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Synthesis of the New Types of N-Substituted Aminomethylenebisorganophosphorus Acids and Their Derivatives

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ABSTRACT: *The interaction of esters of trivalent organophosphorus acids containing PH and $POSiMe_3$ fragments with various derivatives of formamide is proposed as convenient methods for the synthesis of new N-substituted aminomethylenebisorganophosphorus acids and their derivatives with three-, four-, and five-coordinated phosphorus. Also the new functionalized derivatives of the new aminomethylenebisphosphinic acids with substituted hydroxymethyl moieties are synthesized, and some properties of the obtained compounds are presented.*
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INTRODUCTION

The numerous aminomethylenebisorganophosphorus compounds present a great interest as promising polydentate ligands and prospective biologically active compounds [1–7]. In the present work, we report the results of the interaction of the esters of trivalent organophosphorus acids with PH and $POSiMe_3$ fragments with various derivatives of formamide resulting in formation of the new types of N-substituted aminomethylenebisorganophosphorus acids and their derivatives in high yield. Also for the first time we have prepared the aminomethylenebisorganophosphorus compounds with three- and five-coordinated phosphorus.

RESULTS AND DISCUSSION

An excess of dibutoxyphosphine slowly reacts with dimethylformamide dimethylacetal at 20°C, and this reaction is easily completed under heating of the reaction mixture at 130°C giving aminomethylenebisphosphonite **1** (Eq. (1)). The suggested scheme of this reaction includes an electrophilic attack of acetal on the trivalent phosphorus followed by the decomposition of the phosphorane intermediate with methanol being eliminated.

$$2\,(BuO)_2PH \xrightarrow{(MeO)_2CHNMe_2} [(BuO)_2\overset{\displaystyle OMe}{\underset{\displaystyle H}{P}}]_2CHNMe_2 \xrightarrow[-2MeOH]{} [(BuO)_2P]_2CHNMe_2 \quad (1)$$

1

Several aminomethylenebisphosphonites **2–5** are synthesized by the unique reaction of diethyl pivaloylphosphonite with dialkylformamide diethylacetals in the presence of excess ethanol and zinc chloride as a catalyst at 80–110°C with a good yields (Eq. (2)). Thus diethyl pivaloylphosphonite containing labile P—C bond (cf. [8,9]) may be successfully applied in aminomethylation as a synthetic analog of unstable diethoxyphosphine. The

Dedicated to Prof. Ivan F. Lutsenko (1912–1993).

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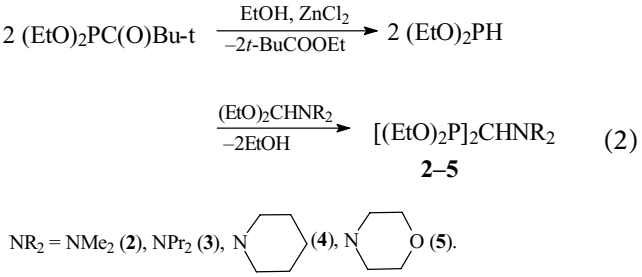
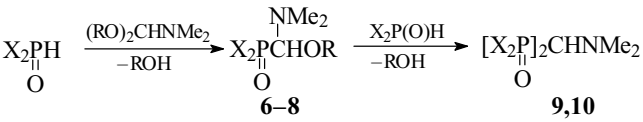
proposed scheme of this reaction involves the cleavage of the P—C bond of the pivaloylphosphonite by ethanol, resulting in diethoxyphosphine, which undergoes further aminomethylation similarly to dibutoxyphosphine.



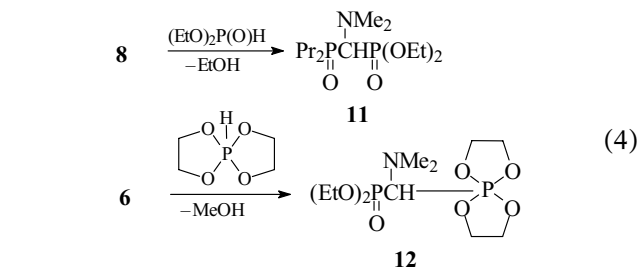
(2)

Also it was shown by us that the interaction of P(O)H acids with dimethylformamide dialkyl acetals proceeds as a two-step process. So diethyl phosphite and dipropyl phosphinoxide react with formamide acetals under heating at 110–130°C, giving mono- or bisorganophosphorus compounds **6–10** (Eq. (3); cf. [10,11]).



