Ministry of Higher Education
and Scientific Research

Journal of Kufa for Chemical Sciences

A refereed

Research Journal Chemical Sciences
Vol.2 No.8 Year 2022
ISSN 2077-2351

New Bis-Chalcon Long-Chain Alkyl Amide Derivatives Synthesized Via Direct Reaction

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Abstract

One of the most prevalent transformations in organic chemistry is the production of amide bonds by chemical processes. Seven new long alkyl amide compounds were produced in this study using a direct reaction. The reaction of 3,3'-(1,4-phenylene) bis(1-(4-aminophenyl) prop-2-en-1-one) with various carboxylic acids (mono and di) in the presence of a suitable solvent and an amount of sodium hydroxide yielded four novel compounds (B1-B4). The remaining three amide derivatives (B5, B6, and B7) were prepared under the same conditions by reacting new compounds (B2, B3, and B4) with acrylamide. All the new prepared compounds were characterized by using FT-IR, 1HNMR, and mass spectroscopy, and they confirmed the expected structures of all the derivatives.

Introduction

The amide bond is widely prevalent in both naturally occurring and synthetic compounds [1]. The presence of the amide functional group in many important compounds, such as proteins, fabrics, fertilizers, insecticides, plastics, drugs, and a wide range of synthetic structures, demonstrates its significance [2].Amide compounds are a very important category of organic compounds, so the formation of amide compounds has attracted significant interest due to their importance in organic and bioorganic chemistry, their value as intermediates in organic synthesis, and a wide range of applications in the chemical industry [3,4,5]. Because of the amide functionality's apparent importance, by far the most popular procedure is condensation between a carboxylic acid and an amine [6]. As environmentally friendly alternatives to standard chemical transformations are sought, the use of condensing and activating agents is becoming increasingly unpopular [7].The amide linkages appear as an important structural component in peptides, many natural polymeric products, and pharmaceuticals. They are not only the primary chemical links between proteins and pharmaceuticals, but they are also the foundation for some of the most commonly used synthetic polymers [3, 8, 9].

In this paper, the reaction of bis-chalcone with two chains of carboxylic acids was used to prepare a novel long-chain amide molecule. Through a direct reaction, the first chain is made up of monocarboxylic acids and the second chain is made up of dicarboxylic acids. In the same way that the compounds above reacted with acrylic amides to produce other new series of compounds.

2. Experimental

2.1 Materials and Instruments

All three Sigma-Aldrich, Merck, and Fluka companies supplied all the chemicals and solvents used in this study. On an open capillary status thermal point device, the uncorrected melting points of the synthesized compounds were recorded (England).

The FTIR spectra of all synthesized compounds were measured as KBr discs in the region of 4000-400 cm-1 by using Shimadzu FTIR-8400 (Japan). The 1H-NMR spectra were obtained using a Bruker Avance DRX 500 MHz (Germany) in deuterated DMSO-d6 solvent and TMS as an internal reference. The expected structure of some synthesized new compounds was also determined based on their mass measurement. The mass spectra were obtained by using the Shimadzu TQ8040 (Japan). Thin layer chromatography of the starting materials and products was performed by using Merck chromatography sheets (Germany). The spot was visualized by exposing the dry plate to UV light.

2.2 Synthesis Methods

2.2.1 General Procedure for Synthesis of New Compounds (B1- B7)

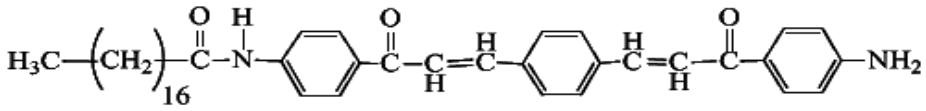
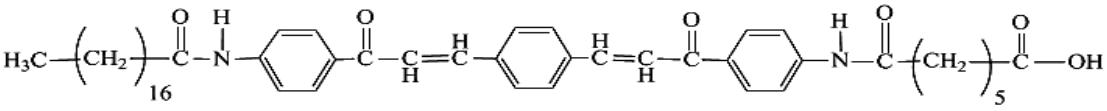
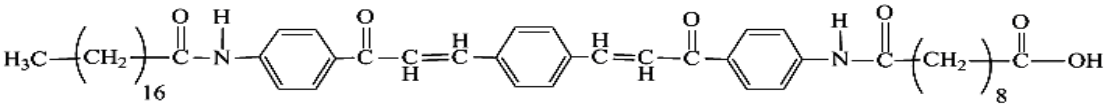
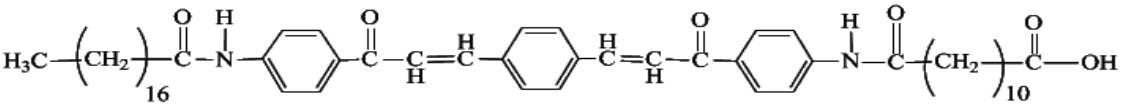
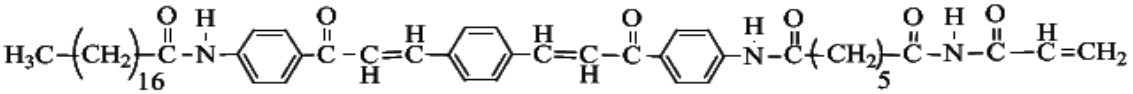
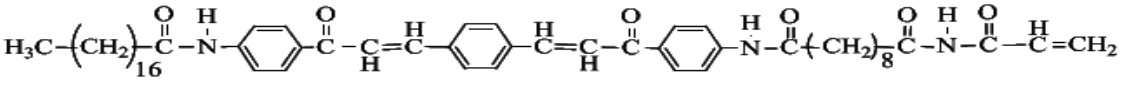
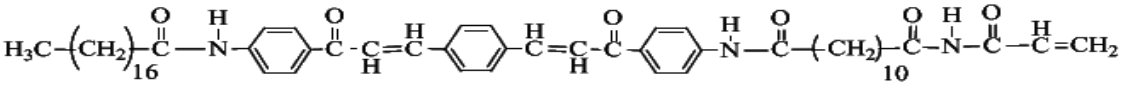
Six mmol of 3,3'-(1,4-phenylene) bis(1-(4-aminophenyl) prop-2-en-1-one) [A] prepared as mentioned in the literature [10] was dissolved in ethanol (25 ml) to make solution (1). In another flask, 6 mmol of carboxylic acid was dissolved in 10 ml of ethanol to make solution (2). Then, solution (2) was added to the solution (1) in a three-neck round-bottom flask with constant stirring. After 20 minutes, 20 ml of 10% aqueous sodium hydroxide solution were added dropwise to the mixture, and then the mixture was refluxed at 80–90 °C for about 6 hours in an oil bath with constant stirring [11]. The reaction was monitored by TLC using an (8:2) [methanol-benzene] eluent. After the reaction was completed, the reaction mixture was cooled to room temperature. The reaction mixture was poured into 150 ml of cold water and the precipitated solid was filtered off, washed several times with water until the filtrate was neutral to litmus, and dried. The obtained product powder had a different color and was recrystallized with a mixture of ethanol and water (1:1). The reactions of preparing the new compounds were illustrated in Scheme (1), Table (1) shows the reactants used, their quantity, and the names of the new prepared compounds (B1-B7), and Table (2) exhibits the symbols of the compounds and their structures.

