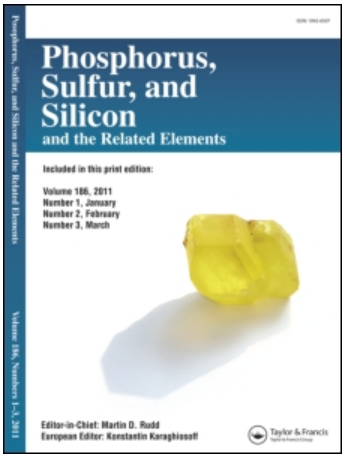

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Synthesis and Characterization of Alkoxyethoxyphosphoryl Amines

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Synthesis and Characterization of Alkoxyethoxyphosphoryl Amines

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A series of potential polidentate ligands were developed. The alkoxyethoxyphosphorylamines were prepared by phosphorylation of different amines with the appropriate dialkoxyethyl phosphite in a two-phase reaction. The characterization of these compounds was carried out by IR, Mass, and NMR spectroscopy. The properties of these compounds as a chelating agent were investigated by direct UV titration technique.

Keywords Dialkoxyethoxyphosphorylamine; dialkoxyalkyl phosphite; dialkoxyethoxyphosphorylguanidine; phosphorylation

INTRODUCTION

Bidentate compounds containing a 1-phosphoryl-3-carbonyl group are known to form colorful complexes with various metal ions.[1] They are powerful extractants for trivalent lanthanides and actinides, as well as alkali metal cations.[2,3] An alternation of the molecular structure might improve the extracting properties of these compounds, such as the chelating capacity as well as their selectivity towards different metal ions. We have reported the syntheses and preliminary complexation studies of the dialkoxyphosphoryl- guanidines and isothioureas.[4–6] Now, we will describe the synthesis and structure analysis of the potential polydentate alkoxy- and alkoxyethoxy- phosphoryl amines.

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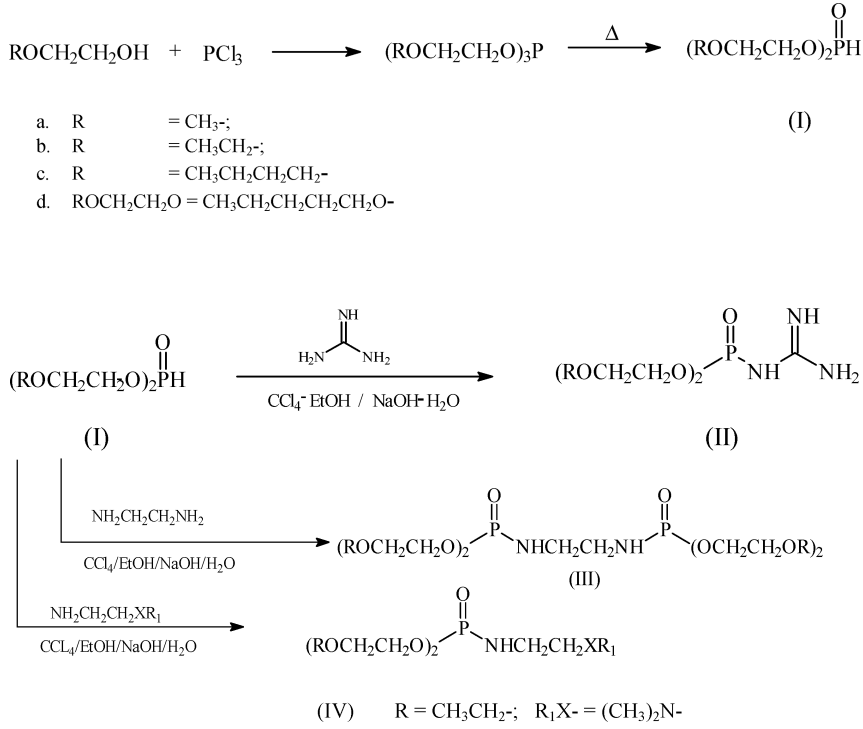
RESULTS AND DISCUSSION

The reaction of alkoxyethanol with phosphorus trichloride without a solvent gave the desired alkoxyalkyl phosphite (I) in good yield. Phosphites of high purity were obtained by vacuum distillation. The reaction of guanidine with the dialkoxyalkyl phosphite in a basic condition in a two-phase reaction involving carbon tetrachloride and water at room temperature led to the formation of phosphorylguanidine (II). The phosphorylation of ethylenediamine and its derivative with the prepared phosphites (I) under similar conditions resulted in the potential polydentate ligands (III) and (IV), respectively.