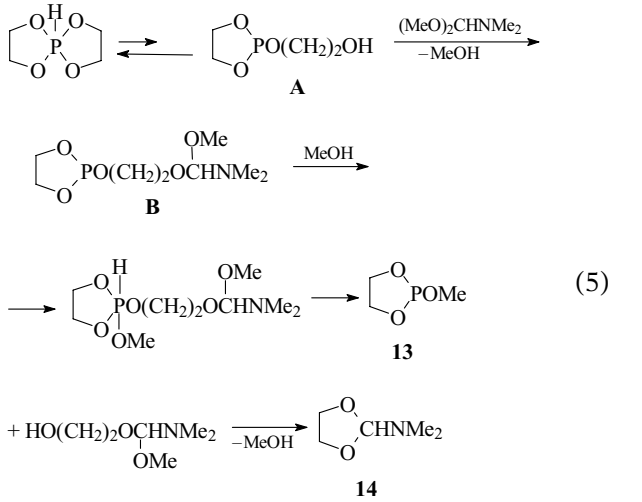
(3)

The compounds **6** and **8** were used as aminomethylating reagents to obtain aminomethylenebisorganophosphorus substances with two different phosphorus-containing groups. So, diethyl phosphite easily reacts with phosphinoxide **8** yielding nonsymmetrical compound **11**, and methoxy(dimethylamino)methylphosphonate **6** reacts smoothly with hydrospirophosphorane to give the aminomethylenebisphosphorus compound **12** with tetra- and penta-coordinated phosphorus (Eq. (4)).



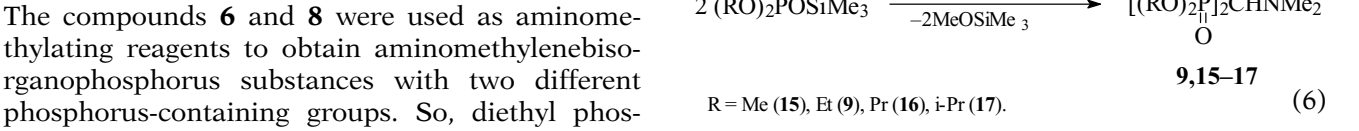
(4)

The proposed scheme of last reaction involves an electrophilic attack of an aminomethylating reagent on the trivalent phosphorus of hydrospirophosphorane tautomer **A** in the common manner (cf. [12]); the elimination of methanol followed by the rebuilding of the phosphorane structure of compound **12**. Another route of the similar reaction was realized by the interaction of hydrospirophosphorane with dimethylformamide dimethylacetal under the same conditions. There is no P—C bond formation, but only trans-esterification of acetal with tautomer **A** occurs yielding via intermediate **B** the mixture of cyclic compounds **13** and **14** (Eq. (5)).



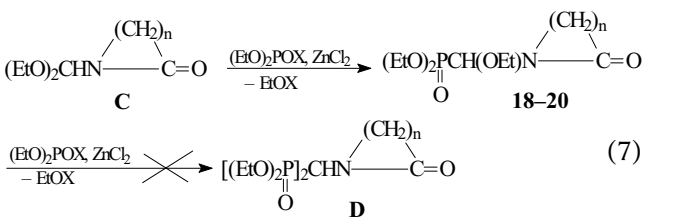
(5)

The excess of dialkyl trimethylsilyl phosphites easily reacts with dimethylformamide acetal in the presence of zinc chloride by heating at 130°C, giving bisphosphonates **9,15–17** in high yields (cf. [13] (Eq. (6)).



