Basrah Journal of Science Vol. 38 (1), 98-110, 2020

New Schiff Base Compounds with Benzene Moiety: FT-IR, ^{1}H and ^{13}C NMR Spectroscopy (1D) Studies

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Doi 10.29072/basjs.20200107

Abstract

New Schiff bases compounds containing benzene moiety has been synthesized from reaction between (4-(((4-(Alkyloxy) benzylidene) amino) methyl) phenyl)methan amine and 4-((6-bromohexyl) oxy)benzaldehyde or 4-(4-bromobutoxy)benzaldehyde. FT-IR of these compounds have been studied. The complete ^{1}H and ^{13}C NMR assignment of these compounds has been obtained using one-dimensional NMR techniques including ^{1}H and ^{13}C experiments. The data deduced from this study show that the alkyl chain and the phenyl rings are in different planes compared to the benzene ring.

Article inf.

Received: *28/3/2020*

Accepted

24/4/2020

Published

30/4/2020

Keywords:

Schiff bases,

Benzene, FT-IR,

1D NMR

1. Introduction

Schiff base (CH=N), is used as a linking group in connecting the rigid core groups. (4-(((4-(Alkyloxy) benzylidene) amino) methyl) phenyl) methane amine and 4-(4-bromobutoxy)benzaldehyde or 4-((6-bromohexyl) oxy) benzaldehyde compounds are capable to form Schiff bases. Though it provides a stepped core structure, yet it retains molecular linearity, hence providing higher stability and enabling the formation of mesophase [1]. On the other hand, the terminal alkoxye chain length plays important roles in the formation, type, thermal stability, and temperature range of the mesophase of the liquid crystalline compounds [2].

2. Experimental

2.1 Materials

Compounds **6aza-14aza** and **6aza'-14aza'**, (Figure 1), were prepared according to the method described by (Majumdar et al.,) [3]. An equimolar amounts of (4-(((4-(Alkyloxy) benzylidene) amino) methyl) phenyl)methan amine with the 4-((6-bromohexyl) oxy) benzaldehyde or 4-(4-bromobutoxy)benzaldehyde., both dissolved in absolute ethanol was refluxed in the presence of a catalytic amount of acetic acid glacial for **3h**, the mixture let to cool at room temperature, the precipitate was filtered then washed with hot ethanol, and let to dry, and finally recrystallized with ethanol.

2.2 Physical Measurements

FT-IR spectra of the intermediate and title compounds were recorded using (FT-IR) 8400s fourier transitions infrared spectrometer Shimadzu, Japan, in the frequency range (4000-400) cm-1 in the form of (KBr) disc.The NMR spectra were recorded in deuterated methyl sulphoxide (DMSO-d6, at 298 K on a Bruker 400MHz Ultrashied FT-NMR spectrometer equipped with a 5mm BBI inverse gradient probe. Chemical shifts were referenced to internal tetramethylsilane)TMS). The concentration of solute molecules was 50 mg in 1.0 mL of (DMSO-d6).

3. Result and Discussion

The data of the FT-IR spectra for compounds 6aza-14aza and 6aza'-14aza' are listed in respective Tables 1 and 2. The FT-IR spectra of compounds 14aza and 14aza' are shown in Figure 2. The aromatic C-H stretching vibrations split into two bands at 3061-3045 cm^{-1} and

3034-3018 cm^{-1} [4]. The aliphatic C-H alkanes stretching vibrations normally are appear in the region of 2925-2920 cm^{-1} and 2852 -2849 cm^{-1} [5]. In addition, the C=N group two bands was observed at the frequency 1651 cm^{-1} and 1637 cm^{-1} [6]. The aromatic C=C stretching vibrations are expected within the region 1605-1552 cm^{-1} and 1513-1471 cm^{-1} [7]. Ethers group (C-O) bands are usually observed in the region 1266-1253 cm^{-1} [8].