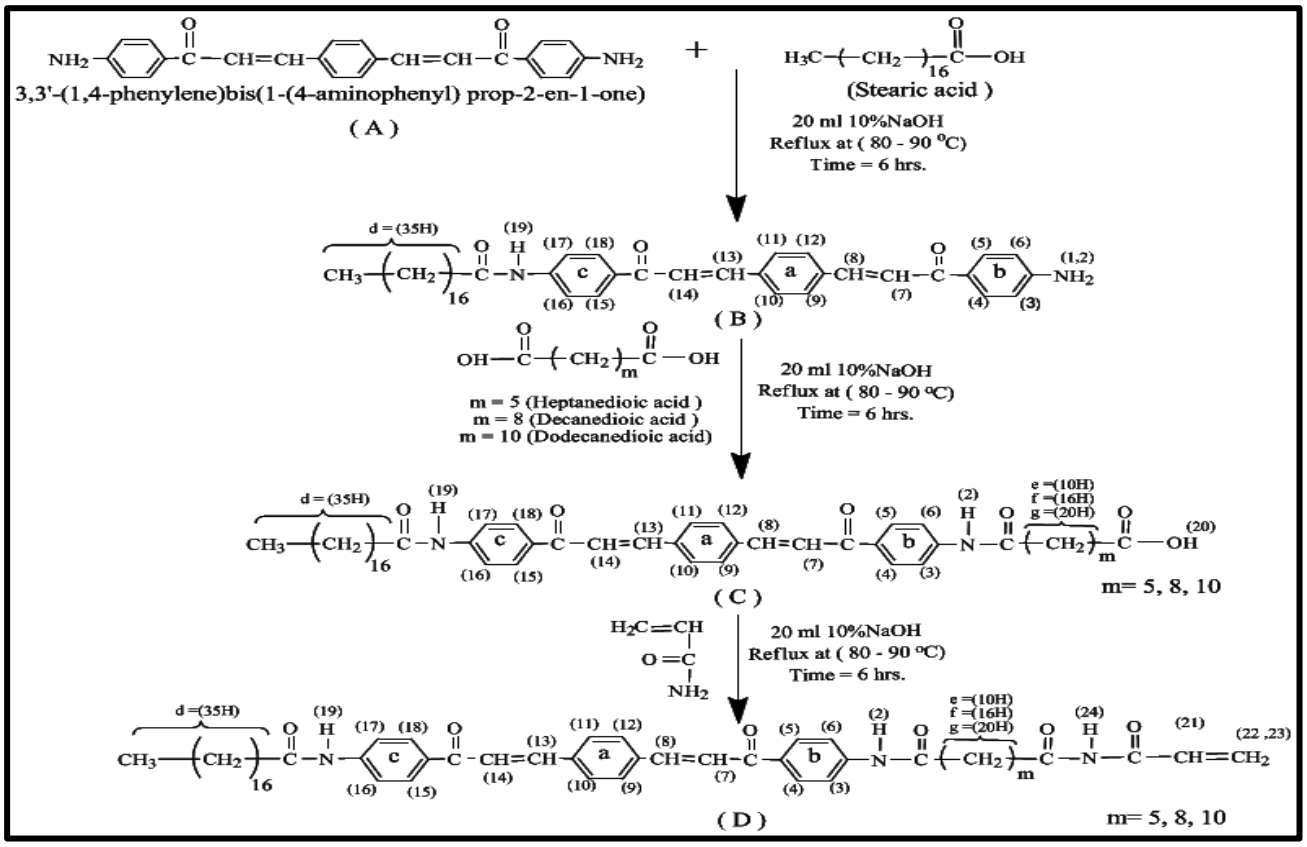
Table (1): The newly prepared compounds [B1-B7] and the reactants used and their amount.

	Reactants		Products
	1	2	
1	3,3'-(1,4-phenylene)bis(1-(4-amino phenyl)prop-2-en-1-one) [A]6m.mole	Stearic acid (6 m.mole)	N-(4-(3-(4-(3-(4-aminophenyl)-3-oxoprop-1-en-1-yl)phenyl) acryloyl)phenyl)stearamide **[B1]**
2	[B1] 6m.mole	Heptanedioic acid(6m.mole)	7-oxo-7-((4-(-3-(4-(3-oxo-3-(4-stearamidophenyl)prop-1-en-1-yl) phenyl)acryloyl)phenyl)amino)heptanoic acid **[B2]**
3	[B1] 6m.mole	Decanedioic acid(6m.mole)	10-oxo-10-((4-(3-(4-(3-oxo-3-(4-stearamidophenyl)prop-1-en-1-yl)phenyl)acryloyl)phenyl)amino)decanoicacid **[B3]**
4	[B1] 6m.mole	Dodecanedioic acid(6m.mole)	12-oxo-12-((4-(3-(4-(3-oxo-3-(4-stearamidophenyl)prop-1-en-1-yl)phenyl)acryloyl)phenyl)amino)dodecanoicacid **[B4]**
5	[B4] 6m.mole	Acrylamide (6m.mole)	N1-acryloyl-N7-(4-(3-(4-(3-oxo-3-(4-stearamidophenyl)prop-1-en-1-yl)phenyl)acryloyl)phenyl)heptanediamide **[B5]**
6	[B3] 6m.mole	Acrylamide (6m.mole)	N1-acryloyl-N10-(4-(3-(4-(3-oxo-3-(4-stearamidophenyl)prop-1-en-1-yl)phenyl)acryloyl) phenyl)decanediamide **[B6]**
7	[B2] 6m.mole	Acrylamide	N1-acryloyl-N12-(4-(3-(4-(3-oxo-3-(4-stearamidophenyl)prop-1-

		(6m.mole)	en-1-yl)phenyl)acryloyl)phenyl)dodecanediamide **[B7]**

Table (2): The compounds Symbols and the Structure of compounds.

