ISSN 1070-3632, Russian Journal of General Chemistry, 2006, Vol. 76, No. 8, pp. 1210–1218. © Pleiades Publishing, Inc., 2006.
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N-Sulfonyl- and *N*-Phosphorylbenzimidoylphosphonates

N. V. Kolotilo, A. A. Sinitsa, Yu. V. Rassukanaya, and P. P. Onys'ko

Institute of Organic Chemistry, National Academy of Sciences of Ukraine,
ul. Murmanskaya 5, Kiev, 02094 Ukraine
e-mail: onysko@rambler.ru

Received June 6, 2005

Abstract — A procedure for preparing *N*-sulfonyl- and *N*-phosphorylbenzimidoylphosphonates by oxidation of the corresponding α-(sulfonylamino)- and α-(phosphorylamino)benzylphosphonates was developed. The σ constants of imidoylphosphonate groups were evaluated by ^{19}F NMR spectroscopy, and specific features of their electronic effects were considered. The reactions of the imidoylphosphonates obtained with O-, S-, P-, and N-nucleophiles were studied. The phosphonate–phosphoramidate rearrangement of α-aminobenzylidenebisphosphonates was found.
DOI: 10.1134/S1070363206080068

Azomethines with activating electron-withdrawing sulfonyl and phosphoryl substituents at nitrogen are widely used in organic synthesis, in particular, for preparing chiral derivatives. C-Phosphorylated imines (imidoylphosphonates) are especially attractive as convenient precursors of α-amino phosphoryl derivatives exhibiting a set of useful properties [1–4]. At the same time, only a few representatives of *N*-phosphorylbenzimidoylphosphonates [5, 6] were described, whereas *N*-sulfonylbenzimidoylphospohonates as well as aryl imidoylphosphonates remained unknown up to our studies [7, 8].

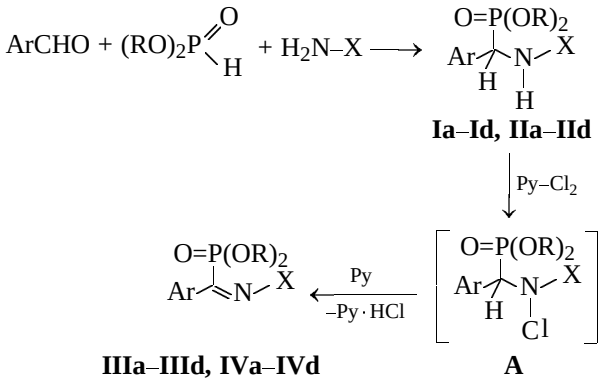
In this study we developed a synthetic route to benzimidoylphosphonates containing sulfonyl or phosphoryl substituent at nitrogen atom, considered the electronic nature of the imidoylphosphonate group, and examined some properties of the imines obtained.

The key stage of our approach (Scheme 1) is the oxidation of accessible sulfonyl- and phosphorylaminobenzylphosphonates **I** and **II** with the Py–Cl_2 complex, probably involving initial chlorination of the nitrogen atom [7–9]. Subsequent hydrogen chloride elimination in the intermediate amide **A** leads to the formation of *N*-sulfonyl- (**III**) or *N*-phosphorylimidoylphosphonates (**IV**) in high yields.

This approach allows preparation of both alkyl and aryl imidoylphosphonates. Note that the reaction of imidoyl chloride with phosphites, the most widely used procedure for preparing imidoylphosphonates [1], is inapplicable to preparation of the aryl esters.

Imidoylphosphonates are liquids (**IIIa**, **IIIb**, **IVa**–**IVd**) or crystalline compounds (**IIIc**, **IIId**) stable in a dry atmosphere but readily hydrolyzed with atmospheric moisture. Spectral data for C-phosphorylated amines confirm their structure. The signals of phosphorus at the imine carbon atom in the ^{31}P NMR spectra are observed in the range typical of imidoylphosphonates [1] (3.6–4.0 ppm for ethyl and –5.2 to –6.4 ppm for phenyl esters). In going from *N*-phosphorylamino phosphonates **II** to imidoylphosphonates **IV**, the coupling constant between the nonequivalent phosphorus nuclei significantly increases ($^3J_{PC=NP}$ 118–126 Hz). The IR spectra contain a strong band of C=N stretching vibrations (1610–1650 cm^{-1}). The presence of the P–C=N fragment is unambiguously confirmed by the ^{13}C NMR spectra containing the

Scheme 1.



I, III, X = SO_2Ph, R = Et, Ar = 4-FC_6H_4 (**a**), 3-FC_6H_4 (**b**); R = Ph, Ar = 4-FC_6H_4 (**c**) [8], 3-FC_6H_4 (**d**) [8]. **II, IV,** X = P(O)$(OEt)_2$, R = Et, Ar = Ph (**a**); R = Ph, Ar = 4-ClC_6H_4 (**b**), Ar = 4-FC_6H_4 (**c**), 3-FC_6H_4 (**d**).

Table 1. Chemical shifts of fluorinated benzenes XC_6H_4F and σ constants of N-substituted imidoylphosphonate (nos. 1–4) and (amino)phosphorylalkyl (nos. 5–8) groups

Run no.	X	$\delta_F(XC_6H_4F)$		σ_I	σ_R	σ_t
		3-F	4-F			
1	$PhSO_2N{=}C[P(O)(OEt)_2]$	2.09	7.42	0.38	0.18	0.56
2	$PhSO_2N{=}C[P(O)(OPh)_2]$	2.48	8.53	0.43	0.21	0.64
3	$(EtO)_2P(O)N{=}C[P(O)(OPh)_2]$	2.05	7.42	0.37	0.18	0.55
4	$PhCH_2N{=}C[P(O)(OEt)_2]$	1.80	2.38	0.34	0.02	0.36 [8]
5	$(EtO)_2P(O)NHCH[P(O)(OPh)_2]$	1.07	0.03	0.24	–0.04	0.20
6	$PhSO_2NHCH[P(O)(OPh)_2]$	0.95	0.14	0.22	–0.03	0.17
7	$PhSO_2NHCH[P(O)(OEt)_2]$	0.26	–0.52	0.12	–0.03	0.09
8	$PhSO_2NHCH[P(O)Ph_2]$	–0.15	–1.08	–0.06	–0.03	–0.09

characteristic signal of the imine sp^2 carbon atom having a high direct coupling constant with phosphorus (δ_C 174.6–181.1 ppm, $^1J_{PC}$ 181–204 Hz).

