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Oxidation of Diethyl *N*-Sulfinylphosphoramidates

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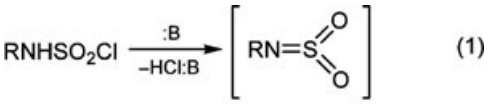
ABSTRACT: *It is reported that the diethyl N-sulfinylphosphoramidate (***1a***) is oxidized with iodoso- and/or iodoxybenzene as well as with organic peroxides, to give diethyl N-sulfonylphosphoramidate (***7a***). The latter was generated in situ at low temperature and trapped with dialkyl phosphoramidates, affording the N,N′ bis(dialkyl phosphor)sulfamides* **3a–c**. *The oxidation of* **1a** *with cumene peroxide in the presence of 2,2-dimethyl-propan-1-ol (***9***) produced the diethylphosphor-N-[2,2-dimethylpropyl]sulfamate (***10a***).* © 2008 Wiley Periodicals, Inc. Heteroatom Chem 19:530–536, 2008; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/hc.20474

INTRODUCTION

N-sulfonylamines, the structural analogs of sulfur trioxide, have been prepared previously [1–3] and are used as highly reactive electrophiles [4–11]. They are mostly available by the dehydrohalogenation of sulfamic acid chlorides as a compound stable only at low temperature in the solution (Eq. (1)).



(1)

R=Alkyl-, Bz-, Cinnamoyl-, AlkylOC(O)-

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The reaction of their formation, as short-lived intermediate products, was documented also in the hydrolysis and ammonolysis of sulfamate esters [12]. The electrophilic reactivity of sulfonylamines was demonstrated by their additions to unsaturated systems. Amongst them, the [2+2] cycloaddition reactions of *N*-sulfonylamines to olefin are known as a convenient route to the synthesis of the 1,2-thiazetidine-1,1-dioxides, β-sultam [13].

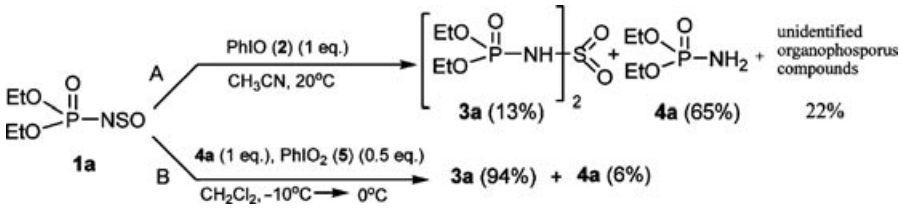
The latter one, being a derivative of taurine and sulfonyl analogs of β-lactams, still merits an investigation as a compound with potential applications in bioorganic and medical chemistry [14–20].

In this paper, the results of our studies on the oxidation of the sulfinyl sulfur atom in diethyl *N*-sulfinylphosphoramidate (**1a**) as a route to diethyl *N*-sulfonylphosphoramidate (**7a**) as an intermediate compound are described.

RESULTS AND DISCUSSION

Our earlier approach to the synthesis of *N*-phosphoryl sulfamoyl chloride, the substrate for the preparation of *N*-sulfonylphosphoramidate, was unsuccessful [21].

It is reasonable to mention that the oxidation reaction of sulfinyl group in *N*-sulfinylphosphoramidate should be considered as a potential route to the synthesis of such compounds. The synthetic approach to *N*-sulfonylamines by the oxidation reaction of thionitrozoarenes, followed by the participation of arenesulfinylamines as intermediate products, was demonstrated [22].