(6)

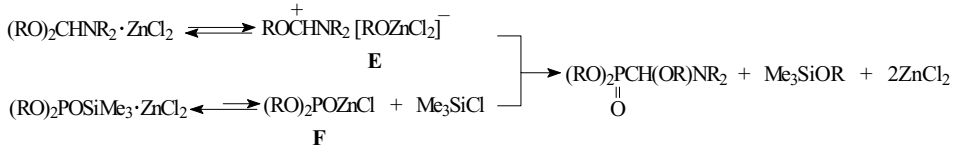
Also in the present work, we propose a convenient synthesis of amido(ethoxy)methylphosphonates **18–20** containing pyrrolidone, valero- and caprolactams fragments. Hence *N*-diethoxymethyl lactams **C**, which were obtained by the procedure as described in [14], react with diethyl trimethylsilyl and diethyl phosphites at 130–150°C in the presence of zinc chloride to form phosphonates **18–20** (Eq. (7)). Using diethyl trimethylsilyl phosphite is optimal and provides high yields of phosphonates, whereas with diethyl phosphite the yield is much lower.



(7)

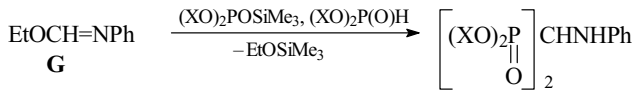
Using an excess of phosphites in this reaction unfortunately did not lead to formation of diphosphorylation products, amidomethylenebisphosphonates **D**. This is connected with the fact that

phosphonates **18–20** are much weaker aminomethylating agents compared with substituted amino(alkoxy)methylphosphonates **6** and **8** because of the electronic and steric effects of the lactam-containing fragments. Evidently, the catalytic effect of zinc chloride is connected with its ability to generate electrophilic carbonio-immonium ions **E** as well as nucleophilic zinc-containing salts **F** in the course of the process (Eq. (8)).



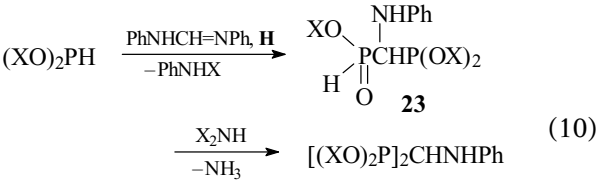
(8)

The new derivatives of *N*-anilinomethylenebisorganophosphorus acids were synthesized by us via interaction of *N*-phenyl ethoxymethylenimine **G** and *N*,*N*′-diphenylformamidine **H** [15] with trimethylsilyl esters of phosphorous and hypophosphorous acids. Hence, imine **G** reacts with a mixture of diethyl- and diethyl-trimethylsilyl phosphites in the presence of boron trifluoride-diethyl etherate as a catalyst for 2 weeks at 20°C, yielding bisphosphonate **21** as white crystals. The similar reaction of imine **G** with a mixture of bis- and tris(trimethylsilyl)phosphites proceeds by heating at 140°C in the presence zinc chloride, giving bisphosphonate **22** in high yield (Eq. (9)).



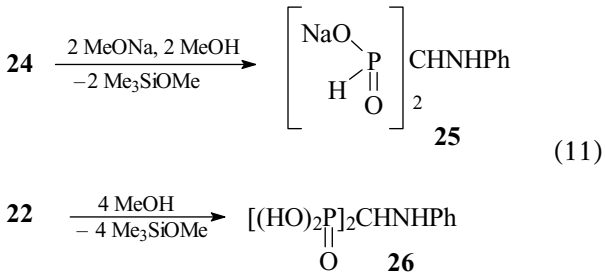
X = Et (**21**), Me_3Si (**22**). (9)

Evidently, there are two simultaneous reactions of the addition to CH=N fragment and substitution of the ethoxy group in the course of this process (cf. [12]). The excess of bis(trimethylsiloxy)phosphine smoothly reacts with formamidine **H** at 140°C to give phosphonite **23** in high yield that was easily silylated by bis(trimethylsilyl)amine to form bisphosphonite **24** (Eq. (10); cf. [16]).