Phosphorylamines (II), (III), and (IV) showed characteristic IR absorption peaks at around 3400 (N-H), 1200 (P═O) and 1000 (P-O-C) cm^{-1}. The ^1H—NMR spectra showed the characteristic triplet peak at around $\delta = 1.00$ ppm for the terminal methyl group and a symmetric multiplet at around $\delta = 4.00$ ppm for the hydrogen atom of —CH_2OP—, suggesting the presence of the coupling between phosphorus and hydrogen nuclei.[6,7] All the hydrogen atoms connected to amino nitrogen appeared as a broad peak at around $\delta = 6.0$ ppm. The phosphites (I)

TABLE I Spectroscopic Data for Phosphites and Phosphorylamines (I)–(III)

Compound	$ROCH_2CH_2O—$	^{31}P-NMR	Mass (M^++1)	Mass Common peak
Ia	$CH_3—OCH_2CH_2O—$	7,20	199	83; 65
Ib	$CH_3CH_2—OCH_2CH_2O—$	5,41	227	83; 65
Ic	$CH_3CH_2CH_2CH_2—OCH_2CH_2O—$	6,98	283	83; 65
Id	$CH_3CH_2CH_2CH_2CH_2O—$	4,00	223	83; 65
IIa	$CH_3—OCH_2CH_2O—$	8,68	256	140; 123
IIb	$CH_3CH_2—OCH_2CH_2O—$	8,42	284	140; 123
IIc	$CH_3CH_2CH_2CH_2—OCH_2CH_2O—$	8,66	340	140; 123
IId	$CH_3CH_2CH_2CH_2CH_2O—$	6,80	280	140; 123
IIIa	$CH_3—OCH_2CH_2O—$	11,42	453	226; 220; 110
IIIb	$CH_3CH_2—OCH_2CH_2O—$	11,65	509	254; 220; 110
IIIc	$CH_3CH_2CH_2CH_2—OCH_2CH_2O—$	11,27	620	310; 220; 110
IIId	$CH_3CH_2CH_2CH_2CH_2O—$	10,75	500 (M^+)	250; 220; 110

as well as the phosphorylated guanidines (II) and symmetric phosphorylamines (III) showed the molecular ion plus one peak in their mass spectra. Table I shows the spectroscopic data for phosphites (I) and the phosphorylamines (II) and (III). These compounds demonstrated fragmentation by McLafferty rearrangement to give characteristic common fragments of m/e = 83 and 65 (Fig. 1) from phosphite (I) and common peaks with high intensity at m/e = 140 from compounds (II). The phosphorylated guanidines (II) also presented a characteristic α-cleavage, eliminating one ammonia molecule to give a phosphorylcyanamide of m/e = 123 (Figure 1). All the symmetric phosphorylated diamines (III) showed the same type of fragmentation to give peaks of high intensity at m/e = $M^+/2$, 220 and 110. Figure 2 shows the classical fragmentations, which lead to these components.

Compounds containing a di-(*iso*-propoxy)phosphorylguanidine present a strong absorption peak at around 210 nm and a much weaker peak at around 250 nm. When a solution of the metal ion is added, the spectrum shows an increase or decrease in its absorption intensity or the appearance of new peaks if a metal complex is formed[5]. No change in the spectrum is observed if there is no complex formation. The prepared phosphorylamines (II) and (III) present UV absorption at 202 nm and 254 nm. The preliminary complexation studies by direct UV titration method showed that the developed potential ligands can selectively chelate metal ions such as Cu^{2+}, Co^{2+}, Cd^{2+}, Hg^{2+}, Pb^{2+}, Fe^{3+}, Zn^{2+}, Ni^{2+} and Al^{3+} in a neutral solution.

