

## SPECTROSCOPIC STUDIES ON SOME METHYLPHOSPHONOTHIOIC DIAMIDES AND RELATED COMPOUNDS

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### ABSTRACT

A series of methylphosphonothioic diamides  $\text{MeP(S)(NHR)}_2$  ( $\text{R} = \text{Me}$ ,  $\text{pr}^i$  and  $\text{Bu}^i$ ) and some related compounds viz,  $\text{PhP(S)(NHBu}^i)_2$ ,  $\text{Ph}_2\text{P(S)(NHCH}_2\text{Ph)}$  are prepared and their infrared, n.m.r. and mass spectra are recorded. The  $\nu(\text{P}=\text{S})$ ,  $\nu(\text{P}-\text{C})$  and  $\nu(\text{P}-\text{N})$  values are determined in each case and indicate very little effect of the substituents on these bond vibrations. On the bases of  $^1\text{H}$ ,  $^{13}\text{C}$  and  $^{31}\text{P}$  n.m.r. spectroscopic data it was shown that the spectra of all diamides agree with the expected structures with the exception of  $\text{Pr}^i$  and  $\text{Bu}^i$  diamides where the  $^1\text{H}$  and  $^{13}\text{C}$  n.m.r. of the methyl groups of the  $\text{Pr}^i$  or  $\text{Bu}^i$  groups showed nonequivalence.

### INTRODUCTION

The thermal bulk condensation reactions of a series of p-phenyl-phosphonothioic diamides have been extensively studied by Shaw and Ibrahime (Shaw and Ibrahime, 1969, 1971, 1981). Some novel inorganic ring systems were obtained, of particular interest, is the cyclodiphosphazane ring system having the general formula  $(\text{PhP(S)NR})_2$ . In the present work a series of analogous P - Methylphosphonothioic diamides were prepared for the purpose of obtaining some cyclodiphosph (V) azane derivatives of expected antitumoral activity (Labarre, 1985). Their analytical data as well as spectroscopic data are reported.

### EXPERIMENTAL

#### Spectroscopic measurements

The infrared spectra of all compounds were obtained from potassium bromide discs using a Pye Unicam Sp 2000 spectrometer.  $^1\text{H}$  and  $^{13}\text{C}$  n.m.r.

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spectra were recorded on Joel, FX-200 n.m.r. spectrometer using TMS as an internal reference for  $^1\text{H}$  n.m.r. and  $^{13}\text{CS}_2$  for  $^{13}\text{C}$  n.m.r. while  $^{31}\text{P}$  n.m.r. spectra were recorded using a F.T. spectrometer operating at 24.15 MHz and using 0.05M solutions in chloroform with a deuterated solvent as an external lock (8 mm tube in 10 mm). The shifts are measured relative to 85%  $\text{H}_3\text{PO}_4$  (0.0 ppm) and reported on the same scale in ppm.

High resolution mass spectra were obtained on an A.E.I. MS-902 instrument operating at 70 eV ionizing voltage, by the direct inlet system. Exact masses were determined by peak matching against known masses of perfluorokerosene reference.

#### Aminolysis of methylphosphonothioic dichloride

To a solution of 1.4 g (0.01 mole) of Methylphosphonothioic dichloride  $\text{MeP(S)Cl}_2$  in 50 ml. acetonitrile was added a solution of alkylamine  $\text{RNH}_2$  ( $\text{R} = \text{Me}, \text{Pr}^i$  and  $\text{Bu}^i$ ) (0.04 mole) in acetonitrile dropwise with constant stirring at room temperature. After filtration of the amine hydrochloride, the filtrate was evaporated under vacuo. The remaining residue was triturated with petroleum ether (40-60 °C) and filtered. From the filtrate the corresponding Methylphosphonothioic di(monoalkylamide)  $\text{MeP(S)(NHR)}_2$  was obtained. The analytical data and spectral properties of these compounds are as follows:

#### Methylphosphonothioic di (monomethylamide) compound

$\text{MeP(S)(NHMe)}_2$  has the following properties: M.P. 52°C,  $^1\text{H}$  n.m.r.:  $\delta$  1.77 (d, 3H), 2.27 (s, 2H), 2.60 (d, 6H).  $^{13}\text{C}$  n.m.r.:  $\delta$  20.03 (d), 26.76 (d),  $^{31}\text{P}$  n.m.r.: 869.3482 (s) MS : m/e = 138 (100%)  $\text{M}^+$ , 123 (7%)  $\text{M}^+ - 15$ , 109 (60%)  $\text{M}^+ - 29$ , 94 (18%)  $\text{M}^+ - 44$ , 76 (87%), 60 (31%). Exact mass for  $\text{C}_3\text{H}_{12}\text{N}_2\text{SP}$  138.0382, calculated 138.0381, deviation  $\Delta = +0.1$  ppm.