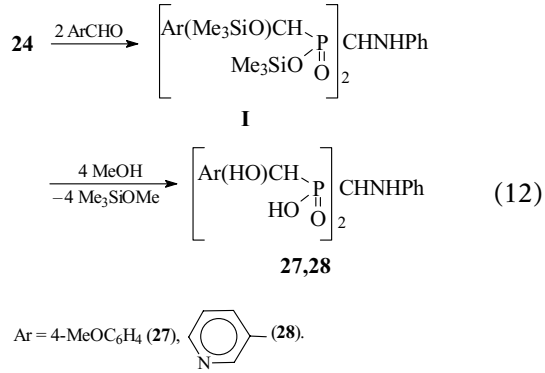
X = Me_3Si. (10)

Also the treatment of bisphosphonite **24** with a diluted solution of sodium methylate in methanol results in formation of water-soluble disodium bisphosphonite **25** as white hygroscopic crystals. Under similar conditions, bisphosphonate **22** was easily transformed to corresponding bisphosphonic acid **26** via treatment with methanol in high yield (Eq. (11)).



(11)

Bisphosphonite **24** is a key synthon for obtaining the several functionalized bisphosphinates. So bisphosphonite **24** readily adds by the carbonyl group of aldehydes in methylene chloride to give phosphinates **I** as intermediates (cf. [17]), and functionalized bisphosphinic acids **27** and **28** were prepared in high yields by the treatment of the reaction mixture with methanol (Eq. (12)).



Ar = 4-$MeOC_6H_4$ (**27**), (**28**). (12)

The synthesized compounds **1–28** are the promising synthons, polydentate ligands and biologically active substances. The structures of N-substituted aminomethylenebisorganophosphorus acids and their derivatives. were confirmed by the ^1H, ^{13}C, ^{31}P NMR spectra, which show characteristic signals of the $PC^1H(NC^2)P$ fragments (see Table 1). The

TABLE 1 Yields, Products Constants and NMR Spectral Data (δ, ppm, J, Hz) for the $PC^1H(NC^2)P$ Fragments[a] of Compounds **1–28**[a]

Compound	Yield (%)	Bp,°C (p, mmHg) (mp, °C)	δ(H) C^1H t	$^2J_{PH}$	δ (C^1) t	$^1J_{PC}$	δ(C^2) t	$^3J_{PC}$	δ_P, s[b]
1	62	145 (1)	3.22	4.7	74.45	31.0	45.23	7.2	191.04
2	77	90 (1)	3.19	4.2	74.14	31.1	45.00	5.1	186.70
3	72	102 (1)	3.40	3.3	71.90	29.8	56.93	7.0	187.50
4	71	106 (1)	3.16	3.7	75.48	31.7	54.08	7.5	184.74
5	71	116 (1)	3.06	4.7	74.65	31.5	53.37	6.7	185.12
6	78	78 (1)	4.04 d	7.2	94.15 d	196.7	40.85 d	9.8	17.31
7	82	83 (1)	4.02 d	7.3	94.26 d	197.5	40.48 d	8.4	17.52
8	69	100 (1), (45)	3.88 d	1.3	93.78 d	76.8	42.98 d	5.7	45.61
9	72	126 (1)	3.43	24.5	61.95	138.8	44.23	5.1	17.80
10	78	142 (1), (75)	3.19	16.8	61.98	53.9	44.99	3.2	54.06
11	74	135 (1), (64)	3.13 dd	15.6, 23.2	62.59 dd	55.4, 138.9	43.65 dd	3.2, 5.8	51.94 d[c], 23.83 d
12	82	149 (1)	[d]	–	68.80 dd	176.3, 139.6	44.41	7.7	−11.25 d[c], 22.51 d
15	77	122 (1)	[d]	–	61.08	139.2	44.09	5.0	21.01
16	72	152 (1)	3.42	24.3	61.90	138.4	44.26	5.1	18.08
17	73	123 (0.5)	3.17	24.5	62.97	141.5	44.22	4.8	17.70
18	78	142 (1)	5.14 d	8.8	76.11 d	202.0	64.40	14.6	15.91
19	74	148 (1)	5.76 d	9.2	76.83 d	200.3	64.49	15.2	16.55
20	64	157 (1)	5.68 d	9.2	77.42 d	202.6	64.73	15.2	16.77
21	59	182 (1), (74)	4.24[e]	24.0	50.35	147.0	146.15 s	–	17.30
22	86	157 (0.5)	[d]	–	51.40	154.2	143.13 s	–	−1.31
23	83	157 (1)	m	–	61.86 dd	93.2, 39.1	147.66 s	–	19.45 d[c], 147.08 d
			m	–	62.12 dd	101.0, 45.8	148.00 s	–	22.58 d[c], 147.27 d
24	78	154 (1)	m	–	71.09	38.5	149.33 s	–	153.77
25	94	*f*	3.64	16.4	56.24	85.3	147.80 s	–	20.06
26	97	(182)	4.20	22.2	50.95	142.4	147.03 s	–	16.99
27	87	f	4.71	16.2	48.39	78.7	147.68 s	–	38.17 d[c], 39.07 d
			4.78	16.0	48.59	81.2	147.85 s	–	39.80
			4.41	16.2	49.64	79.8	148.09 s	–	39.08
28	89	f	4.14	16.4	50.24	84.8	146.33 s	–	29.98 d[c], 31.19 d
			4.31	16.0	48.51	86.9	146.91 s	–	30.22
			3.92	16.3	51.89	87.6	146.10 s	–	30.67