Compounds Symbol	Structure of compounds
[B1]	 **N-(4-(3-(4-(3-(4-aminophenyl)-3-oxoprop-1-en-1-yl)phenyl)acryloyl)phenyl) stearamide**
[B2]	 **7-oxo-7-((4-(-3-(4-(3-oxo-3-(4-stearamidophenyl)prop-1-en-1-yl)phenyl)acryloyl)phenyl)amino) heptanoic acid**
[B3]	 **10-oxo-10-((4-(3-(4-(3-oxo-3-(4-stearamidophenyl)prop-1-en-1-yl)phenyl)acryloyl)phenyl)amino) decanoicacid**
[B4]	 **12-oxo-12-((4-(3-(4-(3-oxo-3-(4-stearamidophenyl)prop-1-en-1-yl)phenyl)acryloyl)phenyl)amino) dodecanoicacid**
[B5]	 **N^1-acryloyl-N^7-(4-(3-(4-(3-oxo-3-(4-stearamidophenyl)prop-1-en-1-yl)phenyl)acryloyl)phenyl) heptanediamide**
[B6]	 **N^1-acryloyl-N^{10}-(4-(3-(4-(3-oxo-3-(4-stearamidophenyl)prop-1-en-1-yl)phenyl)acryloyl) phenyl)decanediamide**
[B7]	 **N^1-acryloyl-N^{12}-(4-(3-(4-(3-oxo-3-(4-stearamidophenyl)prop-1-en-1-yl)phenyl)acryloyl)phenyl) dodecane diamide**



Scheme (1): general reaction of synthesis compounds.

2.2.2 Synthesis of N-(4-(3-(4-(3-(4-aminophenyl)-3-oxoprop-1-en-1-yl) phenyl) acryloyl) phenyl) stearamide (B1)

This compound is synthesized by the reaction of six mmol of compound (A) with six mmol of stearic acid, resulting in a yield of 77% and a melting point of 178–180 °C. m/z [M+] = 634 $C_{42}H_{54}N_2O_3$. . FT-IR Spectrum: ν (cm^{-1}) [12-15]: 1712 (C = O) amide and ketone, 3329-3429 (–NH_2), 3217 (–NH) amide, 1640 (C=C) aliphatic, 3217(C-H) aromatic. ^{1}H-NMR: δ (DMSO-d6/ppm) [16-20]: 1.48-1.90 (m, 30H, −(CH)−), 2.94-2.98 (t, 3H, –CH_3), 3.73-3.79 (t, 2H, –CH_2) , 4.03 (s, 2H, −NH_2), 5.77-5.97 (dd, 4H, aromatic ring c), 3.83 (dd, 4H, aromatic ring b), 7.9 (s, 4H, aromatic ring a), 5.77-5.97 (dd, 2H, O=C-CH=CH & O=C-CH=CH), 10 (s, 1H, -NH).

2.2.3 Synthesis of 7-oxo-7-((4-(-3-(4-(3-oxo-3-(4-stearamidophenyl) prop-1-en-1-yl) phenyl) acryloyl)phenyl)amino)heptanoic acid (B2)

The reaction of six mmol of compound (B1) with six mmol of heptanedioic acid synthesizes this compound. After purification, a light orange powder is obtained. The product had an 80% yield and a melting point of 185-187 °C. M/z [M+] = 777 $C_{49}H_{64}N_2O_6$. . FT-IR Spectrum: ν (cm^{-1}) [12-15]: 1708 (C = O) amide and ketone, 3421 (–OH), 3240 (–NH) amide, 1640 (C=C) aliphatic, 3200 (C-H) aromatic. δ ^{1}H-NMR (DMSO-d6/ppm) [16-20]: 1.48-1.90 (m, 55H, −(CH)−), 2.94 - 2.98 (t, 3H, −CH_3), 3.73 -3.79 (t, 2H, −CH_2), 5.77-5.97 (dd, 4H, aromatic ring c), 3.83 (dd, 4H, aromatic ring b), 7.9 (s, 4H, aromatic ring a), 5.77-5.97 (dd, 2H, O=C-CH=CH & O=C-CH=CH), 10 (s, 1H, -NH), 12 (s, 1H, −OH).

2.2.4 Synthesis of 10-oxo-10-((4-(3-(4-(3-oxo-3-(4-stearamidophenyl)prop-1-en-1-yl)phenyl) acryloyl)phenyl)amino)decanoicacid [B3]

The reaction of six mmol of compound (B1) with six mmol of dodecanedioic acid prepares this compound. After purification, a light orange powder is obtained. The product was

obtained at a yield of 82.2% and a melting point of 194-196 °C. M/z [M+] = 819 $C_{52}H_{70}N_2O_6$. . FT-IR Spectrum: ν (cm^{-1}) [12-15]: 1708 (C=O) amide and ketone, 3425 (–OH), 3390 (–NH) amide, 1630 (C=C) aliphatic, 3190 (C-H) aromatic. δ ^{1}H-NMR (DMSO-d6/ ppm) [16-20]: 1.48-1.90 (m, 55H, −(CH)−), 2.94 - 2.98 (t, 3H, $-CH_3$), 3.73 -3.79 (t, 2H, $-CH_2$), 5.77-5.97 (dd, 4H, aromatic ring c), 3.83 (dd, 4H, aromatic ring b), 7.9 (s, 4H, aromatic ring a), 5.77-5.97 (dd, 2H, O=C-CH=CH & O=C-CH=CH), 10 (s, 1H, -NH), 12 (s, 1H, −OH).