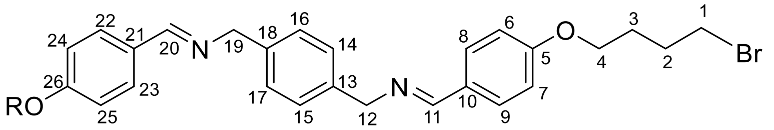
Table 1: FT-IR of compounds 6aza–14aza

υ max (cm^{-1})									
Atom	**6aza**	**7aza**	**8aza**	**9aza**	**10aza**	**11aza**	**12aza**	**13aza**	**14aza**
C-H aromatic	3061.48, 3024.13	3060.37, 3025.28	3061.19, 3024.16	3062.01, 3024.20	3061.49, 3026.19	3062.34, 3024.25	3061.15, 3024.18	306122, 3024.19	3061.18, 3024.18
C-H aliphatic	2924.01, 2851.67	2922.67, 2850.34	2921.23, 2852.48	2923.65, 2852.28	2923.46, 2849.05	2920.03, 2850.31	2921.14, 2852.52	2920.45, 2850.35	2920.03, 2852.52
C=N	1631.87	1632.49	1630.29	1631.45	1632.46	1629.34	1631.26	1630.23	1631.67
C=C aromatic	1604.08-1575.65, 1510.14-1471.61	1604.28-1576.32, 1510.26-1471.79	1603.89-1576.34, 1511.37-1471.41	1604.62-1575.69, 1511.58-1471.78	1604.89-1574.35, 1511.37-1471.93	1605.27-1574.28, 1510.17-1470.34	1604.38-1576.23, 1510.36-1471.35	1604.45-1576.24, 1511.34-1470.43	1604.66-1575.73, 1512.09-1471.59
C-O	1255.79	1254.49	1253.57	1255.34	1253.47	1255.49	1254.68	1255.89	1255.57
p-position	833.15	835.56	833.67	834.89	835.09	834.38	835.73	835.49	835.12

Table 2: FT-IR of compounds 6aza'–14aza'

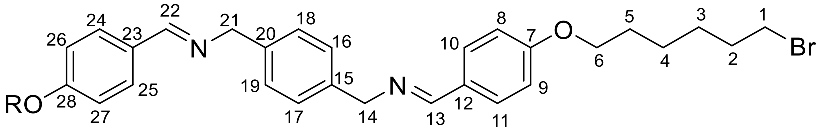
	υ max (cm^{-1})								
Atom	**6aza'**	**7aza'**	**8aza'**	**9aza'**	**10aza'**	**11aza'**	**12aza'**	**13aza'**	**14aza'**
C-H aromatic	3061.29, 3024.38	3061.34, 3024.39	3060.12, 3024.12	3062.41, 3024.20	3060.24, 3024.59	3061.45, 3024.36	3060.78, 3023.89	3060.37, 3024.22	3062.12, 3024.18
C-H aliphatic	2922.67, 2853.14	2920.82, 2850.15	2923.09, 2850.12	2921.39, 2852.26	2923.57, 2850.19	2922.46, 2851.47	2921.03, 2850.57	2920.76, 2851.34	2920.03, 2850.59
C=N	1650.18	1648.75	1649.27	1648.54	1651.78	1650.23	1649.89	1648.96	1649.02
C=C aromatic	1603.38-1578.39, 1513.27-1469.87	1602.74-1577.35, 1510.27-1468.46	1603.36-1576.39, 1511.49-1470.39	1602.65-1577.59, 1512.81-1470.36	1603.56-1578.46, 1511.12-1470.17	1603.43-1576.38, 1511.48-1468.37	1601.34-1575.35, 1511.48-1469.35	1602.69-1576.34, 1510.65-1470.45	1602.74-1577.66, 1512.09-1469.66
C-O	1256.75	1257.32	1256.45	1257.68	1258.45	1257.19	1255.35	1256.89	1257.50
p-position	834.96	833.69	835.85	834.78	833.53	834.86	835.19	834.94	835.45