N-Sulfonyl- and *N*-phosphorylimines, as a rule, exhibit similar reactivity [10], although there are some significant differences [11]. Using the sensitivity of the fluorine nuclei to the electronic effects of substituents, we have quantitatively evaluated for the first time the electronic effects of *N*-sulfonyl- and *N*-phosphorylimidoyl groups. From the chemical shifts of fluorobenzimidoylphosphonates **III** and **IV** and the corresponding aminoalkylphosphonates **I** and **II**, measured against the internal fluorobenzene reference, we evaluated, using the Taft equation [12, 13], the inductive and resonance σ constants of N-substituted imidoylphosphonate {–C[P(O)(OR)$_2$]=NX} and (phosphoryl)aminoalkyl {–CH[P(O)(OR)$_2$]NHX} groups.

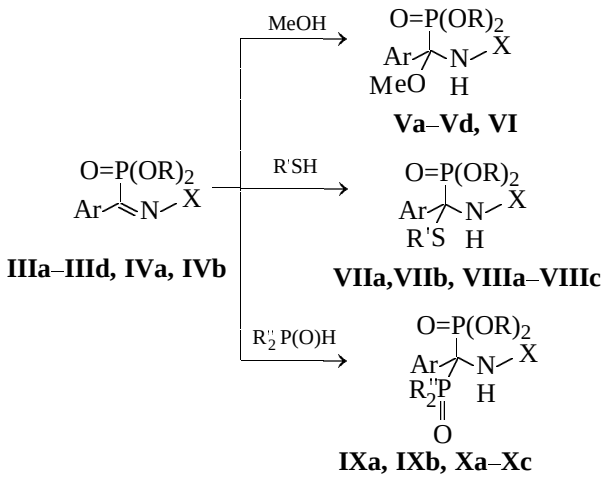
The data obtained show (Table 1) that *N*-sulfonyl- and *N*-phosphorylimidoylphosphonate groups are fairly strong electron acceptors (σ_t 0.55–0.64). In the total electron-withdrawing effect they significantly exceed their *N*-benzyl analog (no. 4). The acceptor effect of the imidoylphosphonate group depends on the nature of substituents at the nitrogen atom [$PhSO_2$ > $(EtO)_2P(O)$ >> $PhCH_2$] and of the phosphorus-containing group [$(PhO)_2PO$ > $(EtO)_2PO$]. An increase in the total acceptor effect in going from *N*-benzyl (no. 4) to *N*-sulfonyl (no. 1) or *N*-phosphoryl substituent (no. 3) is mainly due to the resonance component (σ_R), whereas σ_I varies insignificantly. This fact suggests higher polarizability of the C=N bond in *N*-sulfonyl and *N*-phosphorylimines. Note that substituted (amino)phosphorylalkyl groups (nos. 5–7) also exhibit electron-withdrawing properties (σ_p 0.2, 0.17, and 0.09, respectively). The effect of N-substituents on the variation of the electron-withdrawing properties [$(EtO)_2P(O)$ > $PhSO_2$] in this case is opposite compared to the imidoyl-containing groups (nos. 2, 3). This fact may be due to the difference in the NH acidity of the sulfonamide and phosphamide groups. At the same time, the diphenylphosphinoyl-containing substituent (no. 8) exhibits weak electron-donor properties (σ_t –0.09). In this connection, it should be noted that the strong tendency of the $Ph_2P(O)$ group in geminal aminobisphosphoryl compounds to undergo C–N phosphorotropic transfer is attributed to its electron-donor properties [11, 14]. The difference in the electronic properties of the *N*-sulfonyl- and *N*-phosphorylimine groups is probably one of the factors responsible for different directions of phosphorylation of these types of compounds [7, 11].

Strong electron-withdrawing power of sulfonyl- and phosphorylimidoylphosphonate groups causes high reactivity of phosphorylated imines **III** and **IV**, making it possible to readily carry out reactions untypical of nonactivated imidoylphosphonates. In particular, reactions with O- and S-nucleophiles occur under mild conditions (benzene, 20°C), require no catalyst, and yield adducts **V**–**VIII** stable under ambient conditions (Scheme 2).

Thioglycolic acid adducts **VIIa**, **VIIb**, and **VIIIa** do not tend to intramolecular cyclization to the corresponding thiazolidin-4-ones, contrary to their analogs containing trihalomethyl group instead of the aryl group [7].

The reaction of imines **III** and **IV** with hydrophosphoryl compounds allows preparation of *N*-sulfonyl and *N*-phosphorylaminoarylidenebisphosphoryl derivatives with the same (**Xa**, **Xc**) and different (**IXa**, **IXb**, **Xb**) geminal phosphorus-containing groups at the carbon atom. The reactivity of imidoylphosphonates toward hydrophosphoryl compounds increases

Scheme 2.

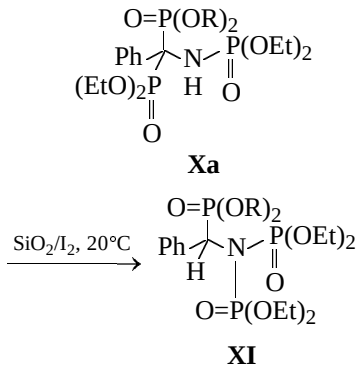


Va–**Vd**, X = SO_2Ph, R = Et, Ar = 4-FC_6H_4 (**a**), 3-FC_6H_4 (**b**); R = Ph, Ar = 4-FC_6H_4 (**c**), 3-FC_6H_4 (**d**). **VI**, X = P(O)$(OEt)_2$. **VIIa, VIIb**, X = SO_2Ph, R' = CH_2COOH, Ar = 4-FC_6H_4 (**a**), 3-FC_6H_4 (**b**). VIIIa–VIIIc, X = P(O)·$(OEt)_2$, Ar = Ph, R' = CH_2COOH (**a**), 4-ClC_6H_4 (**b**), H (**c**). **IXa, IXb**, X = SO_2Ph, R = Et, R'' = Ph, Ar = 4-FC_6H_4 (**a**), R = R'' = Ph, Ar = 4-FC_6H_4 (**b**). **Xa**–**Xc**, X = P(O)$(OEt)_2$, R = Et, R'' = EtO, Ar= Ph (**a**), R = Ph, R'' = EtO, Ar = 4-ClC_6H_4 (**b**), R = Ph, R'' = PhO, Ar= 4-ClC_6H_4 (**c**).

with an increase in the electron-acceptor power of aryl substituent (4-ClC_6H_4 > Ph) or of the phosphoryl group at the azomethine carbon atom [$(PhO)_2PO$ > $(EtO)_2PO$].

