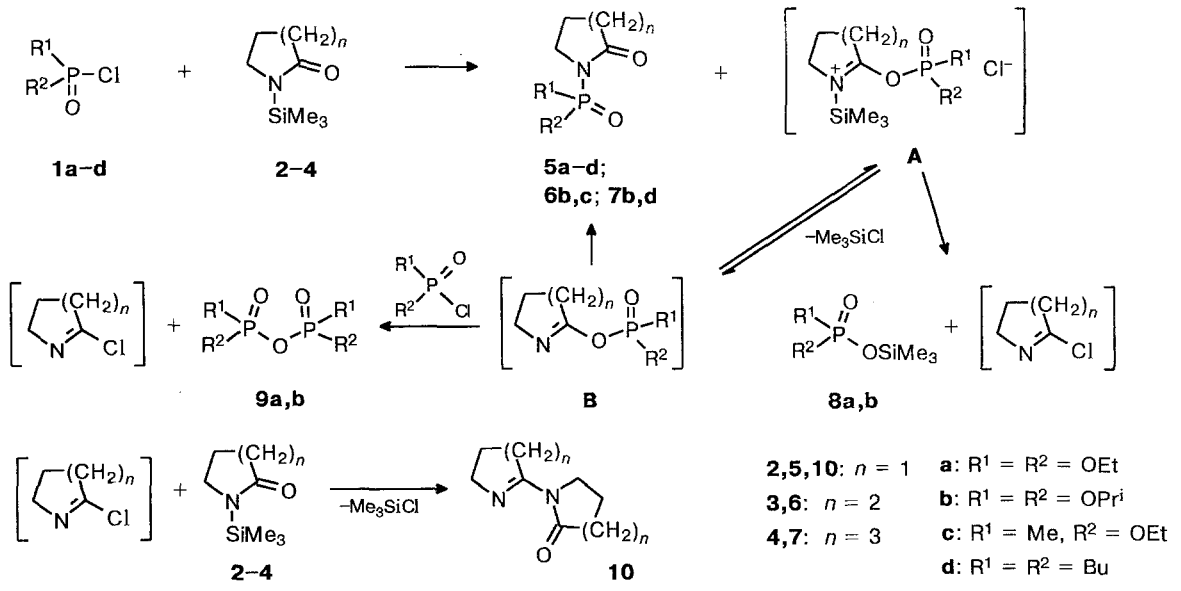
It has been reported previously[6] that *N*-phosphoryllactams can be obtained by treatment of alkyl methyl chlorophosphonates with *N*-trimethylsilyl(*N*-TMS-)-pyrrolidone and *N*-TMS-perhydro-2-azepinone under drastic conditions. In this case, the yields of *N*-phosphoryllactams were relatively low, and the composition of by-products was not studied.

We have recently shown that phosphoryl chlorides react with *N,O*-bis-TMS-amino acids under mild conditions to give the corresponding *N*-phosphorylated amino acids in almost quantitative yields.[7]

In the present work we studied the reaction of phosphoryl chlorides with *N*-TMS-lactams in order to develop a general method for the synthesis of *N*-phosphoryllactams (Scheme 1).

As in the case of silylated amino acids,[7] phosphorylation of *N*-TMS-lactams was carried out in a polar aprotic solvent, acetonitrile, which considerably accelerates the reaction and makes it possible to decrease the reaction temperature to 80—85 °C (compared to 100—130 °C in toluene or without a solvent as reported previously[6]). Simultaneous distillation to remove the chlorotrimethylsilane formed in the reaction increased the yield of *N*-phosphoryllactams. The composition of the reaction mixture was monitored by ^{31}P NMR.

Scheme 1



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In addition to *N*-phosphoryllactams, trimethylsilyl esters of the corresponding phosphorus acids, pyrophosphoryl derivatives, and products of dimerization of the starting lactam are formed. The ratio of the reaction products depends on the electrophilicity of the phosphoryl chloride and on the nature of the *N*-TMS-lactam.