We now carry out a detailed study about the behavior of the developed ligand towards different metal ions as well as the correlation between

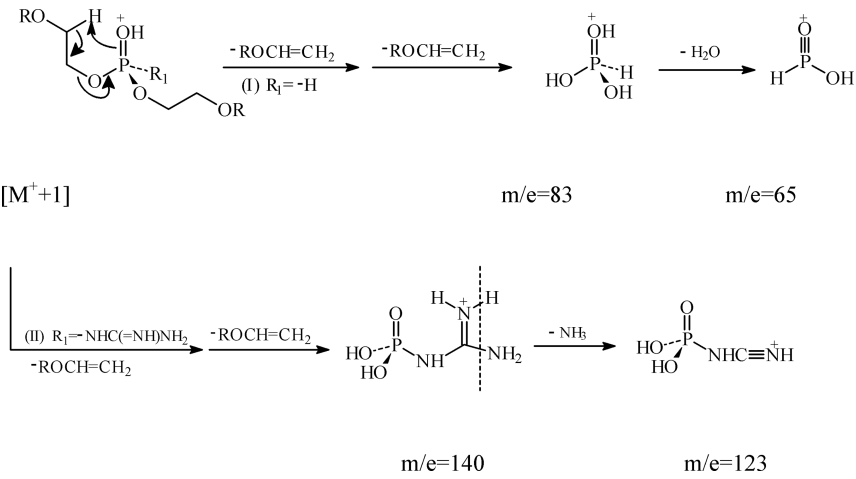


FIGURE 1 Fragmentation of dialkoxyalkyl phosphite (I) and phosphoryl-guanidine (II).

the ligand structure and metal ions. The results should offer guideline for the further development of new ligands and information about its application in practical life.

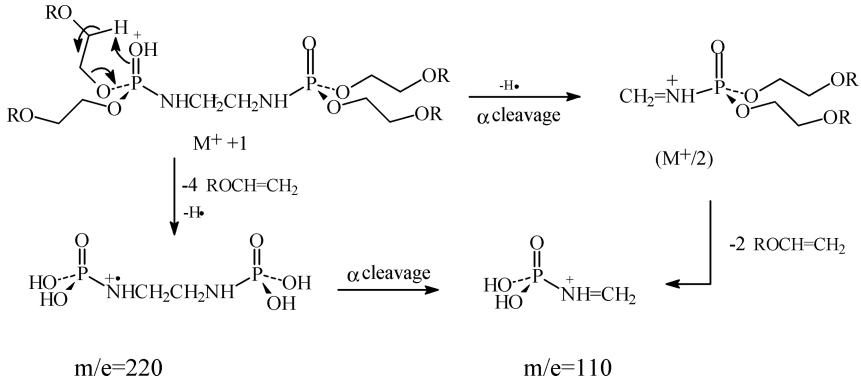


FIGURE 2 Fragmentation of symmetric dialkoxyalkylphosphorylamines (III).

EXPERIMENTAL

The NMR spectra (^{1}H, ^{13}C, and ^{31}P) were recorded on a Bruker AC300 spectrometer. All chemical shifts are reported with respect to TMS or H_3PO_4. IR spectra were obtained on a Perkin-Elmer Model 1710

spectrometer. Mass spectra were taken on a Varian MAT 8500 (70 eV) spectrometer. Melting points were taken on a Fisher-Johns melting point apparatus without correction.

Dialkoxyalkyl Phosphite— General Procedure

Dialkoxyethanol (1.2 mol) was cooled to 5°C and phosphorus trichloride (0.4 mol) was added drop by drop at this temperature. After the addition, the mixture was stirred at room temperature for two hours and then kept under reflux for another 2 h. The excess HCl formed during the reaction was removed by strong agitation under vacuum for two hours to give the crude product.

Di-(2-methoxy)ethyl phosphite (Ia)

Following the general procedure, 2-methoxyethanol (45.6 g, 0.6 mol) and phosphorus trichloride (27.5 g, 0.2 mol) were used; the obtained crude product was distilled at reduced pressure (125–128°C, 1 mmHg) to give a clear transparent liquid in 60% yield. IR(cm^{-1}): 2950; 2435(P—H), 1254(P=O); 1052(P—O). ^{1}H NMR ($CDCl_3$): 3.40(s, 6H, —OCH_3); 3.61(t, J = 4.80Hz, 4H, —OCH_2); 4.17–4.32 (m, 4H, —OCH_2); 6.94(d, J = 717.3Hz, 1H, P—H). ^{13}C NMR: 58.69; 64.22; 71.15. ^{31}P NMR: 7.20. Mass Spec. 199 (M^++1); 141; 83; 65; 58.