Geminal bisphosphonates **X** are stable in a dry atmosphere at room temperature, but heating causes their decomposition along several pathways. Therefore, these compounds were characterized without additional purification; according to the ^{31}P NMR data, they contain about 10–15% impurities. Attempted TLC purification of triphosphorylated adduct **Xa** on silica gel, followed by development with iodine vapor, causes irreversible 1,2-C–N transfer of the phosphoryl group with the formation of N,N-bisphosphorylated aminobenzylphosphonate **XI** (Scheme 3). The structure of **XI** is unambiguously confirmed by the spectral data. Its ^1H NMR spectrum contains a triplet of doublets of the CHP proton (δ 5.2 ppm, $^2J_{HP}$ 27.3, $^3J_{HP}$ 20.2 Hz). In the ^{13}C NMR spectrum, the signal of the CHP carbon atom is a doublet of doublets with the typical direct coupling constant with phosphorus ($^1J_{PC}$ 164 Hz). Under the spin decoupling conditions, the phosphoramidate and phosphonate phosphorus atoms give a doublet and a triplet, respectively (δ_P 3.9 and 22.1 ppm, $^3J_{PP}$ 13 Hz), with 2 : 1 ratio of the integral intensities.

Scheme 3.



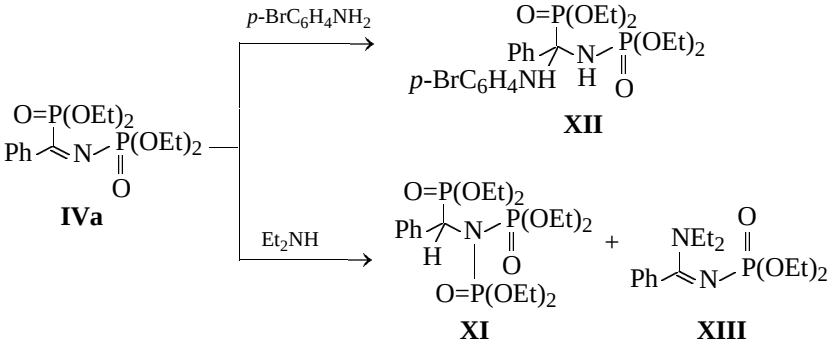
It is interesting that the rearrangement does not take place when keeping a solution of bisphosphonate **Xa** in the presence of iodine (benzene, 16°C, 15 h) or of anhydrous hydrogen chloride, and also when treating a benzene solution of **Xa** with silica gel in the absence of I_2. A base catalyst (Et_3N, refluxing in benzene) favors the rearrangement, but in this case it is accompanied by side processes.

Thus, the C→N phosphorotropic rearrangement that we discovered previously [14] is a fairly general reaction of α-amino-*gem*-bisphoshoryl compounds containing electron-withdrawing substituents at the nitrogen atom. We also discovered the thermal C→N phosphorotropic transfer in C,N-diphosphorylated ketene aminals [15]. On the other hand, generation of carbanions at the benzyl carbon atom of *N*-benzylphosphoramidates $PhCH_2N(X)P(O)(OEt)_2$ with *sec*-BuLi or *i*-Pr_2NLi causes the N→C migration of the phosphoryl group with the formation of amino phosphonates [16]. Hence, phosphoramidates and amino phosphonates tend to mutual transformations, which should be taken into account in synthetic practice as well as in evaluation of the biological activity of these compounds.

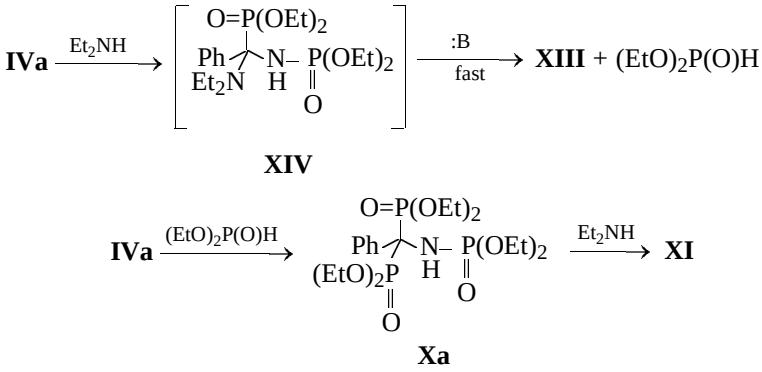
The tendency of amines as bases to induce the C–P bond cleavage evidently predetermines the complex pattern of their reaction with imidoylphosphonates. For example, the reaction with weakly basic 4-bromoaniline is fairly selective and yields amino amide **XII** characterized by elemental analysis and spectral data (Scheme 4).

The reaction with more basic diethylamine leads to an unexpected result. Diphosphorylated imine **IVa** transforms into triphosphorylated amide **XI** and monophosphorylared amidine **XIII** (Scheme 4). We believe that transformation **IVa**→**XI** follows Scheme 5.

Scheme 4.



Scheme 5.



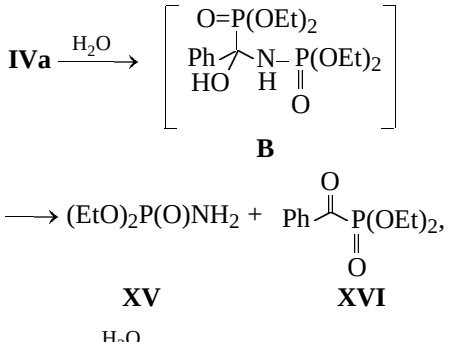
The initially formed adduct **XIV** cannot be detected spectroscopically, because diethylamine promotes fast elimination of diethyl hydrogen phosphite from NH acid **XIV**. Under the reaction conditions, phosphite easily adds to the C=N bond of the starting imidoylphosphonate **IVa** to give bisphosphonate **Xa**, which undergoes 1,2-C–N transfer of the phosphoryl group catalyzed by diethylamine. It was shown in an independent experiment that diethylamine, indeed, promotes the transformation **Xa** → **XI**. Bisphosphonate **Xa**, amidine **XIII** (δ_P 2.9 ppm), and diethyl hydrogen phosphite were detected spectroscopically, and phosphonate **XI** was identified by comparison with a sample prepared following Scheme 4.

Adducts of imidoylphosphonates **IV** with O-, S-, and P-nucleophiles are fairly stable compounds slowly decomposing during prolonged storage. On the contrary, addition of water to imine **IVa** causes decomposition of the molecule (Scheme 6).

Low stability of adduct **B** is apparently due to easy elimination of phosphoramidate **XV** and formation of benzoylphosphonate **XVI**. Hydrolysis of **XVI** yields benzoic acid and diethyl hydrogen phosphite, which were identified by comparison with authentic samples.