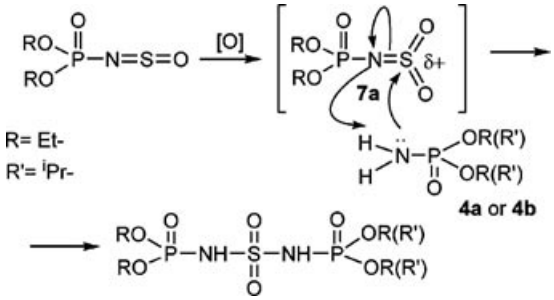
SCHEME 1

Dialkyl *N*-sulfinylphosphoramidates are known and being prepared as stable compounds by the reaction of the corresponding phosphoramidates and/or their *N*-silylated analogs with thionyl chloride [23–26] or *N*-(chlorosulfinyl)imidazole [27].

We have found that diethyl *N*-sulfinylphosphoramidate (**1a**) (Scheme 1, reaction (A)) reacts at 20°C in acetonitrile with 1 eq. of iodosobenzene (**2**). The formation of two products, namely the *N*,*N*′-bis(diethyl phosphor)sulfamide (**3a**) and diethyl phosphoramidate (**4a**), was registered by ^{31}P NMR in this reaction (Scheme 1).

The similar reaction performed between **1a** and 0.5 eq. iodoxybenzene (**5**) in the presence of 1 eq. amidate **4a** in dichloromethane at −10°C results in the formation of sulfamide **3a** in nearly quantitative yield (reaction (B)). The sulfamide **3a** is also formed in this reaction when cumene peroxide and/or *t*-butyl peroxide is used as an oxidizing agent. Without the oxidant, a 1:1 mixture of **1a** and **4a** does not react in dichloromethane solution at 20°C. In the reaction between *N*-sulfinylphosphoramidate **1a** and diisopropyl phosphoramidate (**4b**) (1:1 ratio) performed in the presence of 0.5 eq. of **5**, the formation of four identified compounds, **3a, 3b, 3c, 4a**, was confirmed by ^{31}P NMR (Scheme 2).

The products formed during the reaction, as shown in Scheme 2, were identified by comparing their ^{31}P shifts values with those characteristic for the compounds obtained independently (**3a, 3b** and **4a**). The addition of an excess of pyridine to this mixture of products resulted in the shift of the resonance lines, ascribed to the compounds **3a, 3b** and **3c** for 3–4 ppm, toward lower field as a result of the formation of their pyridinium salts [21]. In addition, in the MS FABpositive ion spectra registered for the

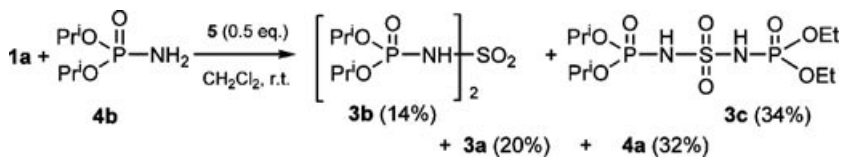


SCHEME 3

reaction mixture, in the presence of traces of sodium chloride, the mass peaks, corresponding to the cluster ions: **6a**, *m*/*z* 413.2, [M(**3a**) − H + 2Na]$^+$; **6b**, *m*/*z* 469, [M(**3b**) − H + 2Na]$^+$; **6c**, *m*/*z* 441, [M(**3c**) − H + 2Na]$^+$, respectively, were detected, providing further evidence for the correct identification of the putative products **3a, 3b**, and **3c** (Scheme 2). The strongly acidic properties of *N*,*N*′-bis-(dialkyl phosphor)sulfamides **3a–c** were demonstrated by the example of pure **3a** recently in [21].

The obtained results of the reaction of diethyl *N*-sulfinylphosphoramidate (**1a**) with iodoso-, iodoxybenzene, and organic peroxides give a clear evidence that these reactions occurred with the formation of a short-lived, electrophilic intermediate, diethyl *N*-sulfonylphosphoramidate (**7a**). This intermediate was trapped immediately by the amidate **4a** or **4b** with the formation of the corresponding *N*,*N*′-bis(dialkyl phosphor)sulfamides, most probably in the reaction as shown in Scheme 3.

