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## Preparation and confirmation of some novel amide and amide siloxane derivatives of ascorbic acid part 1

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### ABSTRACT

The preparation and confirmation of some novel amide and amide siloxane derivatives of ascorbic acid as following, in three necked flask with mechanical stirrer in free solvent the 1M of ascorbic acid is reacted with 1M of diethylene-triamine at 170-190°C for 3hrs., the product is (E)-N-(2-(2-aminoethylamino)-ethyl)-2,3,4,5,6-penta-hydroxyhex-2-enamide(I). After that, the 1M of compound I is mixed with 1M of ethanolamine and added 1M of dimethyl-dichlorosilane dropwise in presence of pyridine as acid acceptor at room temperature and raising it to 170°C for 3hrs., the product is (E)-2-((2-aminoethoxy)dimethylsilyloxy)-N-(2-(2-aminoethylamino)ethyl)-3,4,5,6-tetra-hydroxyhex-2-enamide (II). Also, the 1M of compound I is mixed with 1M of *p*-aminophenol and added 1M of dimethyl-dichlorosilane dropwise in presence of pyridine as acid acceptor at room temperature and raising it to 170°C for 3hrs., the product is (E)-N-(2-(2-aminoethylamino)ethyl)-2-((4-amino-phenoxy)dimethyl-silyloxy)-3,4,5,6-tetra-hydroxyhex-2-enamide(III). Finally, the 1M of compound I is mixed with 2M of *p*-aminophenol and added 2M of dimethyl-dichlorosilane dropwise in presence of pyridine as acid acceptor at room temperature and raising it to 170°C for 3hrs., the product is (E)-N-(2-(2-aminoethylamino)ethyl)-2,3-bis((4-aminophenoxy)-dimethylsilyloxy)-4,5,6-trihydroxyhex-2-enamide (IV). The products I, II, III and IV are purified and confirmed by FT.IR, <sup>1</sup>HNMR and elemental analysis.

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### KEYWORDS

Amide;  
Amide-siloxane compounds;  
FTIR;  
<sup>1</sup>HNMR;  
Elementary analysis.

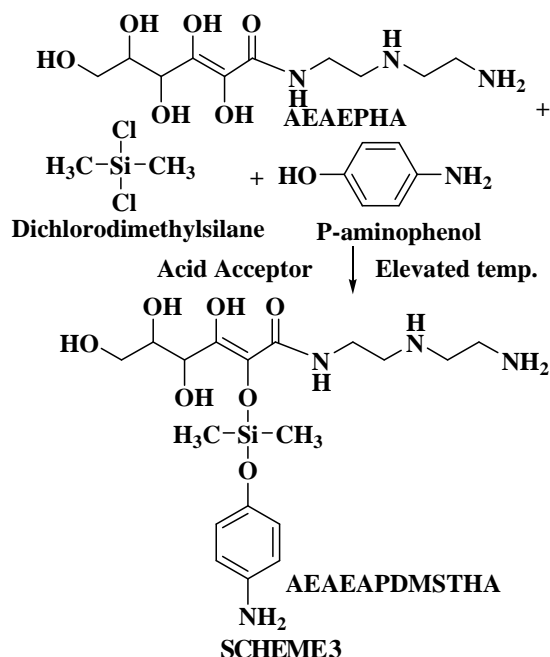
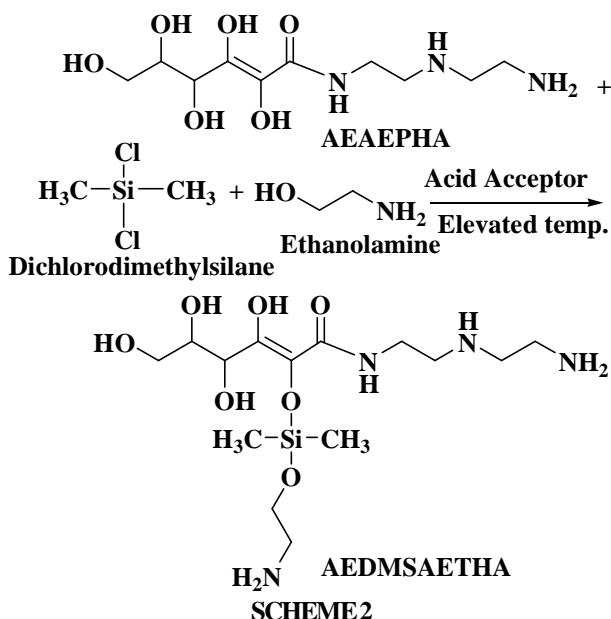
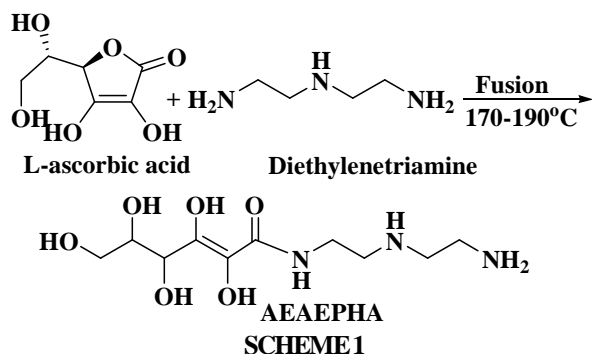
### INTRODUCTION