The reaction of dialkyl chlorophosphates with silyllactams occurs in the most complicated way. The reaction of diethyl chlorophosphate (**1a**) with *N*-TMS-pyrrolidone (**2**) affords the corresponding *N*-(diethoxyphosphoryl)-2-pyrrolidone (**5a**) in only 42 % yield. Diethyl trimethylsilyl phosphate (**8a**) was also isolated from the reaction mixture in 16 % yield. ^{31}P NMR spectra showed that the reaction mixture contained 7 % tetraethyl pyrophosphate (**9a**) (δP −12.5; *cf.* Ref. 8: δP −13), which was identified by comparison with an authentic sample synthesized by the known procedure.[9] Similar results were obtained on treatment of diisopropyl chlorophosphate **1b** with five-, six-, and seven-membered *N*-TMS-lactams **2—4**. In this case, the yields of the target *N*-(diisopropoxyphosphoryl)-2-pyrrolidone (**5b**), *N*-(diisopropoxyphosphoryl)piperidone (**6b**), and *N*-(diisopropoxyphosphoryl)perhydro-2-azepinone (**7b**) were 48, 75, and 42 %, respectively. By-products were also formed: diisopropyl trimethylsilyl phosphate (**8b**) (yield 14—21 %) and tetraisopropyl pyrophosphate (**9b**) (yield 1—16 %), which displayed a signal at δP −14.4 in the ^{31}P NMR spectrum of the reaction mixture (*cf.* Ref. 10: δP −17), and trace amounts of 1-(1-pyrrolin-2-yl)-2-pyrrolidone (**10**).[11] The content of compound **10** in the reaction mixture can be increased by varying the conditions of the reaction of **1b** with **2** (see Experimental).

The replacement of dialkyl chlorophosphates **1a,b** by *O*-ethyl methylchlorophosphonate (**1c**) leads to only a small increase in the yield of the target *N*-phosphoryllactams. Treatment of compound **1c** with *N*-TMS-lactams affords *N*-(methylethoxyphosphoryl)-2-pyrrolidone (**5c**) in 69 % yield and *N*-(methylethoxyphosphoryl)-2-piperidone (**6c**) in 55 % yield, respectively. In addition, bis[ethyl(methyl)pyrophosphonate] (**9c**) is formed (in 7 and 23 % yields, respectively). The latter compound manifests itself by a signal at δP 23 in the ^{31}P NMR spectrum of the reaction mixture (*cf.* Ref. 8: δP 22.4).

Reactions of dibutylphosphinoyl chloride (**1d**) with *N*-TMS-lactams **2** and **4** mainly result in products of *N*-phosphorylation: *N*-dibutylphosphoryl-2-pyrrolidone (**5d**) and *N*-dibutylphosphorylcaprolactam (**7d**) in 87 and 89 % yields, respectively. According to the ^{31}P NMR spectra, dibutylphosphonic acid with δP 56.1 (*cf.* Ref. 8: δP 53.1) is present in the reaction mixtures (5 and 11 %, respectively). Its formation may be due to the hydrolytic instability of the initial by-product, namely, trimethylsilyl dibutylphosphinate.

A general scheme based on the dual reactivity of *N*-TMS-lactams can be suggested to explain the formation of these compounds. The participation of *O*-TMS-lactams,[11] which are the isomeric forms of the corresponding *N*-TMS-lactams, in the reaction seems to be unlikely. It has been previously shown by NMR spectroscopy[12—14] that *O*-TMS-derivatives were absent in acetonitrile solutions of compounds **2—4**.

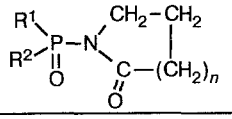
In addition to the direct attack at the nitrogen atom, the formation of *N*-phosphoryllactams may result from O→N isomerization[15] of the *O*-phosphorylated intermediates which also serve as the source of the by-products: silyl phosphates **8** and pyrophosphates **9**. The predominance of one or another route of the decomposition of the intermediates depends on a number of factors, including the nature of the organophosphorus fragment. For example, the yield of *N*-phosphorylated derivatives **5** and **7** decreases (Table 1) on successive substitution of the alkyl groups in the phosphorylating agent **1a—d** with alkoxyl, which can be explained by a decrease in the rate of the nucleophilic attack at the P atom of the intermediate by the N atom[16] and by an increase in the contribution of other routes of the transformation of the intermediates. If the chlorotrimethylsilane formed is continuously distilled off, the yield of *N*-phosphoryllactam **5b** increases and the yield of TMS-phosphate **8b** decreases. This may be due to the existence of an **A** ↔ **B** equilibrium (see Scheme 1), which is shifted in this case towards the formation of the **B** form.