The data of the ^{1}H-NMR chemical shifts for title compounds 6aza-14aza and 6aza'-14aza' in DMSO solution are listed in respective Tables 3 and 4. The ^{1}H-NMR spectra for compounds 12aza and 14aza' are shown in Figure 3. The singlet signal at δ = 8.45-8.35 ppm was attributed to the imine group protons (CH=N) [9]. Resonances of the aromatic protons (H6, H7 and H8, H9) were observed as doublet within the chemical shifts range δ =7.89-7.82 ppm and δ =7.76-7.72 ppm respectively. The doublet signals at δ =7.72-7.67 ppm and δ =7.44-7.36 ppm were ascribed to the phenyl protons (H22, H23 and H14, H15) [10, 11]. While the doublet at δ =7.24-7.21 ppm was assigned to the aromatic protons (H16, H17) [11]. Also spectra are show doublet signal at δ =6.92-6.87 ppm, were assigned for aromatic protons (H24, H25) [10]. A signal attributable to the presence of methylene protons for the NCH_2 group was observed at chemical shifts range δ= 4.80-4.70 [12]. The triplet at the chemical shifts δ =4.20-4.09 ppm and δ= 4.07-4.00 ppm were indicated successful methylene group protons of the OCH_2 [13]. The protons residing in the alkyl chain ($(CH_2)_n$) in all compounds 12aza and 14aza' chain can be substantiated by multiples at the range of δ =2.46-1.09 ppm. Moreover, a signal which appears as triplet at the chemical shift δ =0.88-0.80 ppm can be assigned to the terminal methyl (CH_3) [14, 15].



6aza-14aza

R= C_6H_{13}-$C_{14}H_{29}$



6aza'-14aza'

R= C_6H_{13}-$C_{14}H_{29}$

Figure 1: Molecular structure with numbering scheme for compounds 6aza-14aza and 6aza'-14aza'

Table 3: ^{1}H-NMR chemical shifts (ppm) of compounds 6aza–14aza

	Chemical shift (ppm)								
Atom	**6aza**	**7aza**	**8aza**	**9aza**	**10aza**	**11aza**	**12aza**	**13aza**	**14aza**
CH_2-O	4.10	4.09	4.13	4.11	4.10	4.09	4.11	4.11	4.10
CH_2-N	4.68	4.71	4.69	4.70	4.70	4.71	4.70	4.70	4.71
H6, H7	7.84	7.85	7.86	7.83	7.87	7.84	7.85	7.84	7.87
H8, H9	7.73	7.78	7.77	7.75	7.76	7.74	7.76	7.74	7.75
CH=N	8.40	8.39	8.41	8.40	8.43	8.42	8.44	8.42	8.43
H14, H15	7.39	7.42	7.41	7.43	7.42	7.44	7.40	7.42	7.41
H16, H17	7.20	7.19	7.22	7.23	7.21	7.20	7.22	7.21	7.23
H22, H23	7.70	7.71	7.73	7.70	7.74	7.72	7.73	7.71	7.71
H24, H25	6.98	6.99	6.93	6.94	6.93	6.95	6.93	6.91	6.90
$(CH_2)_n$	2.55-1.13	2.45-1.09	2.46-1.12	2.47-1.12	2.45-1.12	2.46-1.07	2.47-1.10	2.44-1.15	2.45-1.10
CH_3	0.89	0.87	0.91	0.84	0.85	0.86	0.87	0.85	0.86

Table 4: ^{1}H-NMR chemical shifts (ppm) of compounds 6aza'–14aza'