Di-(2-ethoxy)ethyl phosphite (Ib)

Following the general procedure, 2-ethoxyethanol (54.0 g, 0.6 mol) and phosphorus trichloride (27.5 g, 0.2 mol) were used; the obtained crude product was distilled at reduced pressure (132–134°C, 3 mmHg) to give a clear transparent liquid in 57% yield. IR(cm^{-1}): 2950; 2438(P—H), 1259(P=); 1127 (C—O); 1054(P—O). ^{1}H NMR ($CDCl_3$): 0.97 (t, J = 6.9Hz, 6H, —CH_3); 3.29(q, J = 5.8Hz, 4H, —OCH_2); 3.41(t, J = 6.90Hz, 4H, —OCH_2); 3.93–4.04 (m, 4H, —P—OCH_2); 6.71(d, J = 736.3Hz, 1H, P—H). ^{13}C NMR: 58.69; 64.22; 71.15. ^{31}P NMR: 5.41. Mass Spec.: 227 (M^++1); 155; 109; 83; 72; 65; 59.

Di-(2-butoxy)ethyl phosphite (Ic)

Following the general procedure, 2-butoxyethanol (70.8 g, 0.6 mol) and phosphorus trichloride (27.5 g, 0.2 mol) were used; the obtained crude product was distilled at reduced pressure (158–161°C, 3 mmHg) to give a clear transparent liquid in 35% yield. IR(cm^{-1}): 2950; 2438(P—H), 1252(P=O); 1129 (C—O); 1052(P—O). ^{1}H NMR ($CDCl_3$): 0.80 (t, J = 7.2Hz, 6H, —CH_3); 1.21(q, J = 7.2Hz, 4H, —CH_2—); 1.32–1.59(m, 4H, —CH_2—); 3.55–3.89(m, 8H, —OCH_2—); 4.08–4.28 (m, 4H, —P—OCH_2); 6.87(d, J = 708.0Hz, 1H, P—H). ^{13}C NMR: 13.20; 19.16;

31.56; 61.48; 71.07; 72.00. ^{31}P NMR: 6.98. Mass Spec.: 283(M^++1); 167; 138; 111; 97; 83; 65; 57.

Di-pentyl phosphite (Id)[8]

Following the general procedure, n-pentanol (52.8 g, 0.6 mol) and phosphorus trichloride (27.5 g, 0.2 mol) were used; the obtained crude product was distilled at reduced pressure (70–71°C, 3 mmHg) to give a clear transparent liquid in 78% yield. IR(cm^{-1}): 3422; 2435(P—H), 1250(P=O); 1127 (C—O); 1056(P—O). ^{1}H NMR ($CDCl_3$): 0.79 (t, J = 6.6Hz, 6H, —CH_3); 1.20–1.31 (m, 8H, —CH_2—); 1.58(quin, J = 6.6Hz, 4H, —CH_2—); 3.94 (t, J = 6.6Hz, 4H, P—OCH_2—); 6.68 (d, J = 690Hz, 1H, —P—H). ^{13}C NMR: 13.62; 21.91; 27.37; 29.88; 65.55. ^{31}P NMR: 4.00. Mass Spec.: 223(M^++1); 153; 123; 109; 97; 83; 71; 65; 57; 43.

Phosphorylation of Amines—General Procedure

Amine (0.01 mol) and sodium hydroxide (0.02 mol) were dissolved in a mixture of 10 ml of water and 2 ml of ethanol. This solution was cooled to 0–5°C, and a solution of dialkylphosphite (0.01 mol) in 10 ml of carbon tetrachloride was added dropwise at this temperature. The mixture was then stirred at room temperature for 4 h; during this period, the reaction mixture was kept basic by adding a small portion of 5% NaOH solution. After the reaction, 10 ml each of water and chloroform (1:1) were added. The organic layer was separated and the aqueous solution was extracted three times with chloroform (10 ml portion). The combined organic layers were washed once with 10 ml of water and dried over anhydrous magnesium sulfate. The solvent was removed in a vacuum to give the crude product.