There are research works related to the synthetic pathway of imidazoline<sup>[4]</sup>. One of the methods used for their preparation consists in a prolonged heating of carboxylic acids with 1,2-diamines under acid catalysis<sup>[4]</sup>. Another procedure widely used in industry is the thermal reaction between 1,2-diamines and with carboxylic acids in the presence and absence of solvents. However, this process often results in imidazolines of low yields and purity<sup>[4]</sup>. Thiosemicarbazones and their de-

derivatives have continued to be the subject of extensive investigation in chemistry and biology owing to their broad spectrum of antitumour, antibacterial, antiviral, antifungal, antimalarial and antineoplastic activities, and recently reported corrosion inhibiting properties<sup>[2,4]</sup>. Thiosemicarbazone, benzyl thiosemicarbazone, and some amides like urea, thiourea, acetamide, thioacetamide, semicarbazide and thiosemicarbazide, for comparison<sup>[1,3,4,5,6,8]</sup>.

Organic in general require two functionalities: one nucleophilic and the other hydrophobic<sup>[10]</sup>. The nucleo-

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philic functionality gives rise to chemisorption by means of coordination with the acidic surface of iron atoms, while the hydrophobic functionality shields the metal surface from the aqueous corrosion media.

In this study, the some novel amide and amide siloxane derivatives of ascorbic acid are prepared and confirmed by TT.IR, <sup>1</sup>HNMR and elemental analysis. The changing of functional and structural groups/correlations on the characteristics of the molecular structure of the studied compounds are discussed.

## EXPERIMENTAL

## Preparation of amide compound (1)

The preparation of amide compound (1) was carried out in three necked flask with mechanical sterrier and free solvent, 1M of L-ascorbic acid with 1M of diethylenetriamine by fusion where temperature is raising stepwise until to 170-190°C (SCHEME 1). The product is purified and confirmed by FT.IR, <sup>1</sup>HNMR and elemental analysis.

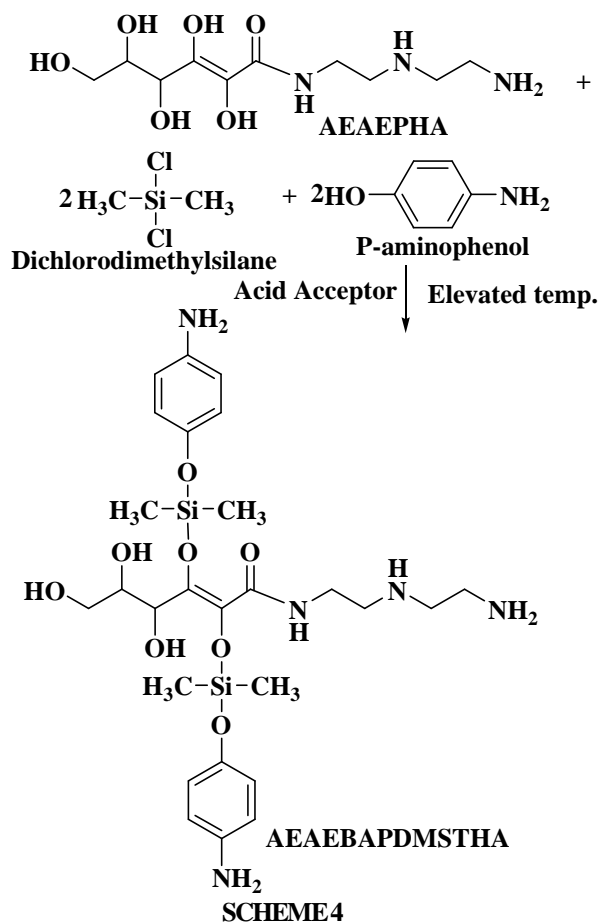
## Preparation of novel amide siloxane compounds (2-4)