The composition and structure of products formed by the reactions of imidoylphosphonate with nucleophiles were confirmed by elemental analysis and spectral data. The transformation of imidoylphosphonates into aminoalkylphosphonates is accompanied by a significant downfield shift (10–15 ppm) of the phosphonate phosphorus signal in the ^{31}P NMR spectra, which convenient for monitoring the reaction progress. An important spectral feature of phosphorylamino phosphonates $RC_6H_4CX[NHP(O)(OR')_2]P(O)\cdot(OR)_2$ is that the coupling constants of the nonequivalent phosphorus nuclei vary in a very wide range (4–66 Hz depending on substituent **X**, Table 2).

Scheme 6.



Note also the remote fluorine–phosphorus coupling in 4-fluorobenzylaminophosphonates **Ia**, **Ic**, **IIc**, **IVb**,

Table 2. Chemical shifts (δ_P) and coupling constants of nonequivalent phosphorus atoms ($^3J_{PP}$) in amino phosphonates of the general formula $RC_6H_4CX[NHP(O)(OR')_2]P(O)(OR)_2$

X	R	R'	δ_{PN}, ppm	δ_{PC}, ppm	$^3J_{PP}$, Hz
MeO	H	Et	5.3	17.1	66
SCH_2COOH	H	Et	5.3	18.4	51
4-ClC_6H_4S	H	Et	4.6	17.3	50
SH	H	Et	5.7	17.9	49
H	H	Et	7.4	22.7	49
H	4-F	Ph	8.6	16.4	42
H	3-F	Ph	8.4	16.0	41
H	4-Cl	Ph	8.1	14.8	40
$(EtO)_2P(O)$	H	Et	4.8	18.5	22
$(PhO)_2P(O)$	4-Cl	Et	4.4	17.2	16.5
$(EtO)_2P(O)$	4-Cl	Ph	4.4	10.1	35
$(PhO)_2P(O)$	4-Cl	Ph	2.0	6.8	27
4-BrC_6H_4NH	H	Et	3.5	18.0	7

and **IXb** ($^6J_{PF}$ 1–6 Hz). Similar coupling also occurs in imidoylphosphonates **III**, but in this case the constant is smaller (for **Ia** and **IIIa**, $^6J_{PF}$ is 1.8 and 4.6 Hz, respectively). We found the similar effect previously for *N*-benzylidenamino-substituted fluorobenzylphosphonates 4-$FC_6H_4CH[P(O)R_2]N=CHPh$ [17]. This is a distinctive feature of N-substituted α-aminobenzylphosphonates. The trends we revealed can be used in structural studies of related systems.

Our results show that *N*-sulfonyl- and *N*-phosphorylbenzimidoylphosphonates are promising for preparing polyfunctional derivatives of α-amino phosphonic acids.

EXPERIMENTAL

The IR spectra were recorded on a UR-20 spectrometer. The ^1H, ^{19}F, ^{31}P, and ^{13}C NMR spectra were taken on a Varian VXR-300 spectrometer, operating frequences 299.95, 282.20, 121.42, and 75.428 MHz, respectively. The chemical shifts are given relative to internal TMS (^1H, ^{13}C), $CFCl_3$ or PhF (^{19}F), and external 85% phosphoric acid (^{31}P). All reactions were carried out in anhydrous solvents under argon.

Compounds **Ic**, **IIIc**, **IIId**, and **IXb** were described by us in a preliminary communication [8]. Sulfonamides **Ia** and **Ib** were prepared according to [8]; phosphoramidate **IIa**, according to [18]; and compounds **IIb**–**IId**, similarly to [19].

Diethyl α-(phenylsulfonylamino)-4-fluorobenzylphosphonate Ia. Yield 58%, mp 148°C. IR spectrum, ν, cm^{-1}: 1070 (POC), 1180, 1350 (S=O), 1250 (P=O), 3160 (NH). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.06 t (3H, CH_3, $^3J_{HH}$ 6.6 Hz), 1.36 t (3H, CH_3, $^4J_{HH}$ 6.6 Hz), 3.61–3.72 m (1H, CH_2), 3.85–3.95 m (1H, CH_2), 4.22–4.28 m (2H, CH_2), 4.80 d.d (1H, CH, $^2J_{HP}$ 24, $^3J_{HH}$ 9.6 Hz), 6.77 t (2H, Ar, $^3J_{HH}$ 8 Hz), 6.97 br.s (1H, NH), 7.17–7.34 m (5H, Ar), 7.90 d (2H, Ar, $^3J_{HH}$ 8 Hz). ^{19}F NMR spectrum ($CDCl_3$), δ_F, ppm: –114.17. ^{31}P NMR spectrum ($CDCl_3$), δ_P, ppm: 20.1 d ($^2J_{PH}$ 24 Hz). Found, %: C 50.44; H 5.12; P 7.84; S 8.21. $C_{17}H_{21}FNO_5PS$. Calculated, %: C 50.87; H 5.27; P 7.72; S 7.79.

Diethyl α-(phenylsulfonylamino)-3-fluorobenzylphosphonate Ib. Yield 55%, mp 116°C. IR spectrum (KBr), ν, cm^{-1}: 1050 (POC), 1180, 1350 (S=O), 1250 (P=O), 3150 (NH). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.07 t (3H, CH_3, $^3J_{HH}$ 6.6 Hz), 1.40 t (3H, CH_3, $^3J_{HH}$ 6.6 Hz), 3.59–3.67 m (1H, CH_2), 3.85–3.96 m (1H, CH_2), 4.28–4.38 m (2H, CH_2), 4.85 d.d (1H, CH, $^2J_{PH}$ 25, $^3J_{HH}$ 10 Hz), 6.78 t (1H, Ar, $^3J_{HH}$ 8 Hz), 6.97–7.15 m (3H, Ar, NH), 7.17 t (2H, Ar, $^3J_{HH}$ 8 Hz), 7.31–7.33 m (1H, Ar), 7.60–7.72 m (1H, Ar), 7.60–7.72 m (3H, Ar). ^{19}F NMR spectrum ($CDCl_3$), δ_F, ppm: –113.55. ^{31}P NMR spectrum ($CDCl_3$), δ_P, ppm: 18.8 d ($^2J_{PH}$ 25 Hz). Found, %: C 50.95; H 5.13; P 7.69; S 8.21. $C_{17}H_{21}FNO_5PS$. Calculated, %: C 50.87; H 5.27; P 7.72; S 7.99.

Diphenyl α-(diethoxyphosphorylamino)-4-chlorobenzylphosphonate IIb [19]. IR spectrum (KBr), ν, cm^{-1}: 960 (P–O–Ph), 1060 (POEt), 1230, 1255 (P=O), 3260 (NH). ^{31}P NMR spectrum (C_6H_6), δ_P, ppm: 8.1 d (1P, PN, $^3J_{PP}$ 40 Hz), 14.8 d (1P, PC, $^3J_{PP}$ 40 Hz).