	Chemical shift (ppm)								
Atom	**6aza'**	**7aza'**	**8aza'**	**9aza'**	**10aza'**	**11aza'**	**12aza'**	**13aza'**	**14aza'**
CH_2-O	4.11	4.09	4.15	4.10	4.10	4.09	4.11	4.12	4.10
CH_2-N	4.69	4.73	4.68	4.71	4.70	4.71	4.70	4.69	4.71
H8, H9	7.86	7.84	7.88	7.87	7.85	7.83	7.85	7.85	7.86
H10, H11	7.75	7.79	7.73	7.77	7.76	7.72	7.76	7.75	7.76
CH=N	8.39	8.38	8.42	8.44	8.45	8.43	8.41	8.41	8.40
H16, H17	7.41	7.44	7.39	7.40	7.45	7.43	7.41	7.40	7.43
H18, H19	7.26	7.18	7.21	7.25	7.24	7.21	7.22	7.20	7.22
H24, H25	7.67	7.68	7.72	7.71	7.73	7.69	7.70	7.72	7.70
H26, H27	6.99	6.97	6.93	6.90	6.91	6.92	6.93	6.90	6.89
$(CH_2)_n$	2.09-1.18	2.11-1.27	2.13-1.25	2.39-1.24	2.14-1.18	2.21-1.20	2.11-1.25	2.19-1.21	2.10-1.25
CH_3	0.84	0.86	0.93	0.89	0.88	0.82	0.87	0.83	0.86

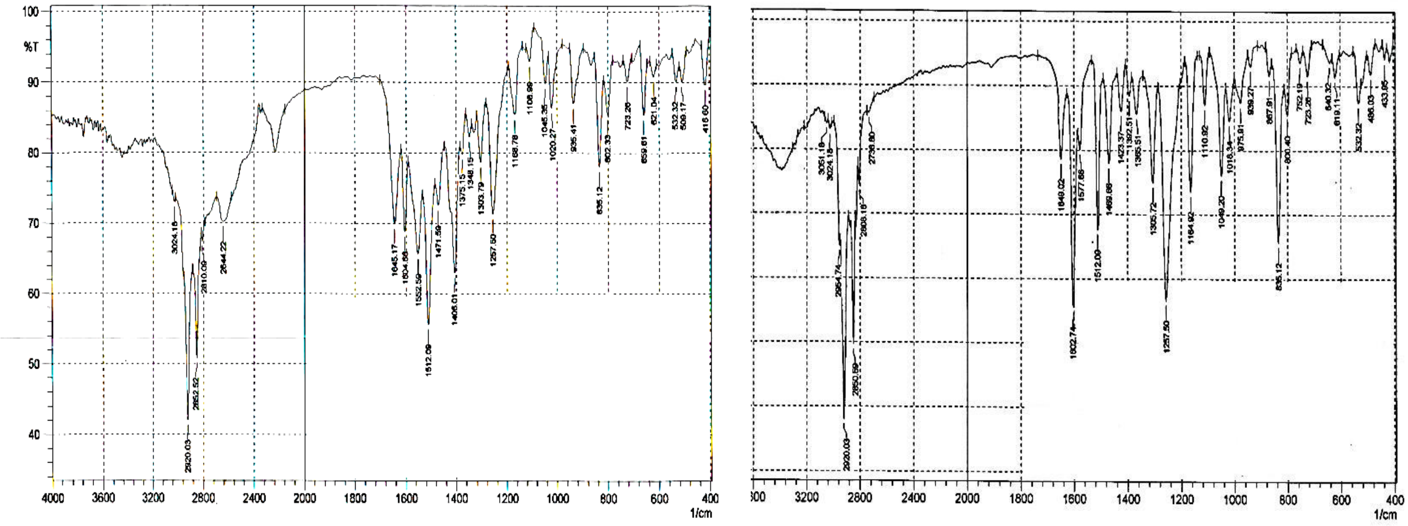
The data of the ^{13}C-NMR chemical shifts for title compounds 6aza-14aza and 6aza'-14aza' in DMSO solution are listed in respective Tables 5 and 6. The ^{13}C-NMR spectra for compounds 12aza and 14aza' are shown in Figure 4. The peak of the imine group CH=N was assigned at δ =166.57-163.38 ppm [14, 16]. The signals within the range of δ =154.13-109.93 ppm were assigned to the aromatic carbons in the phenyl rings [17, 13]. The OCH_2 group appears in the region of δ =69.31-67.92, and δ =66.27-64.73 ppm [18]. Similarly, the ^{13}C-NMR signals for NCH_2 were found at δ =52.13-49.58 ppm and δ =46.87-41.35 ppm [14, 19]. The peaks at δ =32.67-21.66 ppm were attributed to the methylene carbons of the long alkyl chains, while the peak at the chemical shift δ =15.96-14.58 ppm could be assigned to the terminal methyl carbon of the alkyl chains [20].