The preparation of amide siloxane compounds (2-4) were carried out in three necked flask with mechanical sterrier and free solvent, 1M of compound (1) was mixed with 1M of ethanolamine and *p*-aminophenol separately, respectively, were added to each mixture dropwise under inert gas 1M of dimethyl-dichlorosilane at room temperature and raising stepwise until to 170-190°C (SCHEME 2 and 3). The product is purified and confirmed by FT.IR, <sup>1</sup>HNMR and elemental analysis. Finally, The preparation of amide siloxane compound IV was carried out in three necked flask with mechanical sterrier and free solvent, 1M of compound (1) was mixed with 2M of *p*-aminophenol added to this mixture dropwise under inert gas 2M of dimethyl-dichlorosilane at room temperature and raising stepwise until to 170-190°C (SCHEME 4). The product is purified and confirmed by FT.IR, <sup>1</sup>HNMR and elemental analysis.

## Characteristic properties of the prepared compds.

## Infra-red analysis (FTIR)

The IR spectra of the prepared compounds were confirmed by using FTIR spectrometer model type mattson infinity series bench top 961. The wave num-



ber and intensities of the IR bands of the different types of the function groups were determined in a range of 4000- 400 $\text{cm}^{-1}$ .

### Nuclear magnetic resonance $^1\text{H}$ NMR

The  $^1\text{H}$ NMR of the prepared compounds were confirmed by 300 MHz spectrometer W-P-300, Bruker. The solution for  $^1\text{H}$ NMR analysis was prepared by dissolving the prepared compounds in dimethyl sulphoxide (DMSO).

## RESULTS AND DISCUSSION

### Confirmation of the prepared compound:

The chemical structure of the prepared compounds (SCHEMES 1-4) was characterized via:

### FTIR spectrum

Figure 1 illustrates the FTIR spectrum of the prepared compound (1). The characteristic band appeared at 519 $\text{cm}^{-1}$  for stretching vibration of the aliphatic straight chain  $-\text{C}-\text{C}-$  skeleton, the band appeared at 749 $\text{cm}^{-1}$

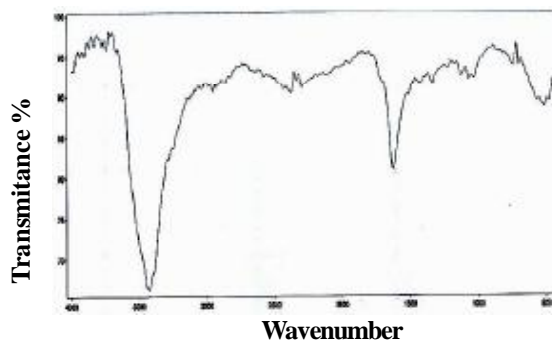


Figure 1 : FTIR diagram for compound (1)

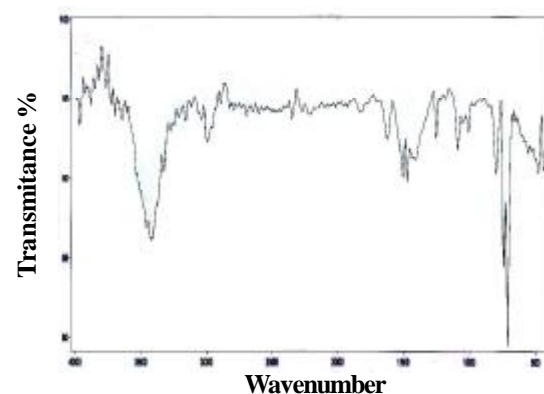


Figure 2 : FTIR diagram for compound (2)