2.2.5 Synthesis of 12-oxo-12-((4-(3-(4-(3-oxo-3-(4-stearamidophenyl)prop-1-en-1-yl)phenyl) acryloyl)phenyl)amino)dodecanoicacid [B4]

This compound is created by reacting six mmol of compound (B1) with six mmol of decanedioic acid. After purification, a light orange powder is obtained. The product was obtained at a yield of 79.9% with a melting point of 191–193 °C. M/z [M+] = 847 $C_{54}H_{74}N_2O_6$. FT-IR Spectrum: ν (cm^{-1}) [12–15]:1712 (C = O) amide and ketone, 3429 (–OH), 3235 (–NH) amide, 1630 (C = C) aliphatic, 3210 (C-H) aromatic1712 (C = O) amide. δ ^{1}H-NMR (DMSO-d6/ppm) [16–20]: 1.48-1.90 (m, 55H, (CH)), 2.94-2.98 (t, 3H, CH3), 3.73-3.79 (t, 2H, CH2), 5.77-5.97 (dd, 4H, aromatic ring c), 3.83 (dd, 4H, aromatic ring b), 7.9 (s, 4H, aromatic ring a), 5.77-5.97 (dd, 2H, O=C-CH=CH & O=C-CH=CH), 10 (s, 1H,-NH), 12 (s, 1H, OH).

2.2.6 Synthesis of N^1-acryloyl-N^7-(4-(3-(4-(3-oxo-3-(4-stearamidophenyl)prop-1-en-1-yl) phenyl) acryloyl)phenyl)heptanediamide [B5]

Six mmol of component (B2) was reacted with six mmol of acrylamide to make this chemical. A pale yellow powder is obtained after purification. The product was obtained at a yield of 72 % and a melting point of 194-197°C. M/z [M+] = 830 $C_{52}H_{67}N_3O_6$. . FT-IR Spectrum: ν (cm^{-1}) [12-15]: 1717 (C=O) amide and ketone, 3444 (–NH) amide, 1650 (C=C) aliphatic, 3230 (C-H) aromatic. δ ^{1}H-NMR (DMSO-d6 /ppm) [16-20]: 1.48-1.90 (m, 55H, −(CH)−), 2.94 - 2.98 (t, 3H, $-CH_3$), 3.73 -3.79 (t, 2H, $-CH_2$), 5.77-5.97 (dd, 4H, aromatic ring c), 3.83 (dd, 4H, aromatic ring b), 7.9 (s, 4H, aromatic ring a), 5.77-5.97 (dd, 2H, O=C-CH=CH & O=C-CH=CH), 10 (s, 1H, -NH), 12 (s, 1H, −OH), 13 (s, 1H, -NH).

2.2.7 Synthesis of N^1-acryloyl-N^{10}-(4-(3-(4-(3-oxo-3-(4-stearamidophenyl)prop-1-en-1-yl) phenyl) acryloyl) phenyl)decanediamide [B6]

This compound is created by reacting six mmol of compound (B3) with six mmol of acrylamide. After purification, light yellow powder is obtained. The product was obtained at a yield of 80 % and a melting point of 202-205°C. M/z [M+] = 872 $C_{55}H_{73}N_3O_6$. FT-IR Spectrum: ν (cm^{-1}) [12-15]: 1710 (C=O) amide and ketone, 3421 (–NH) amide, 1660 (C=C) aliphatic, 3230 (C-H) aromatic. δ ^{1}H-NMR (DMSO-d6 /ppm) [16-20]: 1.48-1.90 (m, 55H, −(CH)−), 2.94 - 2.98 (t, 3H, $-CH_3$), 3.73 -3.79 (t, 2H, $-CH_2$), 5.77-5.97 (dd, 4H, aromatic ring c), 3.83 (dd, 4H, aromatic ring b), 7.9 (s, 4H, aromatic ring a), 5.77-5.97 (dd, 2H, O=C-CH=CH & O=C-CH=CH), 10 (s, 1H, -NH), 12 (s, 1H, −OH), 13 (s, 1H, -NH).

2.2.8 Synthesis of N1-acryloyl-N12-(4-(3-(4-(3-oxo-3-(4-stearamidophenyl)prop-1-en-1-yl) phenyl) acryloyl) phenyl)dodecanediamide [B7]

Six mmol of component (B4) was combined with six mmol of acrylamide to make this chemical. A pale yellow powder is obtained after purification. The product was obtained at a yield of 76 % and a melting point of 198-200°C. M/z [M+] = 900 $C_{57}H_{77}N_3O_6$. FT-IR Spectrum: ν (cm^{-1}) [12-15]: 1708 (C=O) amide and ketone, 3421 (–NH) amide, 1660 (C=C) aliphatic, 3260 (C-H) aromatic. δ ^{1}H-NMR (DMSO-d6 /ppm) [16-20]: 1.48-1.90 (m, 55H, −(CH)−), 2.94 - 2.98 (t, 3H, $-CH_3$), 3.73 -3.79 (t, 2H, $-CH_2$), 5.77-5.97 (dd, 4H, aromatic ring c), 3.83 (dd, 4H, aromatic ring b), 7.9 (s, 4H, aromatic ring a), 5.77-5.97 (dd, 2H, O=C-CH=CH & O=C-CH=CH), 10 (s, 1H, -NH), 12 (s, 1H, −OH), 13 (s, 1H, -NH).

3. Results and Discussion

The compounds (B1-B7) were prepared by the method mentioned in section (2.2), and they were examined by FTIR spectroscopy. The reaction was followed by the appearance of the absorption bands in the range of (1708–1724) cm^{-1} due to the presence of (C=O) stretching. The appearance of peaks in the range (2850–2920) cm^{-1} is attributed to (C-H) aliphatic stretching, and absorption bands in the range (1670–1624) cm^{-1} is assigned to (C=C) aliphatic stretching. While the absorption band of aromatic (C-H) stretching appears within the range of (3113–3260) cm^{-1}, and the band within the range of (1427–1630) cm^{-1} is assigned to (C=C) aromatic stretching. The absorption bands within the range of (3217–3444) cm^{-1} due to the (-NH) stretching of the amide group and the bending (-NH) are shown within the range of (1543–1566) cm^{-1}. FTIR spectra also showed absorption bands at (3329, 3429) cm^{-1} due to symmetric and asymmetric ($–NH_2$) stretching in the (B1) compound. The compounds [B2, B3, and B4) demonstrated the disappearance of stretching bands of (NH_2) that appeared in (3329, 3429) cm^{-1} and the appearance of new bands due to (O-H) stretching at (3421-3444) cm^{-1}.As shown in table (3), (B5, B6, and B7) demonstrated the disappearance of stretching bands for (OH) that appeared in (3421-3444) cm^{-1} and the appearance of new stretching bands (NH, amide, and imide group) at (3421) cm^{-1}as shown in the figures (1-7).