#### Methylphosphonothioic di (monoisopropylamide) compound

$\text{MeP(S)(NHPr}^i)_2$  has the following properties: M.P. 75 °C,  $^1\text{H}$  n.m.r.:  $\delta$  1.17, 1.19 (dd, 12H), 1.77 (d, 3H), 2.72 (s, 2H), 3.36 - 3.51 (m, 2H).  $^{13}\text{C}$  n.m.r.:  $\delta$  23.34 (d), 25.69 (t), 43.50 (d).  $^{31}\text{P}$  n.m.r.:  $\delta$  : 60.5300 (s). MS : m/e = 194 (5%)  $\text{M}^+$ , 136 (8%)  $\text{M}^+ - 58$ , 122 (5%)  $\text{M}^+ - 72$ , 104 (32%)  $\text{M}^+ - 90$ , 94 (17%)  $\text{M}^+ - 100$ , 58 (100%). Exact mass for  $\text{C}_7\text{H}_{19}\text{N}_2\text{SP}$  194.1006, calculated 194.1007, deviation  $\Delta = - 0.5$  ppm.

**Methylphosphonothioic di(monoisobutylamide) compound**

MeP(S)(NHBu<sup>i</sup>)<sub>2</sub> has the following properties : M.P. 71 °C, <sup>1</sup>H n.m.r.: δ 0.92 (d, 12H), 1.70 (septet, 4H), 1.78 (d, 3H), 2.27 (s (broad), 2H), 2.65 – 2.76 (m, 2H). <sup>13</sup>C n.m.r.: δ 20.16 (s), 21.45 (d), 29.74 (d), 48.75 (d). <sup>31</sup>P n.m.r.: δ 66.2300 (s). MS : m/e = 222 (14%) M<sup>+</sup>, 150 (22%) M<sup>+</sup> – 72, 118 (18%) M<sup>+</sup> – 104, 94 (20%) M<sup>+</sup> – 128, 72 (100%). Exact mass for C<sub>9</sub>H<sub>23</sub>N<sub>2</sub>SP 222.1324, calculated 222.1320, deviation Δ = +1.8 ppm.

**Synthesis of P, P-diphenyl – N-benzylphosphinothioic amide Ph<sub>2</sub>P(S)NHCH<sub>2</sub>Ph**

**(i) Preparation of P, P-diphenylphosphinothioic chloride**

To 22.05 g.(0.1 mole) of diphenylphosphinous chloride, was added 3.2 g of powdered sulfur. The mixture was heated under nitrogen atmosphere for 10 minutes. After completion of the reaction the product was cooled and then dissolved in 100 ml. benzene. The resulting solution was filtered under nitrogen to remove the excess sulfur.

**(ii) Preparation of P, P-diphenyl – N-benzylphosphinothioic amide**

A solution of diphenylphosphinothioic chloride in benzene as obtained from (i), was added dropwise to a solution of 42.8 g.(0.4 mole) of benzylamine in 200 ml. benzene at 0 °C, with continuous stirring. The reaction was continued for 2 hours. After the completion of the reaction, benzylamine hydrochloride was filtered off. The filtrate was kept overnight for crystallization, whereby white crystalline solid (m.p. 118 °C) was obtained. After recrystallization twice from ethanol, colourless crystalline solid (m.p. 120 °C) was obtained. The analytical data and spectroscopic properties of Ph<sub>2</sub>P(S)NHCH<sub>2</sub>Ph are as follows: M.P. 120 °C, <sup>1</sup>H n.m.r.: δ = 2.47 ppm. (s (broad), 1H), δ = 4.06 ppm. (d, 2H), δ = 7.20–7.50 ppm. (m, 10H), and at δ = 7.97–8.07 ppm (m, 5H). <sup>13</sup>C n.m.r.: δ = 45.31 ppm. (s) and at δ = 131.76 (s), 131.72(s), 131.58(s), 128.67(s), 128.62(s), 128.41(s), 128.05(s), 127.54 (s), and 127.52 ppm. <sup>31</sup>P n.m.r.: δ = 60.5737 ppm. Ms: m/e = 323 (26%) M<sup>+</sup>; 290 (7%) M<sup>+</sup> – 33, 218 (27%) M<sup>+</sup> – 105, 185 (30%) M<sup>+</sup> – 138, 183 (33%) M<sup>+</sup> – 140, 140 (17%), 139 (12%), 106 (100%), 91 (13%). Exact mass for C<sub>19</sub>H<sub>18</sub>NSP is 323.0902, calculated 323.0898, deviation Δ = +1.2 ppm.