Di-(2-methoxy)ethoxyphosphorylguanidine (IIa)

Following the general procedure, guanidine hydrochloride (0.96 g, 0.01 mol) and di-(2-methoxy)ethyl phosphite (1.98 g, 0.01 mol) were used; the obtained crude product was purified by column chromatography to give a light green liquid in 20% yield. IR(cm^{-1}): 3436, 3300, 3220(—NH); 2941; 2895; 1644(C=N); 1371; 1245(P=O); 1131(C—O); 1042(P—O). ^{1}H NMR($CDCl_3$): 3.22; 3.24(s, 6H, O—CH_3); 3.43–3.47(m, 4H, O—CH_2—); 3.59 (bs, 2H, —NH_2); 3.90–4.05(m, 4H, —CH_2OP—); 6.04 (bs, 2H, —NH—). ^{13}C NMR: 58.58; 64.75; 71.12; 159.95(C=N). ^{31}P NMR: 8.68. Mass Spec. 256 (M^++1); 198; 140; 123; 59; 43.

Di-(2-ethoxy)ethoxyphosphorylguanidine (IIb)

Following the general procedure, guanidine hydrochloride (0.96 g, 0.01 mol) and di-(2-ethoxy)ethyl phosphite (2.26 g, 0.01 mol) were used;

the crude product obtained was purified by column chromatography using mixture of hexane and chloroform as elution solvent to give a light green liquid, in 17% yield. IR(cm^{-1}): 3435, 3300, 3220(—NH); 2976; 2872; 1651(C=N); 1575; 1371; 1242(P=O); 1125(C—O); 1042(P—O). ^{1}H NMR($CDCl_3$): 1.05–1.14(m,6H,—CH_3); 3.41–3.61(m, 10H, O—CH_2—, —NH_2); 3.95–4.07(m, 4H, —PO—CH_2—); 6.09(bs, 2H, —NH—). ^{13}C NMR: 15.00; 64.07; 66.71; 69.13; 159.80(C=N). ^{31}P NMR: 8.42. Mass Spec. 284 (M^++1); 212; 168; 140; 123; 59; 43.

Di-(2-butoxy)ethoxyphosphorylguanidine (IIc)

Following the general procedure, guanidine hydrochloride (0.96 g, 0.01 mol) and di-(2-butoxy)ethyl phosphite (2.80 g, 0.01 mol) were used; the crude product obtained was purified by column chromatography using mixture of hexane and chloroform as an elution solvent to give a light green liquid, in 85% yield. IR(cm^{-1}): 3445; 3354, 3213(—NH); 2959; 2872; 1651(C=N); 1578; 1465; 1363; 1242(P=O); 1128(C—O); 1045(P—O). ^{1}H NMR($CDCl_3$): 0.88–0.96(m,6H,—CH_3); 1.25–1.69(m, 8H, —CH_2—); 2.20 (bs, 2H, —NH_2); 3.40–3.65 (m, 8H, —CH_2O—); 4.00–4.27(m, 4H, —PO—CH_2—); 5.98(bs, 2H, —NH—). ^{13}C NMR: 14.00; 20.00; 32.00; 62.00; 65.00; 70.00; 160.00(C=N). ^{31}P NMR: 8.66. Mass Spec. 340(M^++1); 282; 240; 168; 140; 123; 57.

Dipentoxyphosphorylguanidine (IId)

Following the general procedure, guanidine hydrochloride (0.96 g, 0.01 mol) and dipenthl phosphite (2.24 g, 0.01 mol) were used; the crude product obtained was re-crystallized from toluene, m.p. 72–74°C, in 46% yield. IR (KBr plate): 3434, 3300, 3250(—NH); 3000; 2950; 1652(C=N), 1241(P=O); 1145(C—O); 1043(P—O). ^{1}H NMR($CDCl_3$): 0.87–0.92(m, 6H, —CH_3); 1.29–1.36(m, 8H, —CH_2—); 1.60–1.69(m, 4H, —CH_2—); 3.87–3.97(m, 4H, —CH_2O—) 6.00(bs, 4H, —NH_2, —NH—). ^{13}C NMR: 13.84; 22.14; 27.61; 29.99; 65.93; 159.55(C=N). ^{31}P NMR: 6.80. Mass Spec. 280 (M^++1); 210; 168; 140; 123; 43.