^{1}H-NMR spectra of all the synthesized compounds were measured in deuterated dimethyl sulfoxide (DMSO-d6). All spectra showed peaks in the region of (2.51) ppm, which were due to the DMSO solvent. All compounds (B1-B7) exhibited a singlet signal at (10.00) ppm due to a single proton (2 and 19) for the amide group. In the compounds (B2, B3, and B4), singlet signals at a region of 12.00 ppm were assigned due to one proton (20) for the (OH) group of carboxylic acid. While in compounds, (B5, B6, and B7) showed a singlet signal in the region of 13.00 ppm due to one proton (24) for the (NH) imide group. Because of the mutual attraction of these four protons (9a, 10a, 11a, and 12a), the H-NMR spectral revealed a singlet signal for four protons for aromatic ring (a) in the 7.9 ppm range.

The four protons for aromatic ring (b) in positions (3b, 4b, 5b, and 6b) showed doublet signals within the range (7.57-7.67) ppm due to the mutual attraction between two protons being (3b) with (4b) within the region (7.57-7.59) ppm, as well as the mutual attraction between two protons being (5b) with (6b) within the region (7.65-7.67) ppm. The four protons for aromatic ring (c) in positions (15c, 16c, 17c, and 18c) also showed doublet signals due to mutual attraction between two protons (15c) with (16c) within the range (6.00-6.60) ppm, and mutual attraction between two protons (17c) with (18c) within the range (6.73-6.75) ppm.

^{1}H-NMR spectral showed doublet signals within the range of (5.77-5.97) ppm for the two protons of the double bond are (7 and 8), which is due to the mutual attraction between these two protons. It also showed doublet signals within the range of (5.77–5.80) ppm for the two protons of the double bond (13 and 14), which is due to the mutual attraction between these two protons. The ^{1}H-NMR spectrum of compound (B1) showed a singlet signal within the region of (4.03) ppm due to two protons (1 and 2) for the ($–NH_2$) group.

The aliphatic protons showed multiple signals within the range (1.48-1.90) ppm due to protons (d, e, f, and g). While compounds (B2, B3, and B4) showed triplet signals in the range (3.73–3.79) ppm due to (1d, 2d, 1e, 2e, 9e, 10e, 1f, 2f, 15f, 16f, 1g, 2g, 19g, and 20g) and showed triplet signals in the range (2.94–2.98) ppm due to three protons (29d, 30d, and 31d). In the compounds (B5-B7), the proton (21) showed a triple signal within the range of (5.13-5.19) ppm due to the attraction effect of the proton (21) with two protons (22) and (23). While the two protons (22 and 23) produced a double signal in the (4.47–4.50) ppm range due to the attraction effect of both protons (22 and 23) with proton (21). As shown in the figures (8-14).

From the mass spectra of the synthesized new compounds (B1-B7) from the mass spectra, it was observed that the peak at (m/z = 634, 777, 847, 749, 872, 844, and 802) represented the molecular ions [M+] for (B1, B2, B3, B4, B5, B6, and B7) compounds,

respectively. These peaks indicated that the structures of the synthesized compounds in this study were in agreement with our expectations as shown in the figures (15-21).

Conclusions

The amide moiety is one of the most important functional groups in organic, pharmaceutical, and biological chemistry. Its synthesis has been the focus of many researchers; alternative methods to obtain amides are still needed, and this field is in continuous growth. In this work, we have reacted compound 3, 3'-(1, 4-phenylene) bis(1-(4-aminophenyl) prop-2-en-1-one) which contains two amine groups with mono and di carboxylic acids in the presence of sodium hydroxide under reflux to prepare new four long alkyl amide compounds by using the direct reaction method. Also, in the same method, three new monomers were prepared by using a direct reaction method between new compounds and acrylamide. All synthesized compounds are given a good yield without using a catalyst.

Table (3): FT-IR spectral data of the compounds [B1-B7]

Compound Symbol	(NH)amide stretching (NH)imide stretching	(NH_2) stretching	(C=O) stretching	(CH=CH) ethylene stretching	(CH=CH) aromatic stretching	(CH) aliphatic stretching	(CH) Aromatic stretching	(OH) stretching	(NH) bending	Others
B1	3217	3329, 3429	1712	1640	3217	2850 -2920	1427 – 1597		1554	C-N stretching = 1346 C-O stretching = 1230
B2	3240		1708	1640	3200	2850 -2920	1442 – 1554	3421	1543	C-N stretching = 1430 C-O stretching = 1240 C-OH bending = 1330
B3	3390		1708	1630	3190	2850 -2920	1468 – 1554	3425	1543	C-N stretching = 1419 C-O stretching = 1219 C-OH bending = 1338
B4	3235		1712	1630	3210	2850 -2920	1438 – 1554	3429	1543	C-N stretching = 1423 C-O stretching = 1222 C-OH bending = 1400
B5	3444		1717	1650	3230	2850 -2920	1465 – 1620		1554	C-N stretching = 1419 C-O stretching = 1220
B6	3421		1710	1660	3230	2850 -2920	1465 – 1620		1558	C-N stretching = 1442 C-O stretching = 1240
B7	3421		1708	1660	3260	2850 -2920	1465 – 1630		1558	C-N stretching = 1423 C-O stretching = 1230