Table 5: ^{13}C-NMR chemical shifts (ppm) of compounds 6aza–14aza

	Chemical shift (ppm)								
Atom	**6aza**	**7aza**	**8aza**	**9aza**	**10aza**	**11aza**	**12aza**	**13aza**	**14aza**
C=N	165.81	165.76	166.01	164.45	165.16	163.35	164.19	163.67	165.21
C-O	69.35, 64.32	68.94, 64.78	69.55, 63.78	67.32, 65.18	69.72, 64.67	68.24, 64.89	68.33, 65.14	67.79, 66.35	68.89, 66.10
C-N	51.26, 41.76	51.56, 42.45	51.34, 42.67	51.33, 41.79	50.33, 42.11	52.01, 41.22	50.16, 42.49	52.34, 41.45	51.23, 40.89
C5,C6,C7,C8,C9,C10,C13,C14,C15,C16, C17,C18,C21,C22,C23, C24, C25,C26	135.98-115.29	135.32-114.24	134.29-115.56	135.89-114.98	135.78-112.75	136.03-116.24	134.89-115.92	135.67-114.89	133.89-115.12
$(CH_2)_n$	31.67-21.59	32.13-21.67	30.98-21.89	30.56-22.46	30.24-20.67	30.08-23.39	31.84-22.23	32.09-23.12	30.12-21.34
CH_3	15.79	14.97	15.43	15.89	13.89	16.02	15.22	15.68	14.89

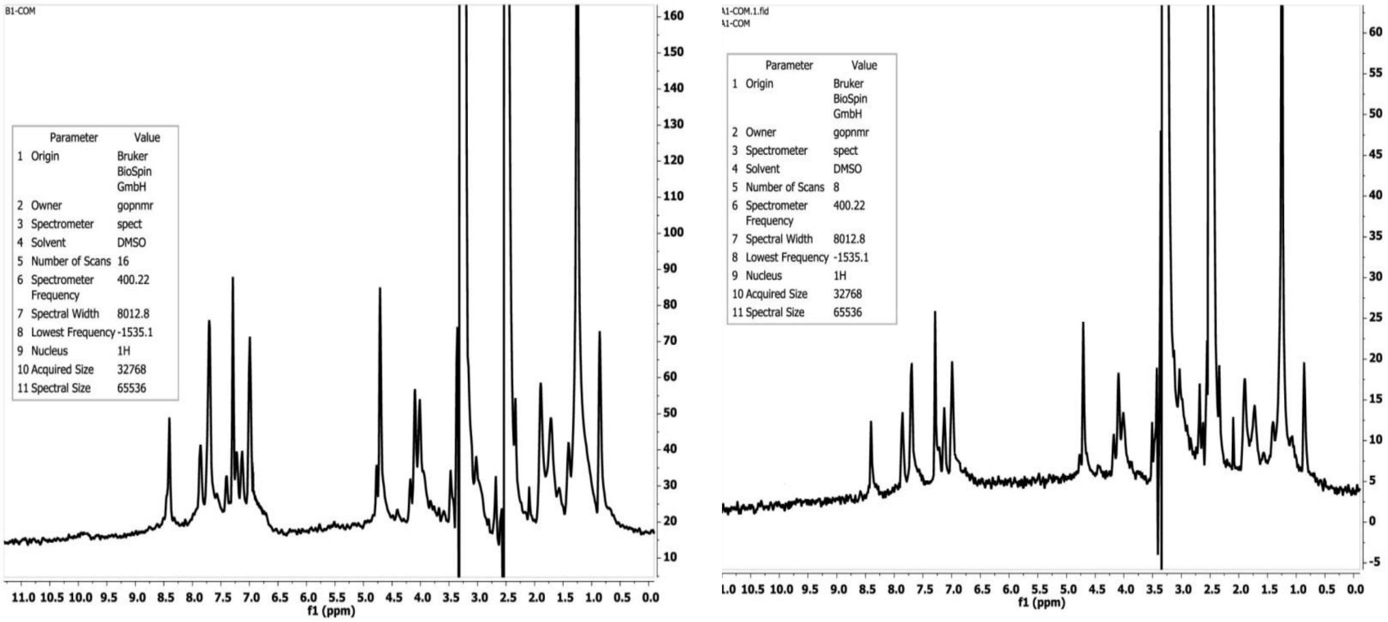
Table 6: ^{13}C-NMR chemical shifts (ppm) of compounds 6aza'–14aza'