for stretching vibration band appeared at 519 $\text{cm}^{-1}$  for stretching vibration of the aliphatic straight chain  $-\text{C}-\text{C}-$  skeleton, the band appeared at 749 $\text{cm}^{-1}$  for stretching vibration of  $-(\text{CH}_2)_2-$  groups, the band appeared at 1078  $\text{cm}^{-1}$  for stretching vibration of the primary  $-\text{CH}_2-\text{OH}$  group, the bands appeared at 1135 and 1340 $\text{cm}^{-1}$  for stretching vibration of  $-\text{CH}(\text{OH})-$  groups, the band appeared at 1627 $\text{cm}^{-1}$  for stretching vibration of the  $-\text{C}=\text{C}-$  group conjugated with  $-\text{C}=\text{O}$  group, the bands appeared at 2303, 2381 and 3424 $\text{cm}^{-1}$  for stretching vibration of  $-\text{NH}-$  and  $-\text{NH}_2$  groups and the band appeared at 2959 $\text{cm}^{-1}$  for stretching vibration of the  $-\text{CONH}-$  group.

Figure 2 illustrates the FTIR spectrum of the prepared compound (2). The characteristic band appeared at 528 $\text{cm}^{-1}$  for stretching vibration of the aliphatic straight chain  $-\text{C}-\text{C}-$  skeleton, the band appeared at 598  $\text{cm}^{-1}$  for stretching vibration of the primary  $-\text{CH}_2-\text{OH}$  group, the band appeared at 801 $\text{cm}^{-1}$  for stretching vibration of  $\text{CH}_3-\text{Si}-\text{CH}_3$  group, the band appeared at 914  $\text{cm}^{-1}$  for stretching vibration of  $\text{CH}_2-\text{NH}-\text{CH}_2$  group, the band appeared at 1025 $\text{cm}^{-1}$  for stretching vibration of the primary  $-\text{NH}_2$  group, the bands appeared at 1078, 1258 and 1401 $\text{cm}^{-1}$  for stretching vi-

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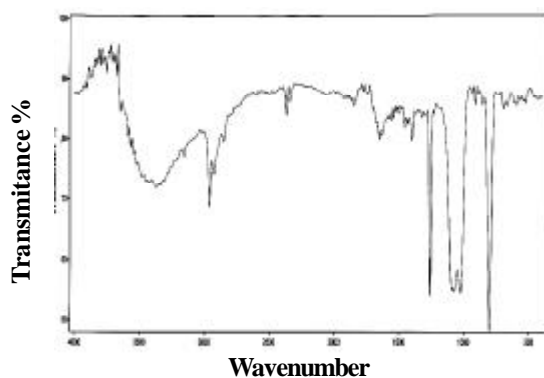


Figure 3 : FTIR diagram for compound (3)

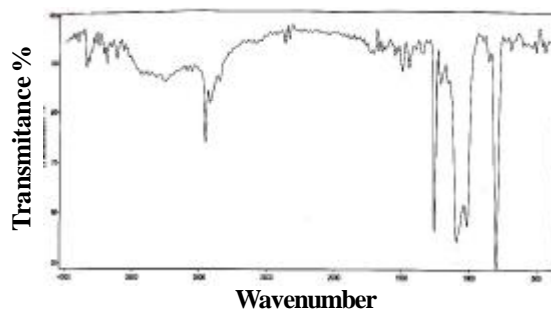
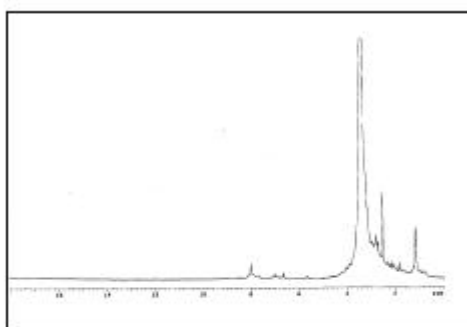


Figure 4 : FTIR diagram for compound (4)

Figure 5 : <sup>1</sup>H NMR diagram for compound (5)

bration of  $-\text{CH}(\text{OH})-$  groups, the band appeared at  $1658\text{cm}^{-1}$  for stretching vibration of the  $-\text{C}=\text{C}-$  group conjugated with  $-\text{C}=\text{O}$  group, the bands appeared at  $2339$ ,  $2367$  and  $3372\text{cm}^{-1}$  for stretching vibration of  $-\text{NH}-$  and  $-\text{NH}_2$  groups and the band appeared at  $2960\text{cm}^{-1}$  for stretching vibration of the  $-\text{CONH}-$  group.