[a] For compounds, n_D^{20}:**6,** 1.4362; **7,** 1.4359; **9,** 1.4500; **12,** 1.4780; **15,** 1.4630; **16,** 1.4492; **17,** 1.4425; **18,** 1.4615; **19,** 1.4660; **20,** 1.4687. All signals of alkyl, aryl, pyridine, and trimethylsilyl groups are in the standard area. The ^{1}H NMR spectra of products fragments show expected signals that look like sometimes as overlapping multiplets. According to the NMR spectra, the compound **23** is the mixture of two stereoisomers, and the compounds **27** and **28** are the mixtures of three stereoisomers. Their ratio was determined from the ^{1}H and ^{31}P NMR spectra. The spectral parameters of the major isomer are given first. The ratio for compounds: **23**, 60:40; **27**, 40:35:25; **28**, 40:35:25. Two phosphorus groups are nonequivalent for the first stereoisomer of compounds **27** and **28**. In ^{1}H NMR spectra, fragment NH for compounds: **21**,4.89 broad s; **23**, 4.29 dd, $^3J_{HH}$ 8,$^4J_{HH}$ 2 and 4.22 d, $^3J_{HH}$10, **24**, 4.44 d, $^3J_{HH}$ 8; fragment PH for compound: **23**, 7.04 d, $^1J_{PH}$ 560 and 7.02 d, $^1J_{PH}$ 560; fragment PCHOH for compounds: **27**, 4.89 d, $^2J_{PH}$ 16.1; **28**, 4.95 d, 4.97 d, 5.05 d, all $^2J_{PH}$ 12.4. In ^{13}C NMR spectra, fragment NC(O) for compounds: **18**, 175.19 d, $^3J_{PC}$ 6.0; **19**, 169.89 d, $^3J_{PC}$ 5.9; **20**, 176.13 d, $^3J_{PC}$ 5.1; fragment PCHOH for compounds: **27**, 70.27 d, $^1J_{PC}$ 103.3, 70.96 d, $^1J_{PC}$ 105.2, and 69.20 d, $^1J_{PC}$ 106.4; **28**, 69.74 d, $^1J_{PC}$ 104.2, 69.97 d, $^1J_{PC}$ 106.4, and 69.13 d, $^1J_{PC}$ 97.8.

[b] Data of $^{31}P\{^1H\}$ spectra.

[c] $^2J_{PP}$ for compounds: **11**, 38.5; **12**, 78.3; **23**, 43.7 and 48.6; **27**, 24.3; **28**, 20.3.

[d] The signals these fragments are overlapping multiplets.

[e] With addition of the one drop of HCOOH to NMR specimen.

[f] This compound is decomposed by heating at 150°C.