Diphenyl α-(diethoxyphosphorylamino)-4-fluorobenzylphosphonate IIc. Yield 70%, mp 143–

145°C (acetone). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.09 t (3H, CH_3, $^3J_{HH}$ 7.2 Hz), 1.20 t (3H, CH_3, $^3J_{HH}$ 7.2 Hz), 3.7–3.8 m (1H, CH_2O), 3.90–4.0 m (3H, CH_2O), 4.4 br.s (1H, NH), 4.94 d.d.d (1H, CH, $^2J_{HP}$ 23.7, $^3J_{HP} \approx {}^3J_{HH}$ 11 Hz), 6.8–7.5 m (14H, Ar). ^{19}F NMR spectrum (benzene), δ_F, ppm: –113.7. ^{31}P–{^{1}H} NMR spectrum ($CDCl_3$), δ_P, ppm: 8.6 d (1P, PN, $^3J_{PP}$ 42 Hz), 16.4 d.d (1P, PC, $^3J_{PP}$ 42, $^6J_{PF}$ 4.6 Hz). Found, %: P 12.47. $C_{23}H_{26}FNO_6P_2$. Calculated, %: P 12.55.

Diphenyl α-(diethoxyphosphorylamino)-3-fluorobenzylphosphonate IId. Yield 75%, mp 167–169°C (acetone). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.08 t (3H, CH_3, $^3J_{HH}$ 7.3 Hz), 1.21 t (3H, CH_3, $^3J_{HH}$ 7.2 Hz), 3.7 m (1H, CH_2O), 4.0 m (3H, CH_2O), 4.3 br.s (1H, NH), 4.95 d.d.d (1H, CH, $^2J_{HP}$ 23.8, $^2J_{HP} \approx {}^3J_{HH}$ 11 Hz), 6.84 m (2H, Ar), 7.0–7.5 m (12H, Ar). ^{19}F NMR spectrum (C_6H_6), δ_F, ppm: –112.5. ^{31}P–{^{1}H} NMR spectrum ($CDCl_3$), δ_P, ppm: 8.4 d (1P, PN, $^3J_{PP}$ 41 Hz), 16.0 d.d (1P, PC, $^3J_{PP}$ 41, $^3J_{PF}$ 1.5 Hz). Found, %: P 12.57. $C_{23}H_{26}FNO_6P_2$. Calculated, %: P 12.55.

***N*-[(Diphenylphosphinoyl)(3-fluorophenyl)methyl]benzenesulfonamide** was prepared similarly to [7]; yield 54%, mp 225°C. ^{19}F NMR spectrum ($CDCl_3$), δ_F, ppm: –113.75. ^{31}P NMR spectrum (C_6H_6), δ_P, ppm: 33.3. Found, %: C 64.82; H 4.47. $C_{25}H_{21}FNO_3PS$. Calculated, %: C 64.51; H 4.55.

Imidoylphosphonates III and IV. To a solution of 0.02 mol of chlorine in 60 ml of carbon tetrachloride, 0.044 mol of anhydrous pyridine was added dropwise with stirring and cooling to 0°C. The resulting mixture was stirred for an additional 30 min and then was gradually warmed to room temperature. After that, 0.02 mol of appropriate amide were added in portions. The reaction mixture was stirred for 8–10 h at room temperature, pyridine hydrochloride was filtered off, and the filtrate was evaporated in a vacuum.

Diethyl *N*-(phenylsulfonyl)-4-fluorobenzimidoylphosphonate IIIa. Yield 83%. IR spectrum (in thin film), ν, cm^{-1}: 1070 (POC), 1180, 1350 (S=O), 1260 (P=O), 1610 (C=N). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.19 m (6H, CH_3), 4.03–4.26 m (4H, CH_2), 7.19 t (2H, Ar, $^3J_{HH}$ 8 Hz), 7.52 t (2H, Ar, $^3J_{HH}$ 8 Hz), 7.62 t (1H, Ar, $^3J_{HH}$ 8 Hz), 7.87–7.94 m (4H, Ar). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 15.8 d (CH_3, $^3J_{CP}$ 7 Hz), 64.5 d (CH_2, $^2J_{CP}$ 7 Hz), 115.3 d (C^3, ArF, $^2J_{CF}$ 23 Hz), 127.2 s (C^2, C^6, $ArSO_2$), 128.8 s (C^3, C^5, $ArSO_2$), 129.6 d.d (*C*C=N, $^2J_{CP}$ 25, $^2J_{CF}$ 3 Hz), 131.0 d.d (C^2, ArF, $^3J_{CF}$ 9, $^3J_{CP}$ 4 Hz), 133.3 s (C^4, $ArSO_2$), 139.6 s (C–SO_2), 164.5 d (C–F, $^1J_{CF}$ 254 Hz), 176.8 d (C=N, $^1J_{CP}$ 198 Hz). ^{19}F NMR spectrum ($CDCl_3$), δ_F, ppm: –106.18. ^{31}P NMR spectrum ($CDCl_3$), δ_P, ppm: 4.0. Found, %: C 51.34; H 4.91; P 7.65; S 7.98. $C_{17}H_{19}FNO_5P$. Calculated, %: C 51.13; H 4.80; P 7.76; S 8.03.

Diethyl *N*-(phenylsulfonyl)-3-fluorobenzimidoylphosphonate IIIb. Yield 87%. IR spectrum (in thin film), ν, cm^{-1}: 1070 (POC), 1180, 1350 (S=O), 1260 (P=O), 1610 (C=N). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.20 m (6H, CH_3), 4.03–4.22 m (4H, CH_2), 7.22 t (1H, Ar, $^3J_{HH}$ 8 Hz), 7.45–7.62 m (6H, Ar), 7.91 d (2H, Ar, $^3J_{HH}$ 8 Hz). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 16.0 d (CH_3, $^3J_{CP}$ 6.6 Hz), 64.7 d (CH_2, $^2J_{CP}$ 6 Hz), 115.2 d.d (C^2, ArF, $^2J_{CF}$ 24, $^3J_{CP}$ 3.7 Hz), 118.6 d (C^4, ArF, $^2J_{CF}$ 19 Hz), 124.0 t (C^6, ArF, $^3J_{CP} = {}^4J_{CP}$ 6 Hz), 127.5 s (C^2, C^6, $ArSO_2$), 128.9 s (C^3, C^5, $ArSO_2$), 130.0 d (C^5, ArF, $^3J_{CF}$ 7.8 Hz), 133.5 s (C^4, $ArSO_2$), 135.3 d.d (*C*C=N, $^2J_{CP}$ 26, $^3J_{CF}$ 9 Hz), 139.5 s (C–SO_2), 161.8 d (C–F, $^1J_{CF}$ 249 Hz), 176.8 d (C=N, $^1J_{CP}$ 201 Hz). ^{19}F NMR spectrum ($CDCl_3$), δ_F, ppm: –111.54. ^{31}P NMR spectrum ($CDCl_3$), δ_P, ppm: 3.6. Found, %: C 50.88; H 4.76; P 7.83; S 8.12. $C_{17}H_{19}FNO_5PS$. Calculated, %: C 51.13; H 4.80; P 7.76; S 8.03.