Using the reaction of compound **1b** with **2** as an example (Table 2), we showed that both increasing and decreasing the temperature favors an increase in the contribution of *O*-phosphorylation of *N*-TMS-lactam and an increase in the relative yield of the products of decomposition of the intermediate (**A** or **B**). An attempt to accelerate phosphorylation (by heating to 110 °C without a solvent) resulted in an increase in the fraction of unidentified products.

The ratio of *N*- and *O*-phosphorylation is presumably determined by the same factors as the acylation of amides, which gives *O*-acyl derivatives under kinetically-controlled conditions or *N*-acyl derivatives under thermodynamically-controlled conditions.[17] The same regularity was observed in the phosphorylation of sodium derivatives of dihydro-5,6-benzo-1,4-diazepinones.[18] In the case studied (see Table 2), silyl phosphate **8b** predominates in the reaction mixture at room temperature, as a result of faster *O*-phosphorylation. 2-Chloropyrroline-1 should appear simultaneously with silyl phosphate and undergo a reaction with the starting silyllactam **2** to give compound **10** mentioned above. The constants and spectral data of compound **10** are consistent with the literature data.[11]

The data on the phosphorylation of derivatives of secondary carboxamides,[19,20] which results in a mixture of the corresponding *N*-phosphorylated products and significant amounts of pyrophosphates, also indicate the validity of the scheme proposed above. These pyrophosphates can also result from attack on the *O*-phosphorylated amide derivatives by chlorophosphates.

Under the conditions that were the most favorable for *N*-phosphorylation, we synthesized *N*-phosphoryl-

Table 1. *N*-Phosphoryllactams 

Compound	R^1	R^2	*n*	Yield (%) according to ^{31}P NMR	Yield (%) After chromatography on SiO_2	B.p./°C (*p*/Torr)	Molecular formula	Found / Calculated (%) C	H	P
5a	OEt	OEt	1	42	37.8[a]	98—110 (0.5)	$C_8H_{16}NO_4P$	43.48 / 43.44	7.32 / 7.29	13.85 / 14.00
5b	OPr^i	OPr^i	1	48	20.8[a]	105—106.5 (0.5)	$C_{10}H_{20}NO_4P$	47.93 / 48.19	7.97 / 8.09	12.02 / 12.43
5c	Me	OEt	1	69	58.1[b]	95—98 (0.5)	$C_7H_{14}NO_3P$	44.02 / 43.98	7.34 / 7.38	15.99 / 16.20
5d	Bu	Bu	1	87	82.3[c]	122—126 (0.1)	$C_{12}H_{24}NO_2P$	58.53 / 58.76	9.93 / 9.86	12.08 / 12.63
6b	OPr^i	OPr^i	2	75	51.3	108—112 (0.5)	$C_{11}H_{22}NO_4P$	49.81 / 50.18	8.66 / 8.42	12.08 / 11.76
6c	Me	OEt	2	55	46.0[d]	104—108 (0.1)	$C_8H_{16}NO_3P$	46.60 / 46.83	7.71 / 7.86	14.92 / 15.09
7b	OPr^i	OPr^i	3	42	36.8[a]	131—138 (0.5)	$C_{12}H_{24}NO_4P$	51.46 / 51.98	8.62 / 8.72	—
7d	Bu	Bu	3	89	74.2[e]	38—40.5[f]	$C_{14}H_{28}NO_2P$	61.22 / 61.51	10.36 / 10.32	10.73 / 11.33

[a] Ethyl acetate as the eluent. [b] MeCN as the eluent, b.p. 127—130 °C (0.4 Torr)[6]. [c] MeCN—ether (2:1) as the eluent. [d] MeCN—ether (1:1) as the eluent. [e] $CHCl_3$—THF (2:1) as the eluent. [f] M.p.

lactams **5—7**. They are colorless viscous liquids, except for the easily melting compound **7d**. After distillation *in vacuo*, they contain an admixture of the corresponding pyrophosphoryl compounds with close boiling points. Therefore, analytical samples were obtained by flash-chromatography.[21] Derivative **7d** was isolated directly from the reaction mixture using flash-chromatography.