	Chemical shift (ppm)								
Atom	**6aza'**	**7aza'**	**8aza'**	**9aza'**	**10aza'**	**11aza'**	**12aza'**	**13aza'**	**14aza'**
C=N	165.49	164.22	164.35	165.89	163.98	164.65	166.01	164.99	165.87
C-O	67.34, 64.76	69.31, 64.28	67.33, 64.25	68.29, 65.36	67.34, 65.37	69.35, 65.28	68.95, 65.77	68.73, 63.28	68.23, 64.52
C-N	45.29, 51.46	46.22, 53.29	45.78, 51.39	44.34, 51.68	44.34, 51.97	44.98, 51.78	44.89, 53.67	45.33, 51.34	45.21, 52.78
C5,C6,C7,C8,C9,C10,C13,C14,C15,C16, C17,C18,C21,C22,C23, C24, C25,C26	134.67-114.38	136.27-115.35	135.39-113.56	134.19-114.89	136.12-115.33	135.21-114.23	134.11-115.32	133.45-115.28	133.81-115.41
$(CH_2)_n$	32.56-22.68	31.48-22.56	32.97-20.34	31.61-22.95	31.56-23.04	31.89-20.89	31.74-23.63	31.34-21.38	32.72-22.83
CH_3	15.22	14.37	115.37	14.68	15.78	15.67	14.47	15.29	14.93



(a) (b)

Figure 2: (a) FT-IR spectrum of compound 14aza. (b) FT-IR spectrum of compound 14aza'.



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8	Lowest Frequency	-1535.1
9	Nucleus	1H
10	Acquired Size	32768
11	Spectral Size	65536