**Synthesis of P-phenylphosphonothioic di (monoisobutylamide)**

**PhP (S) (NHBu<sup>i</sup>)<sub>2</sub>.**

To a solution of isobutylamine 2.56 g (0.04 mole) in 100 ml. of benzene was added a solution of P-phenylphosphonothioic dichloride 2.11 g (0.01 mole) in

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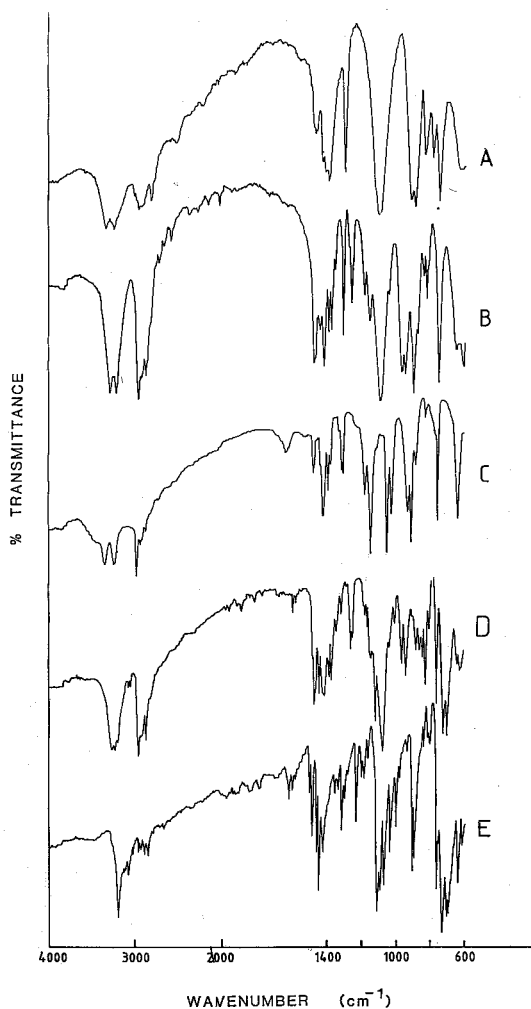
benzene dropwise with continuous stirring at room temperature. After filtration of the amine hydrochloride, the filtrate was evaporated under vacuo. The remaining solid was recrystallized from petroleum ether (40–60) and filtered. The analytical data is in good agreement with the formula  $C_{14}H_{27}N_2SP$ . Its spectral properties are as follows: M.P. 81 °C,  $^1H$  n.m.r.:  $\delta$  0.89 (2d, 12H), 1.69 (septet, 2H), 2.37 (s (broad), 2H), 2.65–2.84 (m, 4H), aromatic protons (o- and p-) at 7.41–7.51 (m, 3H) and (m-) at 7.94–8.04 (m, 2H).  $^{13}C$  n.m.r. (ppm)  $\delta$  = 20.13 (s), 20.18 (s), 29.70 (s), 29.82 (s), 48.96 (s), 49.00 (s), 128.24–136.30 (m).  $^{31}P$  n.m.r.;  $\delta$  = 65.6011 ppm. MS : m/e = 284 (14%)  $M^+$ ; 212 (17%)  $M^+ - 72$ , 180 (26%)  $M^+ - 104$ , 156 (12%)  $M^+ - 128$ , 72 (100%). Exact mass for  $C_{14}H_{27}N_2SP$  284.1480, calculated 284.1476, deviation  $\Delta$  = +1.4 ppm.