Although we tried to keep the reaction mixture under completely anhydrous condition, it is



SCHEME 2

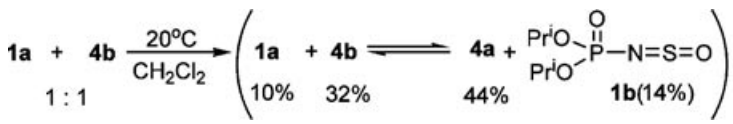
important to state that iodosobenzene cannot be obtained as an absolutely dry compound.

Dialkyl *N*-sulfinylphosphoramidates are extremely sensitive toward traces of water. In the presence of water, they are immediately transformed into the corresponding phosphoramidates [24].

The formation of the unsymmetrical sulfamide **3c** with nitrogen atoms substituted by different dialkoxy phosphoryl groups in the discussed process (Scheme 2) is the result of the reaction between *N*-sulfonylphosphoramidate **7a** and amidate **4b**. The other product formed in 10% yield in this reaction was identified as an *N*,*N*′-bis(diisopropyl phosphor)sulfamide (**3b)**. Its formation was rather unexpected since diisopropyl *N*-sulfinylphosphoramidate (**1b**) was not used as the substrate of the reaction.

However, in the independent experiment we established that the transfer of the sulfinyl group from diethyl *N*-sulfinylphosphoramidate (**1a)** to the phosphoryl atom of diisopropyl phosphoramidate **4b** took place. The formed *N*-sulfinylphosphoramidate **1b** undergoes the oxidation to the corresponding diisopropyl *N*-sulfonylphosphoramidate (**7b**), which is trapped by the amidate **4b** to give a symmetrical sulfamide **3b**.

The mixture of 1:1 molar equivalents of *N*-sulfinylphosphoramide **1a** and amide **4b** reacts at room temperature in dichloromethane solution, and after 4 h the formation of the composition the products, shown in Eq. (2) was registered by ^{31}P NMR.



(2)

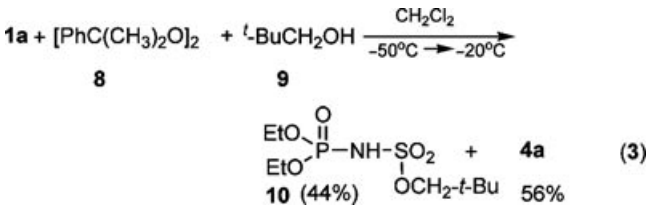
The similar transsulfinylation reaction of phenylenediamine and other amines, by the *N*-sulfinylaniline, was previously reported [28].

The alternative route to the formation of the unsymmetrical substituted sulfamide **3c**, the transphosphorylation reaction between **3a** and amide **4b** as well **3b**, was excluded.

Besides phosphoramidates, alcohols can be used for the trapping the short-lived *N*-sulfonylphosphoramidates, yielding the esters of the *N*-phosphoryl sulfamic acid [21].

An example of *N*-phosphoryl sulfamate was synthesized only recently, in the reaction of *n*-octylsulfamate with trialkyl phosphite in the presence of diisopropylazodicarboxylate and subsequent isomerization of the formed phosphazene with DABCO [29].

The performed oxidations of *N*-sulfinylphosphoramidate **1a** by iodosobenzene (**2**), and/or iodoxybenzene (**5**) in the presence of benzyl alcohol, ethanol, or phenol, in dichloromethane in the wide range of temperature, gave no evidence of formation of the *N*-phosphoryl sulfamates in these reactions. However, in the reaction of **1a** with cumene peroxide (**8**) in the presence of 2,2-dimethyl-propan-1-ol (**9**) at low temperature in dichloromethane the formation of two products was observed in the ^{31}P NMR spectra.