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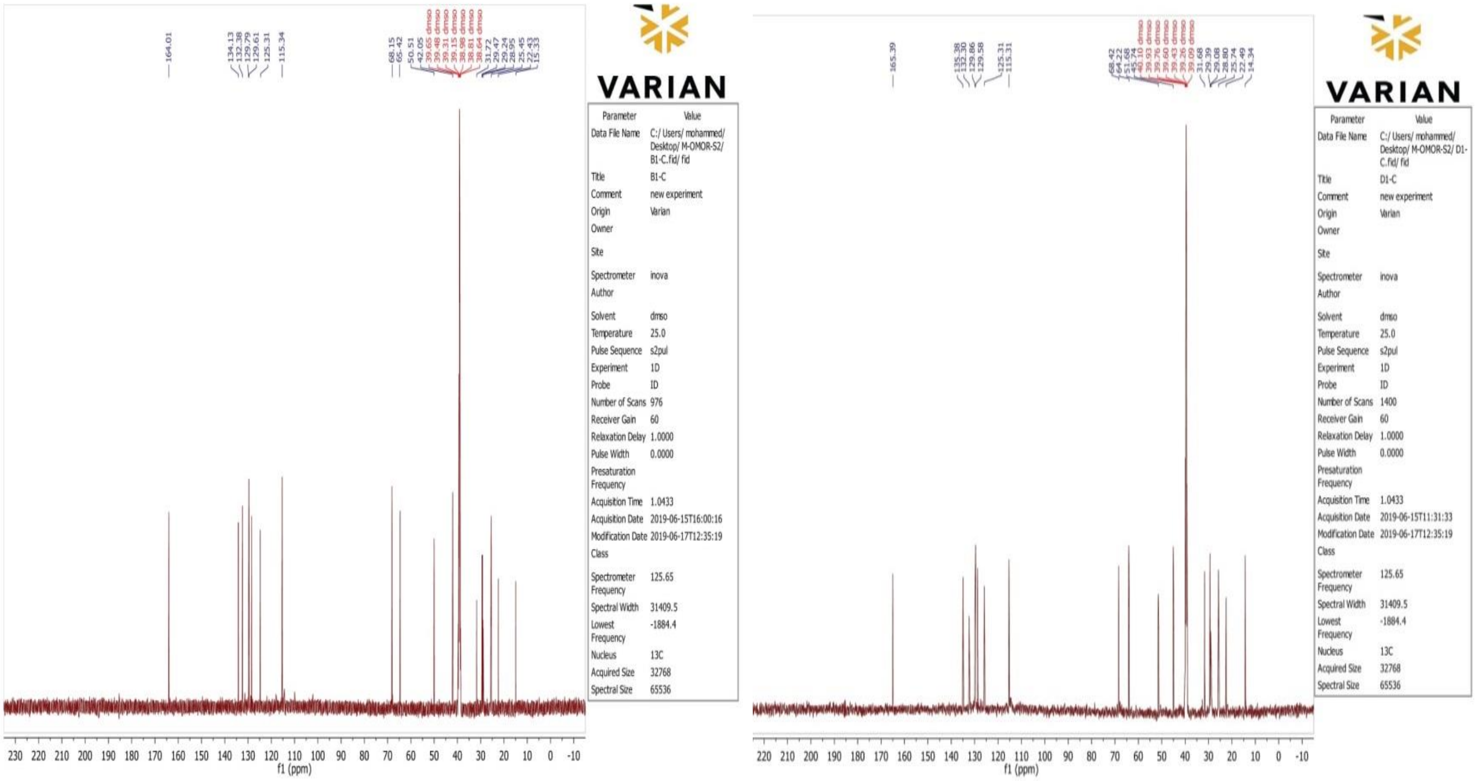
9	Nucleus	1H
10	Acquired Size	32768
11	Spectral Size	65536

(c) (d)

Figure 3: (c) ^{1}H-NMR spectrum of compound 12aza. (d) ^{1}H-NMR spectrum of compound 14aza'.



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(e)

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Nucleus	13C
Acquired Size	32768
Spectral Size	65536

(f)

Figure 4: (e) ^{13}C-NMR spectrum of compound 12aza. (f) ^{13}C-NMR spectrum of compound 14aza'.

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المستخلص

تم تصنيع مركبات قواعد شيف الجديدة التي تحتوي على جزء البنزين من التفاعل بين (4 - (((4- (ألكيلوكس بنزيلايدين) أمينو) ميثيل) فينيل) ميثان أمين و 4 - ((6-بروموهكسيل) أوكسي) بنزالديهايد أو 4- (4-بروموبوتوكسي) بنزالدهايد. وقد تمت دراسة FT-IR لهذه المركبات. تم الحصول على التعيين الكامل لـ H1 و 13 C NMR لهذه المركبات باستخدام تقنيات NMR أحادية البعد بما في ذلك تجارب H1 و 13 C. تشير البيانات المستخلصة من هذه الدراسة إلى أن سلسلة الألكيل وحلقات فينيل في مستويات مختلفة مقارنة بحلقة البنزين.