The structure of compounds **5—7** was confirmed by ^1H and ^{31}P NMR and IR spectroscopy. Trimethylsilyl phosphates **8a,b** were identified on the basis of their physicochemical characteristics and NMR spectra.

It was assumed that unstable intermediate products of *O*-phosphorylation of lactams could be obtained by interaction of chlorophosphates with lactams in the presence of tertiary amine, similarly to acylation of lactams with acyl halides.[2,22] However, it has been previously reported[23] that the interaction of *O*-phenyl methylchlorophosphonate with 2-pyrrolidone (**11**) in the presence of Et_3N affords the corresponding pyrophosphonate instead of the expected *N*-phosphoryllactam. Similarly, in our case pyrophosphates **9a,b** turned out to be the main products of the reaction of **1a,b** with **11** in the presence of an equimolar amount of Et_3N in benzene or ether (yields 44 and 60 %, respectively). The ^{31}P NMR spectrum of the reaction mixture showed the formation of small amounts of *N*-phosphoryllactams **5a,b** in yields up to 10 %. A signal at δP −8.54 with relative intensity 63—70 % appears in the ^{31}P NMR spectrum during the reaction of **1b** with compound **11**. An attempt to isolate the corresponding compound (including flash-chromatography) resulted in its decomposition to give a pyrophosphate **9b**. In a parallel experiment, but in the absence of **11**, product **9b** was not found. This result of the reaction of compound **1b** with **11** can be explained by the formation of an intermediate complex of a product of *O*-phosphorylation of pyrrolidone with triethylamine. This complex can either react with the second molecule of **1b** or undergo hydrolysis during isolation; in both cases, compound **9b** is the ultimate product.

Thus, the interaction of *N*-TMS-lactams with chlorides of tetracoordinated phosphorus acids occurs as competitive *N*- and *O*-phosphorylation. The forma-

Table 2. Effect of temperature on the ratio of the products of the reaction of **1b** with **2** in acetonitrile

Temperature /°C	Reaction time/h	Composition of the reaction mixture (%)[a] **1b**	**5b**	**8b**	**9b**
20	120	31.6	19.3	49.0	—
60	3	11.2	37.1	25.1	9.1
80	3	5.7	47.5	19.4	10.0
110[b]	1	—	24.4	15.3	13.4

[a] According to ^{31}P NMR data. [b] In the absence of a solvent.