## RESULTS AND DISCUSSION

### Infrared spectra

The infrared spectra of  $MeP(S)(NHMe)_2$ ,  $MeP(S)(NHPr^i)_2$ ,  $MeP(S)(NHBu^i)_2$ ,  $PhP(S)(NHBu^i)_2$  and  $(Ph)_2P(S)(NHCH_2Ph)$  are shown in Fig. 1. Their  $\nu(P=S)$  and  $\nu(N-H)$  values are given in Table 1. The infrared spectrum in each case shows the characteristic bands for all substituents and these are very much the same as in normal compounds (Bellamy, 1975). The  $\nu(N-H)$  values occur in the range 3400–3180  $cm^{-1}$ . The relatively low values of such vibrations compared with that of the free N–H, may indicate the existence of H–bonding which involves the sulfur atom in each case. The  $\nu(P=S)$  shows a strong feature in the range 750 – 770  $cm^{-1}$ . Interestingly, in the case of phenylphosphonothioic and diphenylphosphinothioic compounds the value occurs around 765  $cm^{-1}$ , whilst, in the case of methylphosphonothioic compounds the value occurs around 775  $cm^{-1}$ . This may indicate the relative strength of the P = S bond, in the former case due to the expected electron delocalization between the P = S and phenyl group which may increase the  $\pi$  – electron density of the P = S bond and hence increase its vibrational frequency. Such delocalization is forbidden in the case of methylphosphonothioic compounds. The assignments of the strong band in the 750 – 770  $cm^{-1}$  region to  $\nu(P=S)$  are in agreement with the work of Thomas and Bellamy (Thomas and Chittenden, 1964; Bellamy, 1975) on related compounds. Table 2 lists both the  $\nu(P=N)$  and  $\nu(P=C)$  values. The  $\nu(P=N)$  values occur in the region 890–950  $cm^{-1}$ . Thomas (Thomas, 1966) has reviewed the P = N stretching frequency in many compounds and concluded the existence of such mode in the region between 870 and 1055  $cm^{-1}$ . On the other hand, the  $\nu(P=C)$  in our class of compounds are assigned in the range of

1440–1470  $\text{cm}^{-1}$  (see Table 2). The P–phenyl link all occur around 1445  $\text{cm}^{-1}$  and this is in agreement with the work of Daasch and Smith (Smith, 1951) and by Thomas and Chittenden (Chittenden, 1965). They concluded that  $\nu(\text{P} = \text{C})$ , P–Ph link occurs at 1425–1450  $\text{cm}^{-1}$ .



**Fig. 1 : Infrared Spectra of some Methyl-, Phenylphosphonothioic diamides and Diphenylphosphinothioic amide; A:  $\text{MeP}(\text{S})(\text{NHMe})_2$ , B:  $\text{MeP}(\text{S})(\text{NHPr}^i)_2$ , C:  $\text{MeP}(\text{S})(\text{NHBu}^i)_2$ , D:  $\text{PhP}(\text{S})(\text{NHBu}^i)_2$  and E:  $\text{Ph}_2\text{P}(\text{S})(\text{NHCH}_2\text{Ph})$ .**

### Mass Spectra

All the mass spectra of the above mentioned compounds are found to show the exact molecular masses. Thus the mass spectra of the compounds  $\text{MeP(S)(NHR)}_2$  ( $R = \text{Me, Pr}^i \text{ or Bu}^i$ ) show the molecular ion ( $M^+$ ) in the intensity range 5 - 100%. The molecular ion in  $\text{MeP(S)(NHMe)}_2$  is the base peak, whilst in the  $\text{MeP(S)(NHPr}^i)_2$  spectrum, the base peak belongs to the  $(\text{NHPr}^i)^+$  ion. The same pattern is shown in the  $\text{MeP(S)(NHBu}^i)_2$  spectrum where the  $(\text{NHBu}^i)^+$  ion is the base peak. These observations might be attributed to the steric effect of the bulky alkyl groups in these compounds. Similar fragmentation of  $\text{PhP(S)(NHBu}^i)_2$  compound gave the  $(\text{NHBu}^i)^+$  ion which is found to be the base peak while the molecular ion ( $M^+$ ) has an intensity of 14%. The fragmentation of  $\text{Ph}_2\text{P(S)(NHCH}_2\text{Ph)}$  compound also show the fragment with  $m/e = 106$  which corresponds to  $(\text{NHCH}_2\text{Ph})^+$  ion as a base peak, whereas the parent ion ( $M^+$ ) has an intensity of 26%. The mode of fragmentation of the above compounds indicate that the loss of  $(\text{NHR})^+$  moiety is the main fragmentation pattern. Further fragmentation is shown in the experimental section for each compound separately.