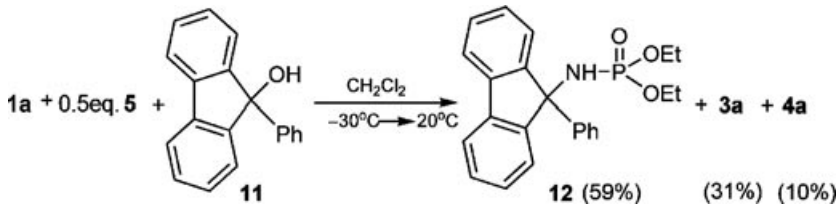


(3)

One of them was identified as an amidate **4a**. The second one resonating at δ −2.36 is most probably diethyl phosphor *N*-(2,2-dimethylpropyl)sulfamate (**10a**). In the MS FAB spectra of the crude reaction mixture, lines of mass *m*/*z* 154 [M + 1] amidate **4a** and *m*/*z* 302 [M + 1], characteristic for 1**0a**, were found. The attempts to isolate the putative ester **10a** by a column chromatography failed.

In the similar reaction of **1a** with 0.5 eq. of **5** performed in the presence of 9-phenylfluorenol (**11**), the formation of three products, shown in Scheme 4, was registered by ^{31}P NMR spectroscopy. In the MS FAB negative ions spectra of the reaction solution, among the lines corresponding to products **3a, 4a**, and **12**, a signal *m*/*z* 456 [M − 1], characteristic for diethyl phosphoramido-9-phenylofluorenyl sulfite **13b** was observed.

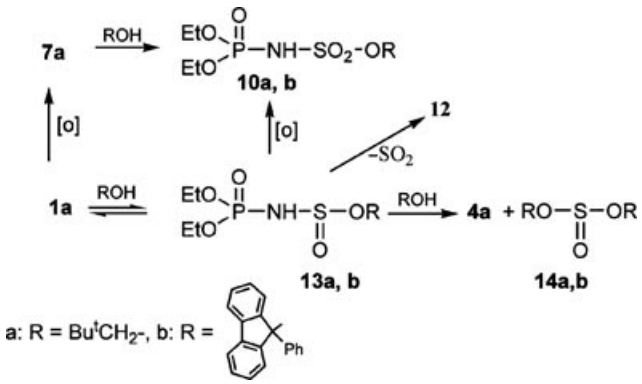
The oxidation of *N*-sulfinylphosphoramidate **1a** in the presence of alcohol seems to be rather a complex reaction. In the first step, the reaction performed at low temperature by addition of oxidant to the solution of **1a**, the formation of *N*-sulfonylphosphoramidate **7a** should take place. This reactive intermediate could interact with corresponding alcohol, introduced in the second step yielding the sulfamate (**10a**).



SCHEME 4

On the other hand, the direct reaction between alcohol and *N*-sulfinylphosphoramidate **1a** cannot be excluded. This possibility led to the formation of amidosulfites **13a** and **13b**. Their subsequent oxidation providing corresponding sulfamates **10a,b** is also possible. However, the value of *m/z* characteristic for **10b** was not found in the mass spectra of the reaction solution.

It is assumed that the reaction of simple alkyl alcohols with *N*-sulfinylamines and amides first led to the corresponding sulfinyl esters, which are unstable and cannot be isolated from the reaction solution. However, it was reported that in the reaction between *p*-toluenesulfinylamide and **11**, a relatively stable sulfinyl ester was formed. This was slowly decomposed in the reaction solution, with the formation of *p*-toluenesulfonyl 9-phenylfluorenylamide [30]. We assume that the amide **12** isolated from the reaction depicted in Scheme 5 is most probably the product of such decomposition of initially formed sulfinyl ester **13b**. The presence of compound **13b** in the reaction mixture was confirmed by MS spectrometry. The formation of the sulfamate **10a** as a reaction product (Scheme 3) may also be the result of the oxidation of sulfinyl ester **13a**. The obtained experimental data cannot give a convincing proof of these possible routes for the formation of **10a**.



SCHEME 5

Further studies on the oxidation of *N*-sulfinylphosphoramidates as a possible route to the *N*-sulfonylphosphoramidates, the precursors of *N*-phosphorylated β-sultams, are in progress.