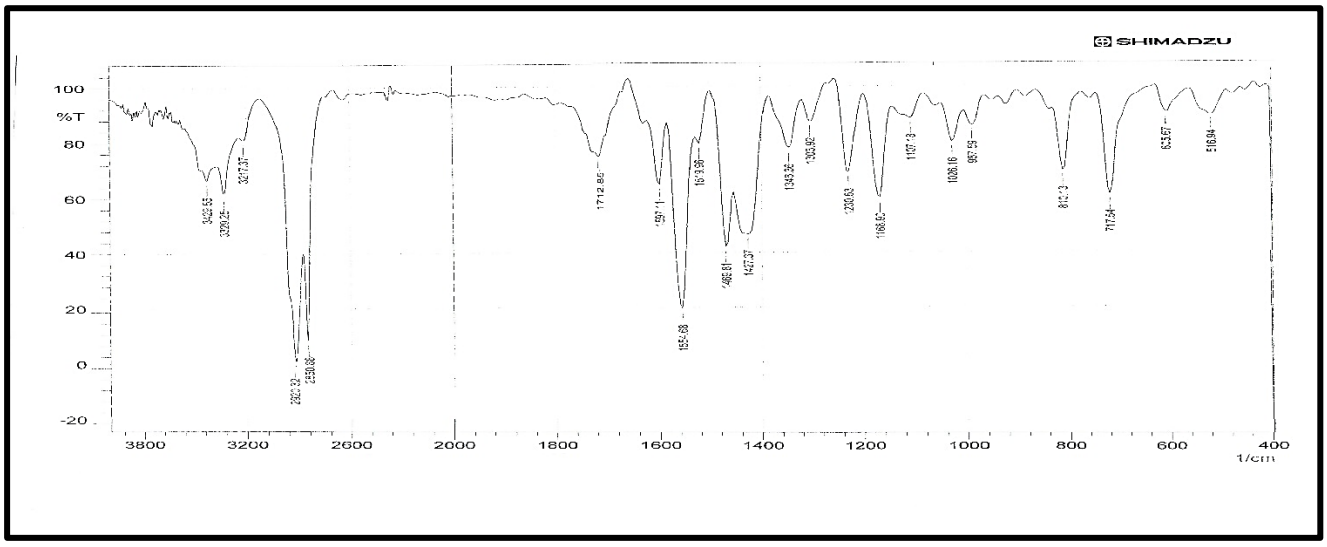


Figure (1): FT-IR for B1

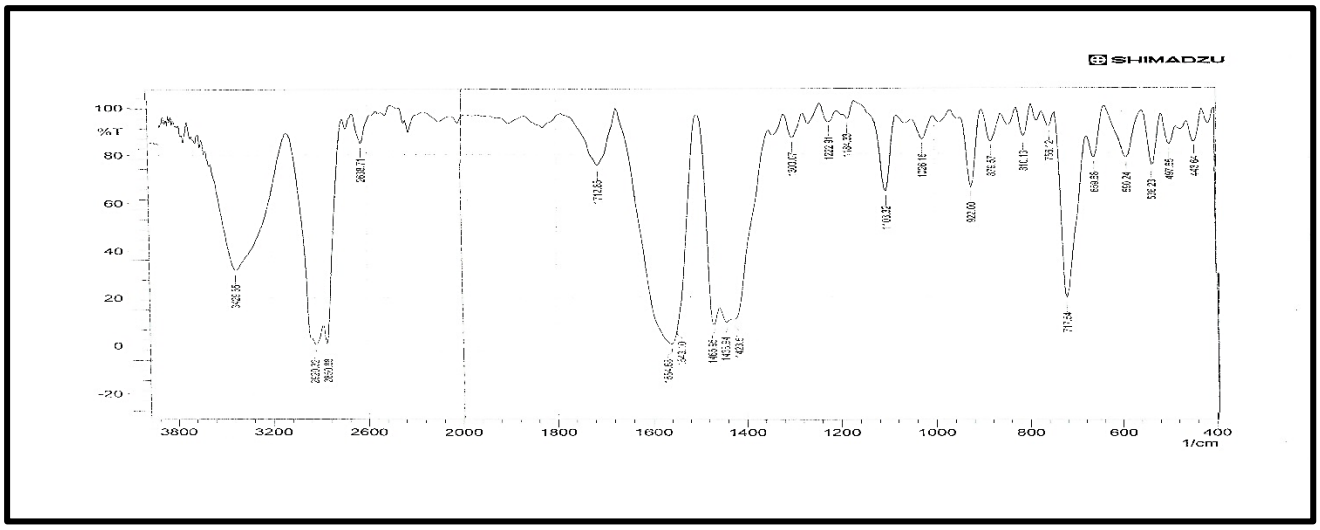


Figure (2): FT-IR for B2

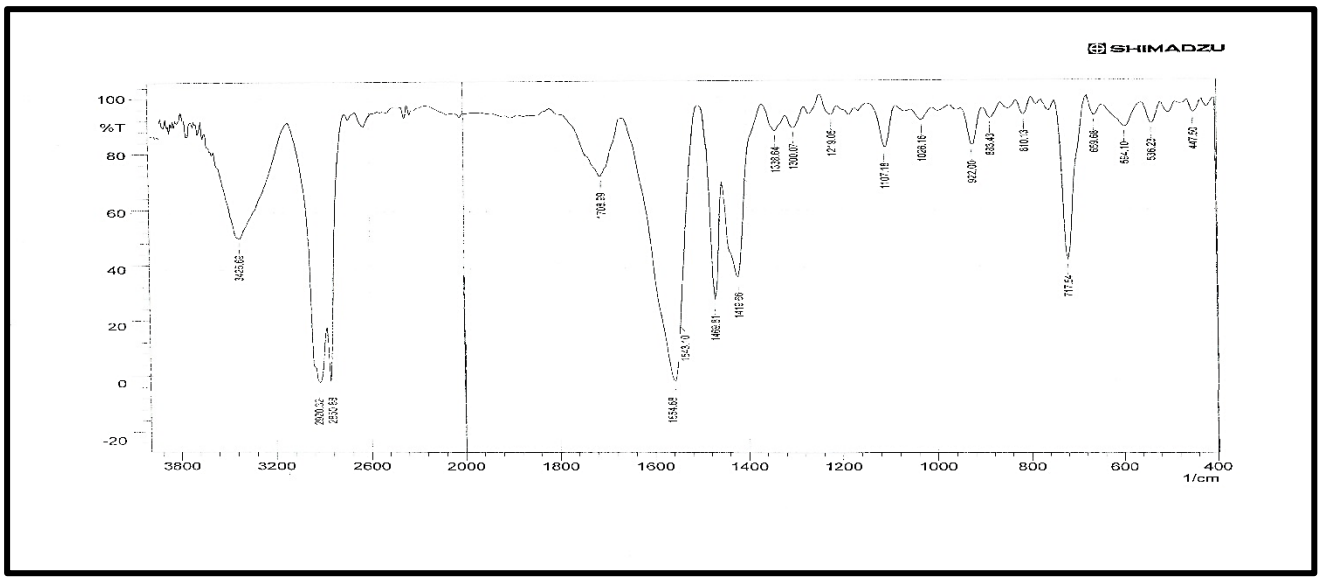