Diethyl *N*-(diethoxyphosphoryl)benzimidoylphosphonate IVa. Yield 89%, bp 175–180°C (0.05 mm Hg). IR spectrum (in thin film), ν, cm^{-1}: 1240, 1250 (P=O), 1650 (C=N). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 0.78 m (6H, CH_3), 0.92 m (6H, CH_3), 3.64 m (4H, OCH_2), 3.85 m (4H, OCH_2), 7.09 m (3H, Ph), 7.7 m (2H, Ph). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 15.1 d (CH_3, $^3J_{CP}$ 6.4 Hz), 15.5 d (CH_3, $^3J_{CP}$ 5.8 Hz), 62.6 d (CH_2, $^2J_{CP}$ 6.7 Hz), 63.3 d (CH_2, $^2J_{CP}$ 6.8 Hz), 127.2, 127.3 (C^2, C^3, Ph), 136.3 d.d (C^1, Ph, $^2J_{CP}$ 36 Hz, $^3J_{CP}$ 16 Hz), 130.6 s (C^4, Ph), 181.0 d (C=N, $^1J_{CP}$ 195 Hz, $^2J_{CP}$ 6.5 Hz). ^{31}P–{^{1}H} NMR spectrum ($CDCl_3$), δ_P, ppm: 0.49 d (1P, PN, $^3J_{PP}$ 118 Hz), 3.81 d (1P, PC, $^3J_{PP}$ 118 Hz). Found, %: P 16.29. $C_{15}H_{25}HO_6P_2$. Calculated, %: P 16.41.

Diphenyl *N*-diethoxyphosphoryl-4-chlorobenzimidoylphosphonate IVb. Yield 80%. Viscous liquid. IR spectrum (in thin film), ν, cm^{-1}: 1215 (POC), 1220, 1285 (P=O), 1645 (C=N). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.09 d.t (6H, CH_3, $^3J_{HH}$ 7.2, $^4J_{PH}$ 0.9 Hz), 3.9 m (4H, OCH_2), 7.1–7.4 m (10H, OPh), 7.40 d (2H, Ar, $^3J_{HH}$ 8 Hz), 7.83 d (2H, Ar, $^3J_{HH}$ 8 Hz). ^{31}P NMR spectrum (benzene), δ_P, ppm: –5.2 d (1P, PC, $^1J_{PP}$ 126 Hz), –0.5 d (1P, PN, $^3J_{PP}$ 126 Hz). Found, %: P 12.09. $C_{23}H_{24}ClNO_6P_2$. Calculated, %: P 12.20.

Diphenyl *N*-diethoxyphosphoryl-4-fluorobenzimidoylphosphonate IVc. Yield 84%. Viscous oil.

^{19}F NMR spectrum ($CDCl_3$), δ_F (PhF), ppm: 7.42. ^{31}P NMR spectrum ($CDCl_3$), δ_P, ppm: –6.4 d (1P, PC, $^3J_{PP}$ 120 Hz), –1.4 d (1P, PN, $^3J_{PP}$ 120 Hz). Found, %: P 12.41. $C_{23}H_{24}FNO_6P_2$. Calculated, %: P 12.60.

Diphenyl *N*-diethoxyphosphoryl-3-fluorobenzimidoylphosphonate IVd. Yield 42%, viscous liquid. ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 15.5 d (CH_3, $^3J_{CP}$ 7 Hz), 63.4 d (CH_2, $^2J_{CP}$ 5.5 Hz), 114.9 d.d (C^2, ArF, $^2J_{CF}$ 24, $^3J_{CP}$ 4 Hz), 118.5 d (C^4, ArF, $^2J_{CF}$ 21 Hz), 119.9 d (C^2, PhO, $^3J_{CP}$ 5 Hz), 123.8 s (C^6, ArF), 125.2 (C^4, PhO), 129.5 s (C^3, PhO), 129.9 d (C^5, ArF, $^3J_{CF}$ 9 Hz), 137.5 d.d.d (*C*C=N, $^2J_{CP}$ 39, $^3J_{CP}$ 15, $^3J_{CF}$ 7 Hz), 149.5 d (C^1, PhO, $^2J_{CP}$ 8 Hz), 161.6 d (C–F, $^1J_{CF}$ 249 Hz), 177.1 d.d (C=N, $^1J_{CP}$ 206 Hz, $^2J_{CP}$ 8 Hz). ^{19}F NMR spectrum ($CDCl_3$), δ_F (PhF), ppm: 2.05. ^{31}P–{^{1}H} NMR spectrum ($CDCl_3$), δ_P, ppm: –5.8 d (1P, PC, $^3J_{PP}$ 122 Hz), –0.6 d (1P, PN, $^3J_{PP}$ 122 Hz). Found, %: P 12.69. $C_{23}H_{24}FNO_6P_2$. Calculated, %: P 12.60.

Diethyl α-(phenylsulfonylamino)-α-methoxy-4-fluorobenzylphosphonate Va. A solution of 0.5 mmol of imidoylphosphonate **IIIa** in 5 ml of absolute methanol was kept at room temperature for 12 h. The solvent was removed in a vacuum, and the residue was washed with petroleum ether. Yield ~100%, mp 111–112°C. IR spectrum (KBr), ν, cm^{-1}: 1070 (POC), 1240 (P=O), 1180, 1350 (S=O), 3170 (NH). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.19 t (3H, CH_3, $^3J_{HH}$ 7 Hz), 1.23 t (3H, CH_3, $^3J_{HH}$ 7 Hz), 3.51 s (3H, CH_3O), 3.89–4.11 m (4H, CH_2), 6.38 d (1H, NH, $^3J_{HP}$ 9.3 Hz), 6.84 t (2H, Ar, $^3J_{HH}$ 8 Hz), 7.37–7.63 m (7H, Ar). ^{19}F NMR spectrum ($CDCl_3$), δ_F, ppm: –114.04. ^{31}P NMR spectrum ($CDCl_3$), δ_P, ppm: 14.9. Found, %: P 6.89; S 7.54. $C_{18}H_{23}FNO_6PS$. Calculated, %: P 7.18; S 7.43.