EXPERIMENTAL

The solvents and reagents were purified by standard procedures before use. The column rapid chromatography was performed on Merck silica gel (60 mesh). ^{1}H NMR spectra were determined at 200.13 MHz with a Bruker AC 200 spectrometer using TMS as internal standard. ^{31}P NMR was taken on a Bruker AC 200 spectrometer at 81 MHz. Positive chemicals shifts are downfield from 85% H_3PO_4 used as an external reference. ^{1}H and ^{31}P NMR spectra were recorded in the presence of dried deuterochloroform. The MS spectra were recorded on Finnigan MAT 95 spectrometer in glycerol matrix using cesium as the primary ion beam. The substrates, diethyl *N*-sulfinylphosphoramidate (**1a**) [23,24], iodosobenzene (**2**) [31], iodoxybenzene (**5**) [32], diethyl phosphoramidate (**4a**) [33], diisopropyl phosphoramidate (**4b**) [34], and *N*,*N*′-bis(diethyl phosphor) sulfamide (**3a)** [21], were prepared as described in the literature.

*Oxidation of Diethyl N-Sulfinylphosphoramidate (***1a***)*

*By Iodosobenzene (***2***).* The suspension of 0.092 g (0.42 mmol) of **2** in the solution of 0.085 g (0.42 mmol) **1a** in 10 mL dry acetonitrile was stirred for 1 h at room temperature. After this time, the presence of two products, diethyl phosphoramidate (**4a)**, δ 9.6 ppm (65%), and *N*,*N*′-bis(diethyl phosphor)sulfamide (**3a**), δ −5.9 ppm (13%), as well ca. 22% of not identified product was registered in the reaction mixture by ^{31}P NMR.

*By Iodoxybenzene (***5***) in the Presence of Amidate* **4a**. To the stirred solution of 0.04 g (0.2 mmol) *N*-sulfinylphosphoramidate **1a** and 0.03 g (0.2 mmol) of **4a** in 5 mL of dichloromethane, cooled to −10°C,

0.023 g (0.1 mmol) of iodoxybenzene (**5**) in 5 mL dry dichloromethane was added. The stirring was continued for 2 h at this temperature. The solvent was removed under reduced pressure. In the integrated ^{31}P NMR spectra of the reaction mixture, two products were identified: diethyl phosphoramidate (**4a)**, δ 10.5 ppm, and *N*,*N*′-bis(diethyl phosphor)sulfamide (**3a**). After crystallization from chloroform/petroleum ether (bp 40–60°C), 0.068 g (94% yield) of pure **3a** was isolated.

Reaction of N-Sulfinylphosphoramidate **1a** *with Iodoxybenzene (***5***) and Diisopropyl Phosphoramidate (***4b***)*

Dry **5**, 0.14 g (0.59 mmol), was added in one portion to a stirred solution of 0.215 g (1.18 mmol) amidate **4b** and 0.235 g (1.18 mmol) of **1a** in 8 mL dichloromethane, under argon at the temperature 20°C. The reaction mixture was stirred at room temperature for 24 h, and after this time the reaction solution was analyzed by ^{31}P NMR. The integrated spectra showed formation of four products identified as *N*,*N*′-bis(diethyl phosphor) sulfamide (**3a**), δ −5.9 ppm (20% yield); *N*,*N*′-bis(diisopropyl phosphor) sulfamide (**3b**), δ −7.9 ppm (14% yield); *N*-[*O*,*O*-diethyl] *N*′-[*O*,*O*-diisopropyl phosphor] sulfamide (**3c**), δ −5.7 ppm and −8.1 ppm (34% yield); diethyl phosphor amidate (**4a**), δ 9.6 ppm (32% yield). The analysis of MS FAB spectra of this mixture showed the presence of lines ascribed to all identified product of the reaction: **3a**, m/z = 367.1, [M − 1]; **3b**, m/z = 423.2 [M − 1]; **3c**, m/z = 395.1 [M − 1]; **4a**, m/z = 154 [M + 1].