Figure 3 illustrates the FTIR spectrum of the prepared compound (3). The characteristic band appeared at  $483\text{cm}^{-1}$  for stretching vibration of the aliphatic straight chain  $-\text{C}-\text{C}-$  skeleton, the band appeared at  $676\text{cm}^{-1}$  for stretching vibration of the primary  $-\text{CH}_2-\text{OH}$  group, the band appeared at  $711\text{cm}^{-1}$  for stretching vibration of  $\text{CH}_2-\text{NH}-\text{CH}_2$  group, the band appeared at  $743$

$\text{cm}^{-1}$  for stretching vibration of  $-(\text{CH}_2)_2-$  groups, the band appeared at  $803\text{cm}^{-1}$  for stretching vibration of  $\text{CH}_3-\text{Si}-\text{CH}_3$  group, the bands appeared at  $1017$ ,  $1098$  and  $1413\text{cm}^{-1}$  for stretching vibration of  $-\text{CH}(\text{OH})-$  groups, the band appeared at  $1260\text{cm}^{-1}$  for stretching vibration of  $\text{Ph}-\text{NH}_2$  group, the bands appeared at  $1477$  and  $1512\text{cm}^{-1}$  for stretching vibration of  $-\text{CH}_2-$  groups, the band appeared at  $1631\text{cm}^{-1}$  for stretching vibration of the  $-\text{C}=\text{C}-$  group conjugated with  $-\text{C}=\text{O}$  group, the bands appeared at  $1830$ ,  $2358$  and  $3424\text{cm}^{-1}$  for stretching vibration of the  $-\text{NH}-$  and  $-\text{NH}_2$  groups and the band appeared at  $2727\text{cm}^{-1}$  for stretching vibration of the  $-\text{CONH}-$  group.

Figure 4 illustrates the FTIR spectrum of the prepared compound (4). The characteristic bands appeared at  $444$  and  $1258\text{cm}^{-1}$  for stretching vibration of  $\text{Ph}-\text{NH}_2$  groups, the band appeared at  $512\text{cm}^{-1}$  for stretching vibration of the aliphatic straight chain  $-\text{C}-\text{C}-$  skeleton, the band appeared at  $697\text{cm}^{-1}$  for stretching vibration of the primary  $-\text{CH}_2-\text{OH}$  group, the band appeared at  $799\text{cm}^{-1}$  for stretching vibration of  $\text{CH}_3-\text{Si}-\text{CH}_3$  group, the bands appeared at  $1019$ ,  $1095$  and  $1353\text{cm}^{-1}$  for stretching vibration of  $-\text{CH}(\text{OH})-$  groups, the band appeared at  $1216\text{cm}^{-1}$  for stretching vibration of  $\text{CH}_3-\text{Si}-\text{CH}_3$  group, the band appeared at  $1453\text{cm}^{-1}$  for stretching vibration of  $-(\text{CH}_2)_2-$  groups, the band appeared at  $1502\text{cm}^{-1}$  for stretching vibration of  $\text{CH}_2-\text{NH}-\text{CH}_2$  group, the band appeared at  $1650\text{cm}^{-1}$  for stretching vibration of the  $-\text{C}=\text{C}-$  group conjugated with  $-\text{C}=\text{O}$  group, the bands appeared at  $2337$ ,  $2368$ ,  $3274$  and  $3513\text{cm}^{-1}$  for stretching vibration of the  $-\text{NH}-$  and  $-\text{NH}_2$  groups and the band appeared at  $2959\text{cm}^{-1}$  for stretching vibration of the  $-\text{CONH}-$  group

**<sup>1</sup>H NMR spectrum**

A representative <sup>1</sup>H NMR spectra of amide compound (1) is shown in figure 5 and amide siloxane derivatives: compound (2) is shown in figure 6, compound (2) in figure 7 and compound (4) in figure 8, respectively. Inspection of figures (5-8) reveal that the splitting pentlet signal at chemical shift  $\delta = 2.5$  is from the solvent DMSO (dimethylsulfoxide) and is disregarded, a splitting singlet at chemical shift  $\delta = 7.998$  for hydrogen proton of  $-\text{C}=\text{C}-\text{OH}$ , a splitting singlet at chemical shift  $\delta = 6.981$  for hydrogen proton of hydroxyl group of  $-\text{CH}-\text{OH}$ , a splitting doublet at chemical shift  $\delta = 6.5$  for hydrogen proton of  $-\text{C}-\text{CH}-\text{O}$ , a splitting high order signal at chemical shift  $\delta = 3.99$  for hydrogen