N,N′-Bis-(di-2-methoxyethoxy)phosphoryl ethylenediamine (IIIa)

Following the general procedure, ethylenediamine (0.6 g, 0,01 mol) and di-(2-methoxy)ethyl phosphite (4.00 g, 0.02 mol) were used; the crude product obtained was purified by column chromatography using a mixture of hexane and chloroform as the elution solvent to give a light green liquid, in 70% yield. IR(cm^{-1}): 3401(—NH); 3300; 2943; 2890; 1455; 1237(P=O); 1131(C—O); 1040(P—O). ^{1}H NMR ($CDCl_3$): 2.90(bs, 4H, —NCH_2); 3.25(s, 12H, —OCH_3); 3.46–3.48(m, 8H, —OCH_2—); 3.92–4.04(m, 8H, —CH_2O—P). ^{13}C NMR: 42.45; 58.79; 65.22; 71.45. ^{31}P NMR:

11.40. Mass Spec.: 453 (M^++1); 226; 220; 214; 197; 170; 152; 139; 110; 59; 45.

N,N′-Bis-(di-2-ethoxyethoxy)phosphoryl ethylenediamine (IIIb)

Following the general procedure, ethylenediamine (0.6 g, 0,01 mol) and di-(2-ethoxy)ethyl phosphite (4.55 g, 0.02 mol) were used; the crude product obtained was purified by column chromatography using a mixture of hexane and chloroform as the elution solvent to give a light green liquid, in 39% yield. IR(cm^{-1}): 3370(—NH); 3300; 2975; 2872; 1653; 1234(P=O); 1123(C—O); 1038(P—O). ^{1}H NMR ($CDCl_3$): 1.21(t, J = 7.20Hz, 12H); 2.15(bs, 2H, —NH—); 3.03–3.09 (bs, 4H, —NCH_2—); 3.54(q, J = 7.20Hz, 8H, —OCH_2—); 3.62(t, J=7.20Hz, 8H, —OCH_2—); 4.05–4.18(m, 8H, —CH_2O—P). ^{13}C NMR: 14.99; 42.37; 62.37; 65.39; 69.38. ^{31}P NMR: 11.65. Mass Spec.: 509 (M^++1); 254; 225; 221; 220; 197; 184; 153; 125; 110; 73; 45.

N, N′-Bis-(di-2-butoxyethoxyl)phosphoryl ethylenediamine (IIIc)

Following the general procedure, ethylenediamine (0.6 g, 0,01 mol) and di-(2-butoxy)ethyl phosphite (5.60 g, 0.02 mol) were used; the crude product obtained was purified by column chromatography using a the mixture of hexane and chloroform as elution solvent to give a light green liquid, in 50% yield. IR(cm^{-1}): 3276(—NH); 2959; 2890; 1653; 1459; 1234(P=O); 1130(C—O); 1051(P—O). ^{1}H NMR ($CDCl_3$): 0.91 (t, J = 7.2Hz, 12H, —CH_3); 1.25–1.58 (m, 16H, —CH_2—); 3.02–3.08(bs, 4H, —NCH_2—); 3.47–3.60(bs, 6.9Hz, 16H, —OCH_2—); 4.09–4.16(m, 8H, —$POCH_2$—). ^{13}C NMR: 13.86; 19.18; 31.61; 41.53; 42.38; 61.57; 65.41; 69.51; 69.57; 71.09; 71.86. ^{31}P NMR: 11.27. Mass Spec. 311 (M^+/2 + 1); 310; 227; 220; 210; 153; 125; 110; 99; 85; 57; 45.