Diethyl α-(diethoxyphosphoryl)amino-α-(methoxy)benzylphosphonate VI was prepared similarly. Yield 96%, viscous oil. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.19 m (12H, CH_3), 3.64 s (3H, CH_3O), 3.96 m (8H, CH_2), 7.32 m (3H, Ph), 7.73 m (2H, Ph). ^{31}P NMR spectrum (benzene), δ_P, ppm: 5.3 d (1P, PN, $^3J_{PP}$ 66 Hz), 17.1 d (1P, PC, $^3J_{PP}$ 66 Hz). Found, %: P 15.24. $C_{16}H_{29}NO_7P_2$. Calculated, %: P 15.13.

Compounds VII. To a solution of 1 mmol of appropriate imidoylphosphonate in 5 ml of diethyl ether, 1 mmol of thioglycolic acid was added. After standing for 12 h, the crystalline product was filtered off.

{[(Diethoxyphosphoryl)(phenylsulfonylamino)(4-fluorophenyl)methyl]thio}acetic acid VIIa. Yield 64%, mp 61°C. IR spectrum (KBr), ν, cm^{-1}: 1060 (POC), 1180, 1350 (S=O), 1240 (P=O), 1720 (C=O), 3210 (NH). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.21 m (6H, CH_3), 2.87 d (1H, SCH^A, $^2J_{H^AH^B}$ 15.6 Hz), 3.31 d (1H, SCH^B, $^2J_{H^AH^B}$ 15.6 Hz), 4.01–4.19 m (4H, CH_2), 6.32 d (1H, NH, $^3J_{HP}$ 8 Hz), 6.94 t (2H, Ar, $^3J_{HH}$ 8 Hz), 7.44 t (2H, Ar, $^3J_{HH}$ 8 Hz), 7.57 m (3H, Ar), 7.81 d (2H, Ar, $^3J_{HH}$ 8 Hz). ^{19}F NMR spectrum ($CDCl_3$), δ_F, ppm: –115.10. ^{31}P NMR spectrum ($CDCl_3$), δ_P, ppm: 16.0. Found, %: P 6.19; S 13.11. $C_{19}H_{23}FNO_7PS_2$. Calculated, %: P 6.30; S 13.05.

{[(Diethoxyphosphoryl)(phenylsulfonylamino)(3-fluorophenyl)methyl]thio}acetic acid VIIb. Yield 55%, mp 142°C. IR spectrum (KBr), ν, cm^{-1}: 1060 (POC), 1180, 1350 (S=O), 1240 (P=O), 1720 (C=O), 3210 (NH). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.25 m (6H, CH_3), 3.18 d (1H, SCH^A, $^2J_{H^AH^B}$ 16.8 Hz), 3.68 d (1H, SCH^B, $^2J_{H^AH^B}$ 16.8 Hz), 3.95–4.17 m (4H, CH_2), 7.04 t (1H, Ar, $^3J_{HH}$ 8 Hz), 7.31 t (2H, Ar, $^3J_{HH}$ 8 Hz), 7.49 m (3H, Ar), 7.62 d (1H, Ar, $^3J_{HH}$ 8 Hz), 7.83 d (2H, Ar, $^3J_{HH}$ 8 Hz). ^{19}F NMR spectrum ($CDCl_3$), δ_F, ppm: –111.89. ^{31}P NMR spectrum ($CDCl_3$), δ_P, ppm: 13.8. Found, %: P 6.23; S 13.17. $C_{19}H_{23}FNO_7PS_2$. Calculated, %: P 6.30; S 13.05.

{[(Diethoxyphosphoryl)(diethoxyphosphorylamino)(phenyl)methyl]thio}acetic acid VIIIa. Yield 81%, mp 109–112°C (from hexane). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.04 t (3H, CH_3, $^3J_{HH}$ 6.9 Hz), 1.2–1.4 m (9H, CH_3), 3.58 d (1H, SCH^A, $^2J_{H^AH^B}$ 12.9 Hz), 3.64 d (1H, SCH^B, $^2J_{H^AH^B}$ 12.9 Hz), 3.9–4.2 m (8H, OCH_2), 4.5 br.s (1H, NH), 7.4 m (3H, Ph), 7.7 d (2H, Ph). ^{31}P NMR spectrum ($CDCl_3$), δ_P, ppm: 5.3 d (PN, $^3J_{PP}$ 51 Hz), 18.4 d (PC, $^3J_{PP}$ 51 Hz). Found, %: P 12.90; S 6.68. $C_{17}H_{27}NO_8P_2S$. Calculated, %: P 13.20; S 6.83.

Diethyl α-(diethoxyphosphoryl)amino-α-(4-chlorophenylthio)benzylphosphonate VIIIb. Yield 93%, viscous oil. ^{31}P NMR spectrum (benzene), δ_P, ppm: 4.6 d (1P, PN, $^3J_{PP}$ 50 Hz), 17.3 d (1P, PC, $^3J_{PP}$ 50 Hz). Found, %: P 11.74; S 6.27. $C_{21}H_{30}ClNO_6P_2S$. Calculated, %: P 11.86, S 6.14.

Diethyl α-[(diethoxyphosphoryl)amino]-α-mercaptobenzylphosphonate VIIIc. Yield 83%, viscous oil. ^{31}P NMR spectrum (benzene), δ_P, ppm: 5.7 d (1P, PN, $^3J_{PP}$ 49 Hz), 17.9 d (1P, PC, $^3J_{PP}$ 49 Hz). Found, %: P 14.64; S 7.48. $C_{15}H_{27}NO_6P_2S$. Calculated, %: P 15.06; S 7.79.