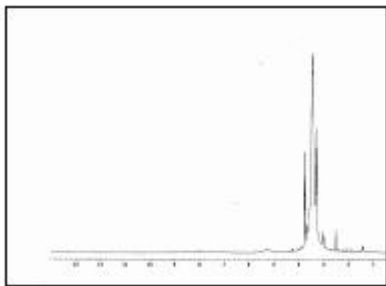
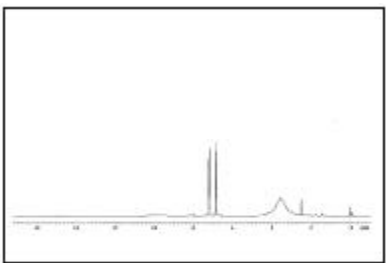
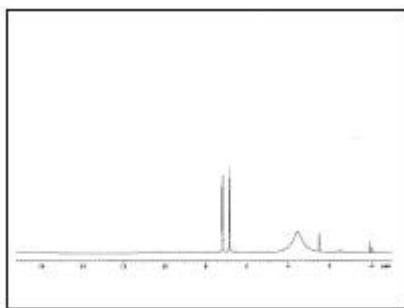
Figure 6 : <sup>1</sup>H NMR diagram for compound (6)Figure 7 : <sup>1</sup>H NMR diagram for compound (3)Figure 8 : <sup>1</sup>H NMR diagram for compound (4)

TABLE 1 : Elemental analyses of the synthesized compounds

Inhibitor	Compd.(1)	Compd.(2)	Compd.(3)	Compd.(4)	
Molecular weight	279.29	396.52	446.58	613.86	
Molecular formula	C <sub>10</sub> H <sub>21</sub> N <sub>3</sub> O <sub>6</sub>	C <sub>14</sub> H <sub>32</sub> N <sub>4</sub> O <sub>7</sub> Si	C <sub>18</sub> H <sub>34</sub> N <sub>4</sub> O <sub>7</sub> Si	C <sub>26</sub> H <sub>47</sub> N <sub>5</sub> O <sub>8</sub> Si <sub>2</sub>	
C%	Calculated	43.01	42.41	48.41	50.87
	Found	43.07	42.35	48.48	50.80
H%	Calculated	7.57	8.13	7.67	7.72
	Found	7.53	8.18	7.58	7.78
N%	Calculated	15.05	14.13	12.55	11.41
	Found	15.09	14.08	12.59	11.47
Si %	Calculated	-	7.08	6.29	9.15
	Found	-	7.10	6.32	9.10

proton of  $-\text{CH}_2-\text{CH}-\text{C}-$ , a splitting two signal at chemical shift  $\delta = 3.568, 3.522$  for hydrogen proton of  $-\text{CH}_2-\text{CH}-$ , a splitting two triplet signals at chemical shift  $\delta = 2.938, 2.717$  for hydrogen proton of ethylene group of  $-\text{CO}-\text{NH}-\text{C}_2\text{H}_4-\text{NH}-$ , a splitting two triplet signals at chemical shift  $\delta = 2.039, 1.788$  for hydrogen proton of ethylene group of  $-\text{NH}-\text{C}_2\text{H}_4-\text{NH}_2$ , a splitting signals at chemical shift  $\delta = 1.232, 1.176$  and  $1.156$  for hy-

drogen proton of  $\text{CO}-\text{NH}-\text{C}-$ ,  $-\text{C}-\text{NH}-\text{C}-$  and  $-\text{C}-\text{NH}_2$  respectively. a splitting signal for hydrogen proton of  $-\text{Si}-\text{CH}_3$  at chemical shift  $\delta = 0.053, 0.058, 0.061$  for compounds (1-4) respectively, a splitting quartet at chemical shift  $\delta = 6.84, 7.8$  for hydrogen proton of phenyl ring for compounds (3 and 4), there are no signal at chemical shift  $= 7.998$  of  $-\text{C}=\text{C}-\text{OH}$  for compd. (4).

### Elementary analysis

TABLE 1 illustrates the calculated of elemental values are the same value of measured value.

### CONCLUSION

- Preparation of novel organo amide and amidesiloxane compounds (1-4).
- Purification of these compounds and confirmation by FT.IR, <sup>1</sup>H NMR and elemental analysis.
- The measuring and calculated elemental values the same.
- These compounds have more than one active groups.
- The silicone compound gave new properties.

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