TABLE 2 Elemental Analyses Data of Compounds **1–28**[a]

Compound	Empirical Formula	Formula Weight	Calcd. (%)		Found (%)	
			C	H	C	H
1	$C_{19}H_{43}NO_4P_2$	411.51	55.46	10.53	55.09	10.37
2	$C_{11}H_{27}NO_4P_2$	299.29	44.15	9.09	44.01	9.03
3	$C_{15}H_{35}NO_4P_2$	355.39	50.69	9.93	50.55	9.89
4	$C_{14}H_{31}NO_4P_2$	339.35	49.55	9.21	49.64	9.18
5	$C_{13}H_{29}NO_5P_2$	341.32	45.75	8.56	45.81	8.49
6	$C_8H_{20}NO_4P$	225.23	42.66	8.95	42.52	8.87
7	$C_9H_{22}NO_4P$	239.25	45.18	9.27	44.86	8.95
8	$C_{11}H_{26}NO_2P$	235.31	56.15	11.14	55.88	11.02
9	$C_{11}H_{27}NO_6P_2$	331.29	39.88	8.21	39.64	8.12
10	$C_{15}H_{35}NO_2P_2$	323.39	55.71	10.91	55.49	11.07
11	$C_{13}H_{31}NO_4P_2$	327.34	47.70	9.55	47.54	9.26
12	$C_{11}H_{25}NO_7P_2$	345.27	38.27	7.30	38.14	7.49
15	$C_7H_{19}NO_6P_2$	275.19	30.55	6.96	30.43	6.83
16	$C_{15}H_{35}NO_6P_2$	387.41	46.51	9.11	46.40	9.03
17	$C_{15}H_{35}NO_6P_2$	387.41	46.51	9.11	46.42	9.06
18	$C_{11}H_{22}NO_5P$	279.27	47.31	7.94	47.03	7.81
19	$C_{12}H_{24}NO_5P$	293.30	49.14	8.25	48.87	8.03
20	$C_{13}H_{26}NO_5P$	307.33	50.81	8.53	50.64	8.42
21	$C_{15}H_{27}NO_6P_2$	379.34	47.50	7.17	47.29	7.08
22	$C_{19}H_{43}NO_6P_2Si_4$	555.85	41.06	7.80	40.94	7.74
25	$C_7H_9NNa_2O_4P_2$	279.09	30.13	3.25	30.03	3.28
26	$C_7H_{11}NO_6P_2$	267.13	31.48	4.15	31.37	4.19
27	$C_{23}H_{27}NO_8P_2$	507.42	54.44	5.36	54.26	5.28
28	$C_{19}H_{21}N_3O_6P_2$	449.35	50.79	4.71	50.57	4.78

[a]The air-sensitive compounds **23** and **24** were analyzed as their air-stable derivative **25**. The compounds **13** and **14** were obtained as a mixture.

elemental analysis data of synthesized compounds are summarized in Table 2.

EXPERIMENTAL

The ^1H, ^{13}C, and ^{31}P NMR spectra were registered on the Varian VXR-400 and Bruker Avance-400 spectrometer (400, 100, and 162 MHz, respectively) in C_6D_6, $CDCl_3$ (**1–24**) or CD_3OD, $(CD_3)_2SO$, D_2O(**25–28**)against TMS (^1H and ^{13}C) and 85% H_3PO_4 in D_2O (^{31}P). All reactions were carried out under dry argon in anhydrous solvents. The starting derivatives of formamide were prepared as described in [15], and the starting esters of trivalent phosphorus acids were discussed in [12].

O, *O*, *O*, *O-Tetrabutyl Dimethylaminomethylenebisphosphonite* (**1**). A mixture of 6.3 g of dibutoxyphosphine and 2.7 g of dimethylformamide dimethylacetal was heated to 120–130°C for 1 h and then distilled in a vacuum to obtain 4.5 g of phosphonite **1**.

Phosphonites **2–5** and **23** were prepared similarly. Phosphonites **2–5** were synthesized in the presence of 2 mL of ethanol and 0.1 g of zinc chloride, phosphonite **23** was synthesized without catalyst at 140°C.