Diethyl α-diphenylphosphinoyl-α-(phenylsulfonylamino)-4-fluorobenzylphosphonate IXa. Diphenylphosphine oxide, 0.12 g, was added to a solu-

tion of 0.29 g of imidoylphosphonate **IIIa** in 5 ml of benzene. After 1 h, the solvent was evaporated, and the residue was washed with petroleum ether. Yield 0.26 g (64%), mp 119–120°C. IR spectrum (KBr), ν, cm^{-1}: 1060 (POC), 1180, 1340 (S=O), 1250 (P=O), 3220 (NH). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 0.66 t (3H, CH_3, $^3J_{HH}$ 7.2 Hz), 1.19 t (3H, CH_3, $^3J_{HH}$ 7 Hz), 3.35 m (2H, CH_2), 3.99 m (1H, CH_2), 4.11 m (1H, CH_2), 6.68 t (2H, Ar, $^3J_{HH}$ 8 Hz), 7.09 d.d (1H, NH, $^3J_{HP}$ 16.8 Hz, $^3J_{HP}$ 9.3 Hz), 7.26 m (2H, Ar), 7.36–7.54 m (8H, Ar), 7.81–8.00 m (7H, Ar). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 15.5 d (CH_3, $^3J_{CP}$ 7 Hz), 16.2 d (CH_3, $^3J_{CP}$ 6 Hz), 62.1 d (CH_2, $^2J_{CP}$ 6 Hz), 64.1 d (CH_2, $^2J_{CP}$ 7 Hz), 68.4 d.d (CN, $^1J_{CPOEt}$ 160, $^1J_{CPPh}$ 49 Hz), 114.0 d.d (C^3, ArF, $^2J_{CF}$ 21, $^4J_{CR}$ 3.3 Hz), 127.2, 128.2, 128.3 (C^2, C^3, C^4, $PhSO_2$), 128.5 m (C^1, ArF), 128.9 d (C^1, PhP, $^1J_{CP}$ 63 Hz), 132.0, 133.3, 133.4 (C^2, C^3, C^4, PhP), 132.2 d.d (C^2, ArF, *J* 11 and 3 Hz), 142.5 s (C–SO_2), 162.1 d (C–F, $^1J_{CF}$ 251 Hz). ^{19}F NMR spectrum ($CDCl_3$), δ_F, ppm: –114.48. ^{31}P–{^{1}H} NMR spectrum ($CDCl_3$), δ_P, ppm: 16.2 d.d (EtOP, $^2J_{PP}$ 9.8, $^6J_{PF}$ 1.1 Hz), 38.0 d.d (PhP, $^2J_{PP}$ 9.8 Hz, $^6J_{PF}$ 5.3 Hz). Found, %: P 10.24; S 5.09. $C_{29}H_{30}FNO_6P_2S$. Calculated, %: P 10.30; S 5.33.

Bisphosphonates Xa–Xc. To a solution of 1 mmol of appropriate imidoylphosphonate **IV** in 5 ml of benzene, an equimolar amount of the hydrophosphoryl compound was added with stirring. The reaction mixture was kept at room temperature for 5 (R'' = PhO) or 14 (R'' = EtO) days. The solvent was removed in a vacuum.

Tetraethyl α-[(diethoxyphosphoryl)amino]-benzylidenephosphonate Xa. Yield 70%, viscous oil. ^{31}P NMR spectrum (benzene), δ_P, ppm: 4.8 t (1P, PN, $^3J_{PP}$ 22 Hz), 18.5 d (2P, PC, $^3J_{PP}$ 22 Hz). Found, %: P 17.89. $C_{19}H_{36}NO_9P_3$. Calculated, %: P 18.02.

***O,O*-Diphenyl *O'*,*O'*-diethyl α-[(diethoxyphosphoryl)amino]-4-chlorobenzylidenebisphosphonate Xb.** Yield 75%, viscous oil. ^{31}P–{^{1}H} NMR spectrum ($CDCl_3$), δ_P, ppm: 4.4 d.d (1P, PN, $^3J_{PNCPOPh}$ 35, $^3J_{PNCPOEt}$ 16.5 Hz), 10.1 d.d (1P, PCOPh, $^2J_{PP} \approx {}^3J_{PP} \approx$ 16.5 Hz), 17.2 d.d (1P, PCOEt, $^2J_{PP}$ 16.5, $^3J_{PP}$ 35 Hz). Found, %: P 14.07. $C_{27}H_{35}ClNO_9P_3$. Calculated, %: P 14.38.

Tetraphenyl α-[(diethoxyphosphoryl)amino]-4-chlorobenzylidenebisphosphonate Xb. Yield 80%, viscous oil. ^{31}P–{^{1}H} NMR spectrum ($CDCl_3$), δ_P, ppm: 2.0 t (1P, PN, $^3J_{PP}$ 27 Hz), 6.8 d (2P, PC, $^3J_{PP}$ 27 Hz). Found, %: P 12.27. $C_{35}H_{35}ClNO_9P_3$. Calculated, %: P 12.52.

Diethyl α-[bis(diethoxyphosphoryl)amino]benzylphosphonate XI. Bisphosphonate **Xa** was applied onto a thin nonfixed layer of Silicagel 60 (0.040–0.063 mm, Merck) on a glass plate and eluted with ethyl acetate. After drying, the plate was developed with iodine vapor for 15 min. The band containing phosphonate **XI** was washed out with methanol, and after evaporation of the solvent a clear viscous oil was obtained, yield 72%. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.2–1.3 m (18H, CH_3), 4.0–4.3 m (12H, OCH_2), 5.2 d.t (1H, CHP, $^2J_{HP}$ 27.3, $^3J_{HP}$ 20.2 Hz), 7.3 m (3H, Ph), 7.7 m (2H, Ph). ^{13}C NMR spectrum ($CDCl_3$, ppm: 15.8 d (CH_3, $^3J_{CP}$ 4 Hz), 15.9 d (CH_3, $^3J_{PC}$ 4 Hz), 16.1 d (CH_3, $^3J_{CP}$ 6.5 Hz), 16.2 d (CH_3, $^3J_{CP}$ 5 Hz), 59.3 d (CP, $^1J_{CP}$ 164 Hz), 61.3 d (CH_2, $^2J_{CP}$ 7.5 Hz), 63.3 d (CH_2, $^2J_{CP}$ 4 Hz), 63.4 d (CH_2, $^2J_{CP}$ 4 Hz), 64.1 d (CH_2, $^2J_{CP}$ 7.5 Hz), 127.8 s (C^3, Ph), 128.0 s (C^4, Ph), 130.3 d (C^2, Ph, $^3J_{CP}$ 5.8 Hz), 135.1 (C^1, Ph). ^{31}P–{^{1}H} NMR spectrum ($CDCl_3$), δ_P, ppm: 3.9 d (2P, PN, $^3J_{PP}$ 13 Hz), 22.1 t (1P, PC, $^3J_{PP}$ 13 Hz). Found, %: P 17.91. $C_{19}H_{36}NO_9P_3$. Calculated, %: P 18.02.

Diethyl α-(4-bromophenylamino)-α-[(diethoxyphosphorylamino]benzylphosphonate XII. Yield 60%, viscous oil. ^{31}P–{^{1}H} NMR spectrum ($CDCl_3$), δ_P, ppm: 3.5 d (1P, PN, $^3J_{PP}$ 7 Hz), 18.0 d (1P, PC, $^3J_{PP}$ 7 Hz). Found, %: Br 15.07; P 11.25. $C_{21}H_{31}Br \cdot N_2O_6P_2$. Calculated, %: Br 14.55; P 11.28.

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