N,N′-Bis-(dipentoxy)phosphoryl etilenediamine (IIId)

Following the general procedure, ethylenediamine (0.6 g, 0.01 mol) and dipentyl phosphite (4.50 g, 0.02 mol) were used; the crude product obtained was purified by column chromatography using a mixture of hexane and chloroform as the elution solvent to give a red liquid, in 30% yield. IR(cm^{-1}): 3244(—NH); 2957; 2869; 1463; 1231(P=O); 1118(C—O); 1048(P—O). ^{1}H NMR ($CDCl_3$): 0.85–0.94(m, 12H,—CH_3); 1.31–1.40(m, 8H, —CH_2—); 1.60–1.68(m, 16H, —CH_2—); 2.99–3.02(s, 4H, N—CH_2—); 3.90–4.03(m, 8H, —CH_2—O). ^{13}C NMR: 13.92; 22.23; 27.70; 30.00; 42.71; 64.82. ^{31}P NMR: 10.75. Mass Spec. 501(M^++1); 500 (M^+); 431; 361; 251; 250; 221; 194; 111; 110.

N,N′-Di-(2-ethoxyethoxy)phosphoryl- N,N′-dimethyl ethylenediamine (IV)

Following the general procedure, N,N'-dimethyl ethylenediamine (0.88 g, 0.01 mol) and di-(2-ethoxy)ethyl phosphite (4.56 g, 0.02 mol) were used; the obtained crude product was purified by column chromatography using a mixture of hexane and chloroform as the elution solvent to give a liquid, in 39% yield. IR(cm^{-1}): 3411; 2975; 2870; 1654; 1457; 1235(P═O); 1126(C─O); 1041(P─O). ^{1}H NMR ($CDCl_3$): 1,21(t, J = 6.90Hz, 6H, $-CH_3$); 2.21 (s, 6H, $-NCH_3$); 2.38(t, J = 6.30Hz, 2H, $-NCH_2-$); 2.98–3.05(m, 2H, $-PNCH_2-$); 3.54(q, J = 6.90Hz, 4H, $-OCH_2-$); 3.62–3.66(m, 4H, $-OCH_2-$); 4.10–4.20(m, 4H, $-POCH_2-$). ^{13}C NMR: 14.97; 38.30; 44.87; 59.46; 66.43; 65.32; 69.33. ^{31}P NMR: 11.20. Mass Spec. 313(M^++1); 312 (M^+); 240; 168; 110; 58.

Complexation Studies by UV Spectroscopy

Inorganic salts were used as purchased and kept under nitrogen in a desiccator. The ligand solutions in anhydrous methanol were prepared by diluting a 0.1 M stock solution to the expected suitable concentration. For the differential spectra, an inorganic salt solution was placed in the reference cell. Metal ion solutions were prepared in 0.01 M, 0.1 M, and 1.0 M concentrations; these solutions were added to the ligand solution through a 10 μl GC syringe, so that the changes in volume could be neglected. After each addition, the cell was thoroughly shaken before the UV spectrum was recorded. For the qualitative studies, a 10-fold excess of the salts was used.

REFERENCES

[1] (a) F. A. Cotton and B. H. C. Winguist, *J. Inorg. Chem.*, **9,** 688 (l970); (b) F. A. Cotton and P. A. Schunn, *J. Amer. Chem. Soc.*, **85,** 2394 (l963).
[2] T. H. Siddallii, *J. Inorg. Dhem.*, **25,** 883 (l963).
[3] G. Petrov, I. Velinov, and M. Kirilov, *Monatsh. Chem.*, **104,** 1301 (l973).
[4] S. K. Si, Y. F. Zhao, W: O. Lin, and M. C. Souza, *Syn. Comm.*, **20,** 3295 (1990).
[5] W. O. Lin, C. N. Guimaraes, M. C. de Souza, J. B. N. da Costa, and H. G. Alt, *Phosphorus, Sulfur, and Silicon*, **92,** 1 (1994).
[6] W. O. Lin, M. C. de Souza, L. de Amorim, and H. G. Alt, *Phosphorus, Sulfur, and Silicon*, **163,** 68 (2000).
[7] W. O. Lin, M. C. de Souza, and J. B. N. da Costa, *Química Nova*, **18,** 431 (1995).
[8] M. G. Imaev, V. M. Gorina, and V. G. Maslennikov, *, Zhurnal Obshchei Khimii*, **35**(2), 372, (1965).