Figure (3): FT-IR for B3

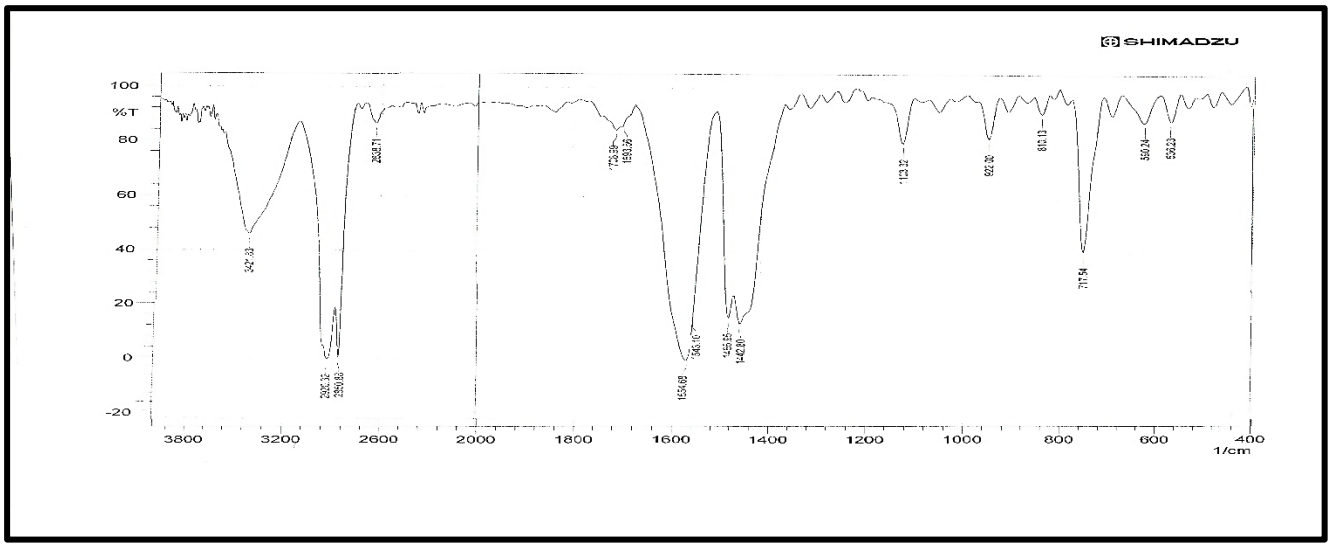


Figure (4): FT-IR for B4

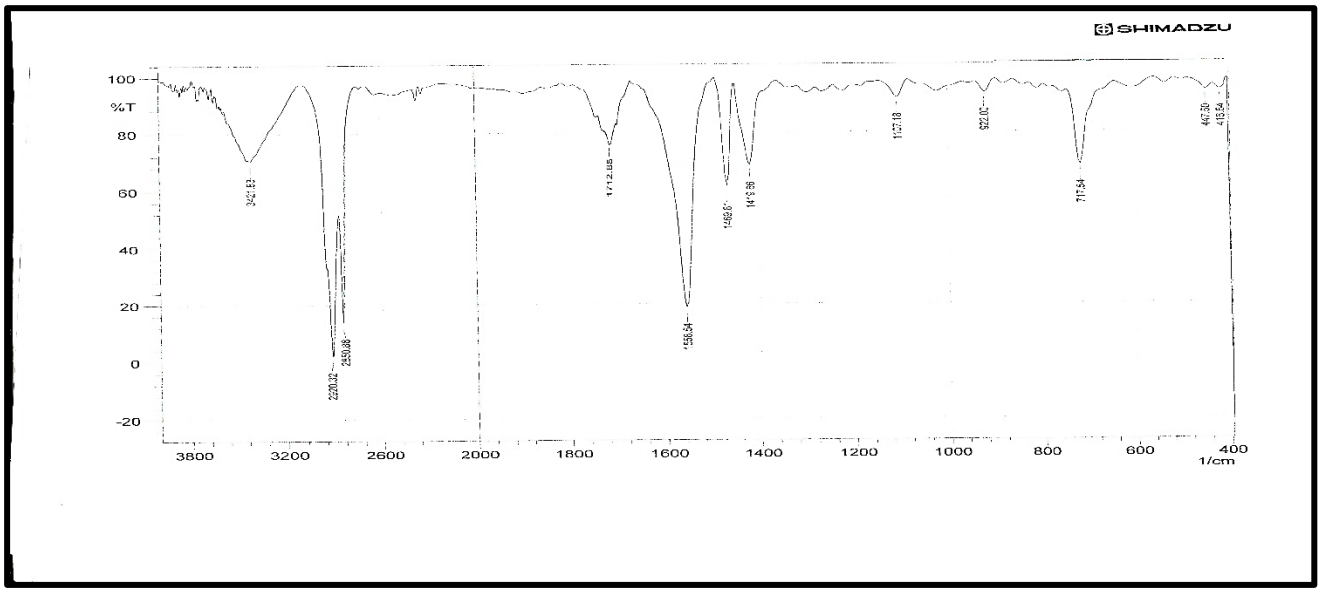


Figure (5): FT-IR for B5

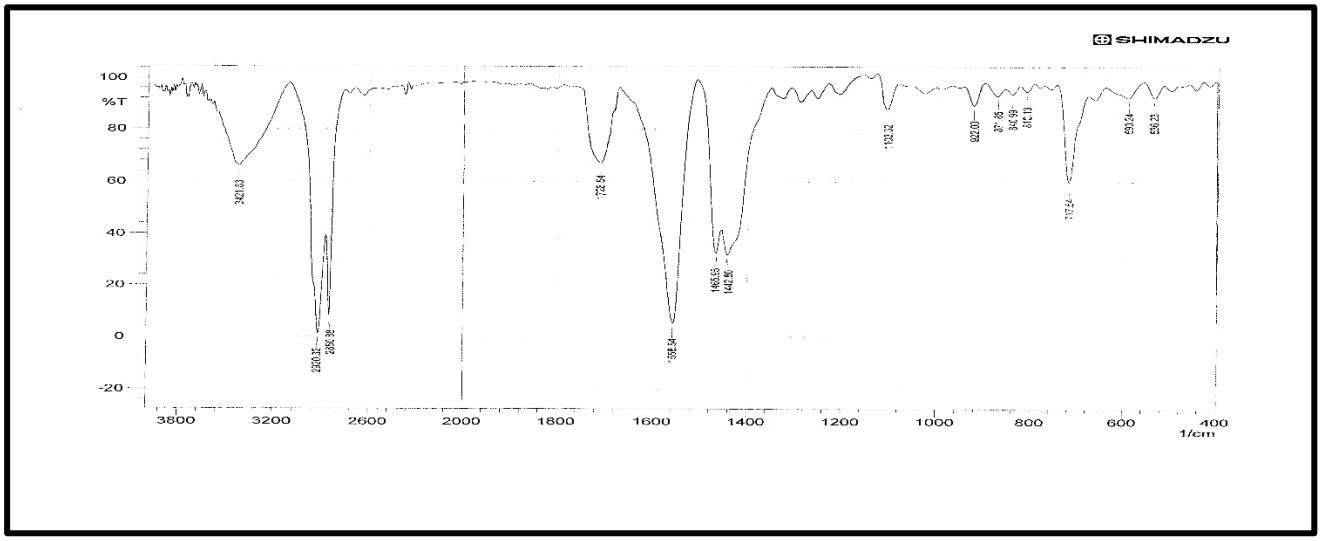