### N.M.R. Spectra

$^1\text{H}$ ,  $^{13}\text{C}$  and  $^{31}\text{P}$  n.m.r. spectra of all methylphosphonothioic di (monoalkylamides) are in accordance with the expected structure (Ibrahime *et.al.*, 1986).  $^1\text{H}$  n.m.r. of  $\text{MeP(S)(NHMe)}_2$  showed a doublet at  $\delta = 1.77$  ppm., a broad singlet at  $\delta = 2.27$  ppm. and another doublet at  $\delta = 2.60$  ppm. in the intensity ratio 3:2:6 corresponding to  $\text{Me-P}$  ( $J_{\text{P-H}} = 18.5$  Hz),  $\text{N-H}$  and  $\text{Me-N-P}$  ( $J_{\text{P-H}} = 13.85$  Hz), respectively.  $^{13}\text{C}$  n.m.r. of the same compound showed two types of doublets at  $\delta = 20$  ppm. and  $\delta = 27$  ppm. in the intensity ratio 1:2 corresponding to  $^{13}\text{C-P}$  ( $J^{13}\text{C-P} = 90.12$  Hz) and  $^{13}\text{C-N-P}$  ( $J^{13}\text{C-P} = 3.0$  Hz) respectively. It should be noted that  $^1\text{H}$  n.m.r. spectrum of  $\text{MeP(S)(NHPr}^i)_2$  showed two doublets at  $\delta = 1.12$  ppm. and  $\delta = 1.20$  ppm. indicating that the two methyl protons of the isopropyl group are non-equivalent due to their intrinsic asymmetry. This non-equivalence is also observed in the  $^{13}\text{C}$  n.m.r. spectrum of the same compound. Similar features have also been detected both in the  $^1\text{H}$  and  $^{13}\text{C}$  n.m.r. spectra of  $\text{MeP(S)(NHBu}^i)_2$  where both the methyl's protons and carbons of the two methyl groups of each isobutyl show chemical shift non-equivalence because they are diastereotopic. Also, the correlation between the  $^{13}\text{C}$  chemical shift of the methyl group attached directly to phosphorus and the  $^{31}\text{P}$  chemical shift

showed linear relationship, (Ibrahime *et. al.*, 1986) indicating that the substituents has minor effect on the values of chemical shift. It is not out of place to mention that the  $^1\text{H}$  n.m.r. spectrum of  $\text{PhP(S)(NHBu}^i)_2$  showed the following signals at  $\delta = 0.8847$  (2d, 12H) of the methyl groups,  $\delta = 1.6970$  (septet, 2H) of the methine protons,  $\delta = 2.3760$  (s, 2H) of the N-H protons,  $\delta = 2.7385$  (m, 4H) of the methylene protons,  $\delta = 7.4845$  (m, 3H) of the o- and p- aromatic protons and  $\delta = 7.9543$  ppm. (m, 2H) of the m- aromatic protons. Also its  $^{31}\text{P}$  n.m.r. showed a signal at  $\delta = 65.6011$  ppm. The decoupled  $^{13}\text{C}$  n.m.r. spectrum of the compound showed the following signals at  $\delta = 20.13, 20.16$  ppm. (2s) showing the non-equivalence of the methyl groups,  $\delta = 29.70, 29.82$  ppm. (2s) of the methylene groups,  $\delta = 48.96, 49.00$  ppm. (2s) of the methine carbons together with six signals corresponding to the aromatic carbons. Again the coupled spectrum showed the following signals at  $\delta = 20.153$  (q,  $J^{13}\text{C-H} = 0.453$  ppm.),  $\delta = 29.513$  (d,  $J^{13}\text{C-H} = 0.126$  ppm.) and at  $\delta = 30.000$  ppm. (d,  $J^{13}\text{C-H} = 0.125$  ppm.) indicating that there are two types of methine carbon environments due to the intrinsic assymetry encountered in such compounds, whilst, the methylene carbon appeared as a triplet at  $\delta = 48.980$  ppm. (t,  $J^{13}\text{C-H} = 0.626$  ppm.) and the aromatic carbons appeared as six doublets. at  $\delta = 127.65 - 136.31$  ppm.