Dipropyl Ethoxy(dimethylamino)methyl Phosphinoxide (**8**). A mixture of 7 g of dipropyl phosphinoxide and 7.7 g of dimethylformamide dimethylacetal was heated to 110–130°C for 2.5 h and then distilled in a vacuum to obtain 8.5 g phosphinoxide **8**.

The compounds **6,7,9–12** were prepared similarly. At the same conditions from the mixture of 9 g of hydrospirophosphorane **A** and 9.5 g of dimethylformamide dimethylacetal, it was obtained 7 g of the mixture 2-methoxy-1,3,5-dioxaphospholane **13**, δ_P 132.3 ppm and 2-dimethylamino-1,3-dioxolane **14**, δ_H(CH) 5.33 ppm, bp 30–34°C at 1 mmHg (cf. [18,19]).

O, *O*, *O*, *O-Tetramethyl Dimethylaminomethylenebisphosphonate* (**15**). A mixture of 16.1 g of dimethyl trimethylsilyl phosphite, 4.8 g of dimethylformamide dimethylacetal, and 0.2 g zinc chloride was heated to 130°C for 1.5 h and then distilled in a vacuum to obtain 8.5 g of bisphosphonate **15**.

The compounds **9,16–20** were prepared similarly.

O, *O*, *O*, *O-Tetraethyl N-Anilinomethylenebisphosphonate* (**21**). To a stirredmixture of diethyl phosphite (4.3 g), diethyl trimethylsilyl phosphite (8.4 g), and *N*-phenyl ethoxymethylenimine (4.5 g), boron trifluoride diethyl etherate (0.1 g) was added. The mixture was stirred for 0.5 h and was left for

2 weeks. The white crystals were washed with the mixture of hexane, 5 mL, and diethyl ether, 5 ml, then were kept in a vacuum of 1 mmHg for 1 h to obtain 6.8 g of bisphosphonate **21**.

O, *O*, *O*, *O-Tetra(trimethylsilyl) N-Anilinomethylenebisphosphonate* (**22**). A mixture of bis(trimethyl)phosphite (3.3 g), tris(trimethylsilyl)phosphite (10 g), *N*-phenyl ethoxymethylene imine (3.3 g), and zinc chloride (0.1 g) was heated to 150°C for 1 h and then it was distilled in a vacuum to obtain 10.6 g of bisphosphonate **22**.

O, *O*, *O*, *O-Tetra(trimethylsilyl) N-Anilinomethylenebisphosphonite* (**24**). A mixture of bisphosphonite **23** (13.7 g) and bis(trimethylsilyl)amine (31 g) was heated under reflux with stirring for 1 h, and the residue was distilled to obtain 12.5 g of bisphosphonite **24**.

Disodium N-Anilinomethylenebisphosphonite (**25**). To solution of 0.02 g of sodium methylate in 20 mL of methanol, a solution of 3 g of bisphosphonite **24** in 10 mL of ether was added with stirring at 5°C. The resulting mixture was heated to boiling, the solvent was removed, and residue was kept in a vacuum for 1 h (1 mmHg) to give 1.5 g of salt **25**.

N-Anilinomethylenebisphosphonic Acid (**26**). A solution of bisphosphonate **22**, 10.6 g in ether (10 mL) was added with stirring to 50 mL of methanol cooled to 10°C. The mixture was heated to boiling, the solvent was distilled off, and residue was kept in a vacuum (1 mmHg) for 1 h to obtain 5 g of salt **26**.

N-Anilinomethylenebis[hydroxy(pyrid-3-yl)methylphosphinic] Acid (**28**). A solution of 1.6 g of 3-pyridinecarboxaldehyde in 10 mL of methylene chloride, was added with stirring and cooling to 10°C to a solution of 3.5 g of bisphosphonite **24** in 10 mL of methylene chloride. The solvent was removed, and the mixture of methanol (10 mL) and diethyl ether (20 mL) was added to the residue, and the mixture was heated to boiling. The crystals were filtered off, washed with ether, and exposed to a vacuum of 1 mmHg for 1 h to obtain 2.7 g of acid **28**.

Acid **27** was prepared similarly.

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