Table 3. Spectral characteristics of *N*-phosphoryllactams

Compound	IR, ν/cm^{-1}			^{31}P NMR,	^{1}H NMR,[a] δ (*J*/Hz)
	C=O	P=O	POC	δ	
5a	1735	1275	1035	−2.4[b]	1.15 (t, 6 H, C<u>H</u>$_3$CH$_2$O, *J* = 7.1); 1.92 (quint, 2 H, C<u>H</u>$_2$CH$_2$C=O, *J* = 7.5); 2.27 (t, 2 H, CH$_2$C=O, *J* = 8.0); 3.54 (t, 2 H, CH$_2$N, *J* = 7.0); 3.86–4.12 (m, 4 H, CH$_3$C<u>H</u>$_2$)
5b	1730	1275	1015	−4.8[b]	1.27 (d, 12 H, (C<u>H</u>$_3$)$_2$CHO, *J* = 6.7); 2.01 (quint, 2 H, C<u>H</u>$_2$CH$_2$C=O, *J* = 7.5); 2.37 (t, 2 H, CH$_2$C=O, *J* = 8.0); 3.66 (t, 2 H, CH$_2$N, *J* = 7.0); 4.56–4.78 (m, 2 H, (CH$_3$)$_2$C<u>H</u>)
5c	1715	1257	1045	26.3[c]	1.18 (t, 3 H, C<u>H</u>$_3$CH$_2$O, *J* = 7.0); 1.61 (d, 3 H, CH$_3$P, *J* = 18.3); 1.97 (quint, 2 H, C<u>H</u>$_2$CH$_2$C=O, *J* = 7.0); 2.34 (t, 2 H, CH$_2$C=O, *J* = 7.0); 3.63 (t, 2 H, CH$_2$N, *J* = 7.0); 3.73–4.03 (m, 2 H, CH$_3$C<u>H</u>$_2$O)
5d	1710	1235	—	47.0[c]	0.75 (t, 6 H, C<u>H</u>$_3$(CH$_2$)$_3$, *J* = 6.9); 1.14–1.95 (m, 12 H, CH$_3$(C<u>H</u>$_2$)$_3$P); 1.96 (quint, 2 H, C<u>H</u>$_2$CH$_2$C=O, *J* = 7.5); 2.33 (t, 2 H, CH$_2$C=O, *J* = 8.0); 3.57 (t, 2 H, CH$_2$N, *J* = 6.9)
6b	1695	1275	1015	−1.2[b]	1.15 (dd, 12 H, (C<u>H</u>$_3$)$_2$CH, J_1 = 9.0, J_2 = 6.2); 1.60–1.67 (m, 4 H, (C<u>H</u>$_2$)$_2$CH$_2$C=O); 2.27 (t, 2 H, CH$_2$C=O, *J* = 7.0); 3.44–3.52 (m, 2 H, CH$_2$N); 4.51–4.67 (m, 2 H, (CH$_3$)$_2$C<u>H</u>O)
6c	1675	1245	1050	29.3[c]	1.08 (t, 3 H, C<u>H</u>$_3$CH$_2$O, *J* = 7.0); 1.55 (d, 3 H, CH$_3$P, *J* = 18.0); 1.54–1.75 (m, 4 H, (C<u>H</u>$_2$)$_2$CH$_2$C=O); 2.23 (t, 2 H, CH$_2$C=O, *J* = 6.0); 3.43 (m, 2 H, CH$_2$N); 3.60–3.97 (m, 2 H, CH$_3$C<u>H</u>$_2$O)
7b	—	—	—	−0.4[b]	1.21 (dd, 12 H, (C<u>H</u>$_3$)$_2$CH, J_1 = 8.8, J_2 = 6.2); 1.63 (m, 6 H, (C<u>H</u>$_2$)$_3$CH$_2$C=O); 2.48 (m, 2 H, CH$_2$C=O); 3.61–3.67 (m, 2 H, CH$_2$N); 4.52–4.68 (m, 2 H, (CH$_3$)$_2$C<u>H</u>O)
7d	1657	1195	—	54.3[d]	0.70 (t, 6 H, C<u>H</u>$_3$(CH$_2$)$_3$, *J* = 6.9); 1.10–2.05 (m, 12 H, CH$_3$(C<u>H</u>$_2$)$_3$P); 1.45–1.61 (m, 6 H, (C<u>H</u>$_2$)$_3$CH$_2$C=O); 2.38–2.44 (m, 2 H, CH$_2$C=O); 3.45–3.52 (m, 2 H, CH$_2$N)

[a] In $CDCl_3$. [b] In CH_2Cl_2. [c] In ether. [d] In MeCN.

tion of trimethylsilyl phosphates, as well as the change in the composition of the products when the substituents at the phosphorus atom and the reaction conditions are varied, testify to the *O*-phosphorylated intermediate. The reaction discussed can sometimes be used as a preparative method for the synthesis of *N*-phosphoryllactams.

Experimental

^{1}H and ^{31}P NMR spectra were recorded on a Bruker WP 200-SY spectrometer (200 and 81 MHz) with HMDS as the internal standard and 85 % H_3PO_4 as the external standard. IR spectra (thin films) were obtained on a UR-20 spectrometer. *N*-Trimethylsilyllactams **2—4** were obtained by the known procedure.[24]

The syntheses were carried out in an atmosphere of dry argon. The solvents used were freshly distilled: acetonitrile was repeatedly distilled over P_2O_5, benzene and ether were dried with metallic sodium.

Synthesis of *N*-phosphoryllactams (5—7). Compound **1** (35.4 mol) was added with stirring at 80—85 °C over 15 min to a solution of *N*-TMS-lactam **(2—4)** (35.4 mol) in 35 mL of MeCN and the mixture was heated for 3 h. Chlorotrimethylsilane was simultaneously distilled off, and the temperature of distillation was kept below 70 °C. After that, the mixture was concentrated; distillation *in vacuo* (0.5 Torr) afforded phosphoryllactams **5—7** containing pyrophosphates as admixtures and TMS-phosphates **8a,b**. Compounds **5—7** were purified by flash-chromatography on silica gel (60—100 μ). The yields and characteristics of the products are given in Tables 1 and 3.