The  $^1\text{H}$  n.m.r. spectrum of  $\text{Ph}_2\text{P(S)NHCH}_2\text{Ph}$  showed the following signals at  $\delta = 2.47$  ppm. (s broad) of the N.H. protons, at  $\delta = 4.06$  ppm. (d, 2H) corresponding to the  $\text{CH}_2$  protons, at  $\delta = 7.20 - 7.50$  ppm. (m, 10H) of the two Ph-P aromatic protons and at  $\delta = 7.87 - 8.07$  ppm. (m, 5H) of the benzyl aromatic protons. The decoupled  $^{13}\text{C}$  n.m.r. spectrum showed signals at 45.31 ppm. (s) corresponding to the methylene carbon and 12 singlets in the range  $\delta = 127.52 - 139.41$  ppm. that corresponds to the aromatic carbons. The coupled spectrum showed the following signals at  $\delta = 44.55 - 46.05$  ppm. (t) for the methylene carbon and other signals at  $\delta = 126.94 - 129.25$  (m), 130.98 - 132.35 (m), 133.21 - 134.89 (m), and 139.24 - 139.41 (m) that correspond to the aromatic carbons.

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Table 1  
N-H and P=S Stretching frequencies \* of some Methyl-,  
Phenylphosphonothioic diamides and Diphenyl-  
phosphinothioic amide.

Compound	$\nu(\text{N-H})$ ( $\text{cm}^{-1}$ )	$\nu(\text{P=S})^a$ ( $\text{cm}^{-1}$ )
MeP(S)(NHMe) <sub>2</sub>	3400 m, 3280 m	752 sm
MeP(S)(NHPr <sup>i</sup> ) <sub>2</sub>	3320 s, 3255 s	755 s
MeP(S)(NHBu <sup>i</sup> ) <sub>2</sub>	3390 wm, 3270 wm	758 s
PhP(S)(NHBu <sup>i</sup> ) <sub>2</sub>	3300 m, 3240 m, 3200 sh	765 s
Ph <sub>2</sub> P(S)(NHCH <sub>2</sub> Ph)	3180 m, 3230 sh	768 s

\* : s, strong; m, medium; sh, shoulder; w, weak

a: The 765 and 768  $\text{cm}^{-1}$  bands may also be attributed to vibrations of monosubstituted benzene derivatives.

Table 2  
P-C and P-N stretching frequencies\* of  
some Methyl, - Phenylphosphonothioic diamides  
and Diphenylphosphinothioic amide.

Compound	$\nu(\text{P-C})$ ( $\text{cm}^{-1}$ )	$\nu(\text{P-N})$ ( $\text{cm}^{-1}$ )
MeP(S)(NHMe) <sub>2</sub>	1452 m	920 s, 892 s
MeP(S)(NHPr <sup>i</sup> ) <sub>2</sub>	1470 m	930 sm, 908 s
MeP(S)(NHBu <sup>i</sup> ) <sub>2</sub>	1470 s	953 sm, 903 s
PhP(S)(NHBu <sup>i</sup> ) <sub>2</sub>	1445 ms	945 m, 890 mw
Ph <sub>2</sub> P(S)(NHCH <sub>2</sub> Ph)	1446 s	940 w, 905 m

\* : s, strong; m, medium; w, weak.



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## دراسات طيفية عن بعض ثنائي الأمينات لمثيل الفسفونوثيويك وبعض المركبات المتعلقة بها

عز الدين حرب إبراهيم - إبراهيم صالح النعيمي و المتولي السيد نور

يعني هذا البحث بدراسات طيفية لبعض المركبات ثنائي الأميدات لمثيل الفسفونوثيويك وكذلك بعض المركبات المتعلقة بها وقد اشتمل البحث على تحضير هذه المركبات وكذلك إجراء القياسات الطيفية لها ومنها تحليل أطيف الأشعة دون الحمراء والرنين النووي المغناطيسي لكل من الأنوية  $^1\text{H}$ ,  $^{13}\text{C}$ ,  $^{31}\text{P}$  وكذلك تحليل أطيف الكتلة.

وقد أوضحت الدراسات تحديد الأشكال الفراغية لجميع المركبات وكذلك طبيعة الروابط بها كما أوضحت دراسة الرنين النووي لأنويه  $^1\text{H}$  وكذلك  $^{13}\text{C}$  لبعض المركبات على وجود بعض الظواهر الفراغية الشاذة. وجدير بالذكر ان هذه المركبات تعتبر المواد الأساسية لتحضير بعض المشتقات والتي يتوقع أن يكون لها نشاط بيولوجي فعال مضاد لمرض السرطان.