Interaction of the Crude Mixture of the Products **3a, 3b, 3c**, *and* **4a**

With Pyridine. To a stirred sample 0.1 g of a mixture **3a, 3b, 3c**, and **4a** dissolved in 5 mL of dry dichloromethane, 1 mL of pyridine was added at room temperature. In the ^{31}P NMR spectra of this solution, the presence of signals ascribed to the pyridinium salts of starting sulfamides was observed δ: −4.6, −2.1 ppm, **3c**; −4.2 ppm, **3b**; −2.5 ppm, **3a**. The resonance signal at δ ^{31} of **4a** was unchanged.

With Sodium Chloride. To the sample of 0.1 g mixture of products **3a, 3b, 3c**, and **4a** in 3 mL dry dichloromethane, 0.05 g of sodium chloride was added. The solvent was evaporated, and oily liquid material was obtained and analyzed by MS FAB spectrometry. In their positive ion spectra, three lines of mass characteristic for clusters ions: **6a**, m/z = 413.2, $[M(\mathbf{3a}) - H + 2Na]^+$; **6b**, m/z = 469.0, $[M(\mathbf{3b}) - H + 2Na]^+$; **6c**, m/z = 444.1, $[M(\mathbf{3c}) - H + 2Na]^+$ were detected.

Reaction of N-Sulfinylphosphoramidate **1a** *with Diisopropyl Phosphoramidate (***4b***)*

A mixture of 0.063 g (0.34 mmol) of **4b** and 0.069 g (0.34 mmol) of **1a**, dissolved in 5 mL of dry dichloromethane, was stirred for 4 h at room temperature. After this time in ^{31}P spectra of the reaction solution, the lines of diethyl phosphoramidate (**4a)** at δ 9.7 ppm and diisopropyl phosphor *N*-sulfinylamidate (**1b)** at δ −12.8 ppm were observed.

*Reaction of Diisopropyl Phosphoramidate (***4b***) with Thionyl Chloride*

A solution of 2.28 g (12.5 mmol) of amidate **4b** and 1.49 g (12.5 mmol) of freshly distilled thionyl chloride in 20 mL of benzene was heated under reflux for 6 h. The solvent was removed in vacuo. After the distillation, 2.1 g (74%) of oily liquid reaction product, identified as a diisopropyl *N*-sulfinylphosphoramidate (**1b**), was obtained: bp 84°C/0.8 mmHg; ^{1}H NMR, δ 1.37 ppm (dd, 12H, $(\underline{CH_3})_2CH$—), 4.88 (m, 2H, $(CH_3)_2\underline{CH}$—); ^{31}P NMR δ −12.4 ppm.

Reaction of **1a** *with Cumene Peroxide (***8***) in the Presence of 2,2-Dimethyl-propan-1-ol (***9***)*

To the solution of 1 g (5.0 mmol) *N*-sulfinylphosphoramidate **1a** in dry dichloromethane, 1.37 g (5.0 mmol) of cumyl peroxide (**8**) in 10 mL anhydrous dichloromethane was added at −50°C. The stirring was continued for 2 h and then the temperature was increased slowly for 40 min to −20°C. Then 0.44 g (5.0 mmol) of 2,2-dimethyl-propan-1-ol (**9**) in 5 mL of dichloromethane was added, and reaction mixture was stirred at this temperature for next 2 h. The solvent was removed in vacuo, and in the ^{31}P NMR spectra of the crude reaction mixture signals at δ −2.36 ppm (44%) and 9.7 ppm (56%) were observed. The first one we identified as a characteristic for diethyl phosphor *N*-(2,2-dimethylpropyl) sulfamate (**10a**), and the second one was ascribed to amidate **4a**. In the MS FAB spectra, lines of the mass m/z 154 [M + 1] **4a**, and m/z 302 [M − 1] **10a**, were observed. The attempt to isolate the ester **10a** by rapid column chromatography (petroleum ether (bp 40–60°C):acetone:chloroform) was unsuccessful.