Diethyl trimethylsilyl phosphate (8a): yield 16.3 %, b.p. 50—52 °C (0.5 Torr), n_D^{20} 1.4088. ^{1}H NMR ($CDCl_3$), δ: −0.11 (s, 9 H, Me_3Si); 0.9 (t, 6 H, CH_3, *J* = 7 Hz); 3.57—3.71 (m, 4 H, CH_2). ^{31}P NMR (CH_2Cl_2), δ: −8.99. (Lit. data:[25] b.p. 97—98 °C (2 Torr), n_D^{20} 1.4070).

Diisopropyl trimethylsilyl phosphate (8b): yield 13.7—21.4 %, b.p. 47.5—48 °C (0.1 Torr). Found (%): C, 42.03: H, 8.87: P, 12.10. $C_9H_{23}O_4PSi$. Calculated (%): C, 42.50: H, 9.12: P, 12.18. ^{1}H NMR ($CDCl_3$), δ: −0.01 (s, 9 H, Me_3Si); 1.01 (d, 12 H, CH_3, *J* = 6 Hz); 4.20—4.36 (m, 2 H, CH). ^{31}P NMR (MeCN), δ: −9.93.

1-(1-Pyrrolin-2-yl)-2-pyrrolidone (10). Compound **1b** (40.3 mmol) was added at 50 °C with stirring over 15 min to a solution of **2** (40.3 mmol) in 40 mL of MeCN and the mixture was heated for 6 h at 50—70 °C with stirring. Chlorotrimethylsilane was simultaneously distilled off. Then the mixture was concentrated *in vacuo*. The residue was treated with 50 mL of ether and the resin insoluble in ether was separated, mixed with 20 mL of benzene, and washed with 10 mL of saturated aqueous Na_2CO_3. The aqueous layer was extracted with benzene, dried with anhydrous Na_2SO_4, and concentrated. The residue was crystallized from hexane to give 0.28 g (9.2 %) of **10**, m.p. 59—60 °C. ^{1}H NMR ($CDCl_3$), δ: 1.86—2.14 (m, 4 H, CH_2); 2.51 (t, 2 H, CH_2CO, *J* = 8 Hz); 3.07 (t, 2 H, CH_2C=N, *J* = 8 Hz); 3.68 (t, 2 H, CH_2N=C, *J* = 7 Hz); 3.84 (t, 2 H, $CH_2NC(O)$, *J* = 7 Hz). IR (KBr), ν/cm^{-1}: 1720 (C=O); 1630 (C=N). (Lit. data[11]: m.p. 60—60.5 °C).

Reaction of diethyl chlorophosphate (1a) with 2-pyrrolidone (11). A mixture of **1a** (30 mmol), **11** (30 mmol), and Et_3N (30 mmol) in 50 mL of benzene was heated with a reflux

condenser for 3 h at 80 °C, filtered, and concentrated. The residue was distilled *in vacuo* to give 1.91 g of tetraethyl pyrophosphate **9a**, yield 44 %, b.p. 128–129 °C (1 Torr), n_D^{20} 1.4193. ^{1}H NMR, δ: 1.12 (t, 12 H, CH_3, J = 7 Hz); 3.94–4.02 (m, 8 H, CH_2). ^{31}P NMR (MeCN), δ: −12.4. (Lit. data: b.p. 95–97 °C (0.1 Torr), n_D^{25} 1.4182[26], δP −13.1[8]).

Reaction of diisopropyl chlorophosphate (1b) with 2-pyrrolidone (11). A solution of **1b** (11 mmol) in 3 mL of benzene was added with cooling and stirring (2–5 °C) to a solution of **11** (11 mmol) and Et_3N (15 mmol) in 19 mL of benzene. The reaction mixture was stirred for 48 h at ~20 °C, then filtered and concentrated. Flash-chromatography on silica gel (60–100 μ, ethyl acetate as the eluent) afforded 1.14 g (60 %) of pyrophosphate **9b**, n_D^{22} 1.4175. ^{1}H NMR ($CDCl_3$), δ: 1.295 (d, 24 H, CH_3, J = 5 Hz); 4.68–4.76 (m, 4 H, CH). ^{31}P NMR (MeCN), δ: −14.5. (Lit. data: n_D^{25} 1.4163[27], δP −17.0[10]).

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