Reaction of **1a** *with* **5** *and 9-Phenylfluorenol (*11*)*

To the suspension of 0.17 g (0.72 mmol) dry iodoxybenzene (**5**) and 0.37 g (1.43 mmol) of **11** in 10 mL dry dichloromethane, 0.285 g (1.43 mmol) of diethyl *N*-sulfinylphosphoramidate (**1a**), under argon at −30°C, was added. After 3 h, the temperature was slowly increased to 20°C, and the stirring was continued for next 3 h. The solvent was removed in vacuo, and obtained products were separated by column chromatography on silica gel (chloroform:hexane:ethyl acetate 3:1:1); 0.34 g (59%) of *O*,*O*-diethyl phosphor *N*-9-phenylfluorenol amidate (**12**), ^{1}H NMR δ 0.95 (t, 6H, $-OCH_2\underline{CH_3}$), 3.53 (q, 2H, $-O\underline{CH_2}CH_3$), 3.68 (q, 2H, $-O\underline{CH_2}CH_3$), 3.86 (d, 1H, NH), 7.25 (m, 6H, Ph), 7.36 (m, 6H, Ph), 7.66 (d, 1H, Ph), ^{31}P NMR δ5.1, MS FAB *m/z* 394.3[M + 1]; 0.16 g (31%) *N*,*N*′-bis(diethyl phosphor) sulfamide (**3a**), ^{31}P δ −6.1, MS FAB *m/z* 367.2 [M − 1]; 0.02 g (11%) diethyl phosphoramidate (**4a**), ^{31}P NMR δ 9.6, MS FAB *m/z* 154.0 [M + 1].

Reaction of **4b** *with Sulfuryl Chloride*

A solution of 0.24 g (1.32 mmol) of diisopropyl phosphoramidate (**4b**) and 0.35 g (2.6 mmol) of freshly distilled sulfuryl chloride in 15 mL dry dichloromethane was heated under reflux for 16 h. The solvent and the excess of sulfuryl chloride were removed in vacuo, and in ^{31}P NMR spectra of the remaining crude product, recorded after this time, the signal at δ −7.6 ppm was observed. After crystallization from chloroform:heptane, 0.35 g (64% yield) the pure *N*,*N*′-bis(diisopropyl phosphor) sulfamide (**3b**) was obtained. ^{1}H NMR, δ 1.36 (dd, 24 H, $-CH(\underline{CH_3})_2$; 4.82 (m, 4H, $\underline{CH}(CH_3)_2$; MS FAB *m/z* 423.2 [M − 1], mp 124–125°C.

Reaction of **3a** *with Diisopropyl Phosphoramidate (*4b*)*

A mixture of 0.1 g (0.26 mmol) of *N*,*N*′-bis(diethyl phosphor) sulfamide (**3a**) and 0.05 g (0.26 mmol) of diisopropyl phosphoramidate (**4b**) was stirred in 5 mL of dry dichloromethane at room temperature. After 24 h in the ^{31}P NMR spectra of this reaction solution, only the presence of signals −5.7 and 7.5 ppm, characteristic for starting substrates, was observed.

Reaction of **3a** *with* **3b**

A mixture of 0.1 g (0.26 mmol) of *N*,*N*′-bis(diethyl phosphor) sulfamide (**3a**) and 0.11 g (0.26 mmol) of *N*,*N*′-bis(diisopropyl phosphor) sulfamide (**3b**) was stirred in 5 mL of dry dichloromethane at room temperature. After 24 h in the ^{31}P NMR spectra of this solution mixture, only the presence of signals at δ −5.7 and −7.5 ppm, characteristic for starting sulfamides, was observed.

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