Figure (6): FT-IR for B6

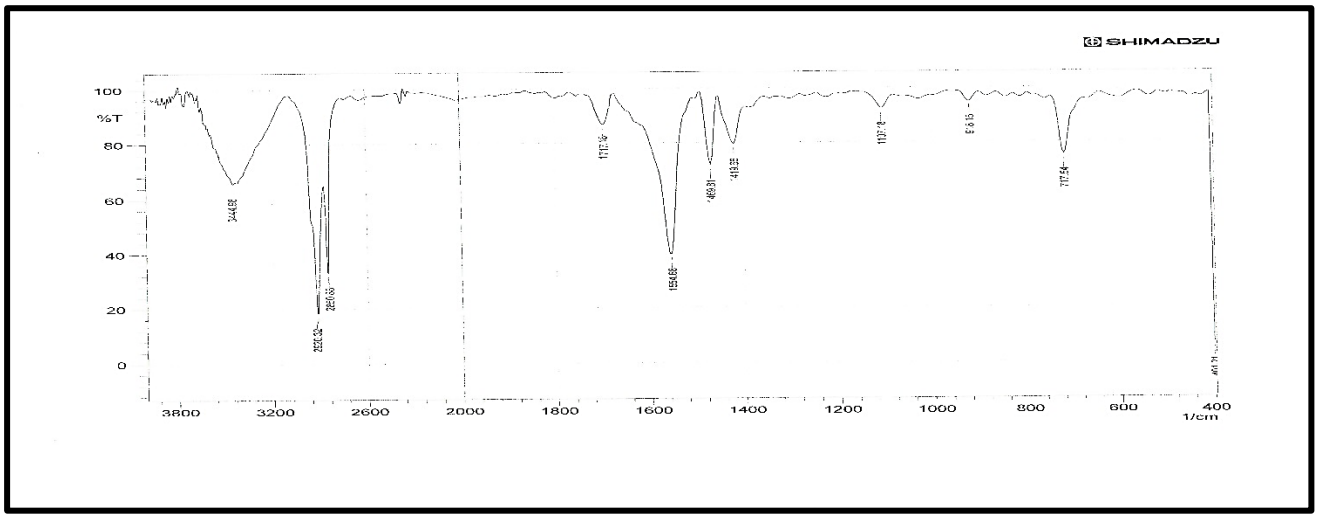
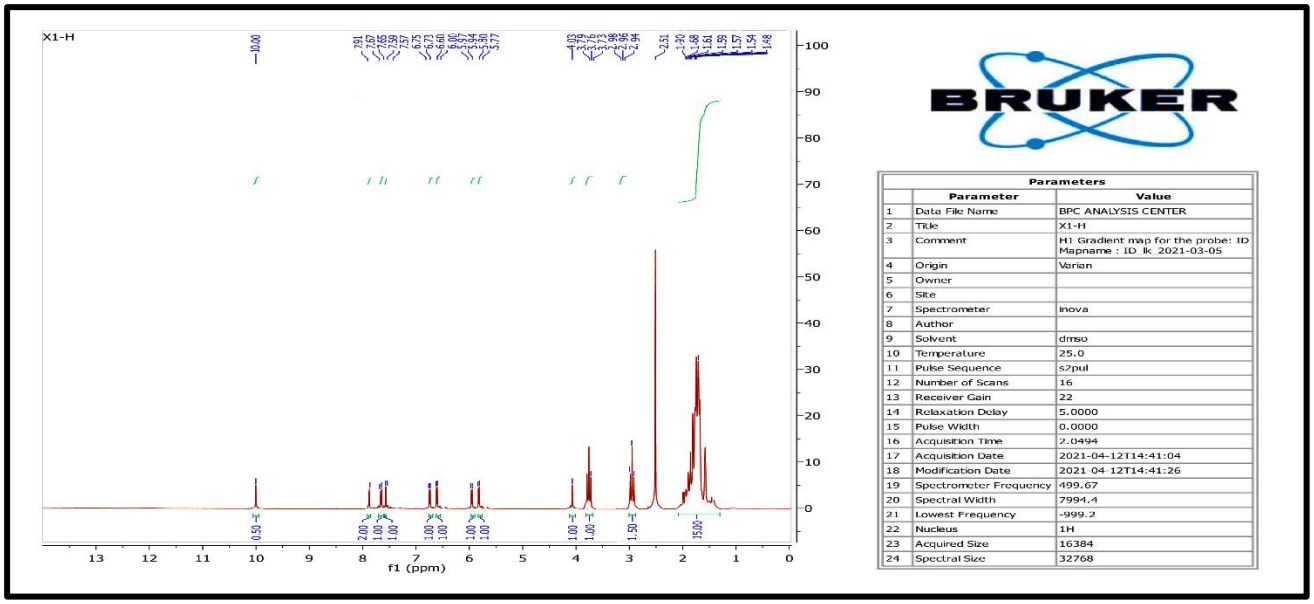


Figure (7): FT-IR for B7



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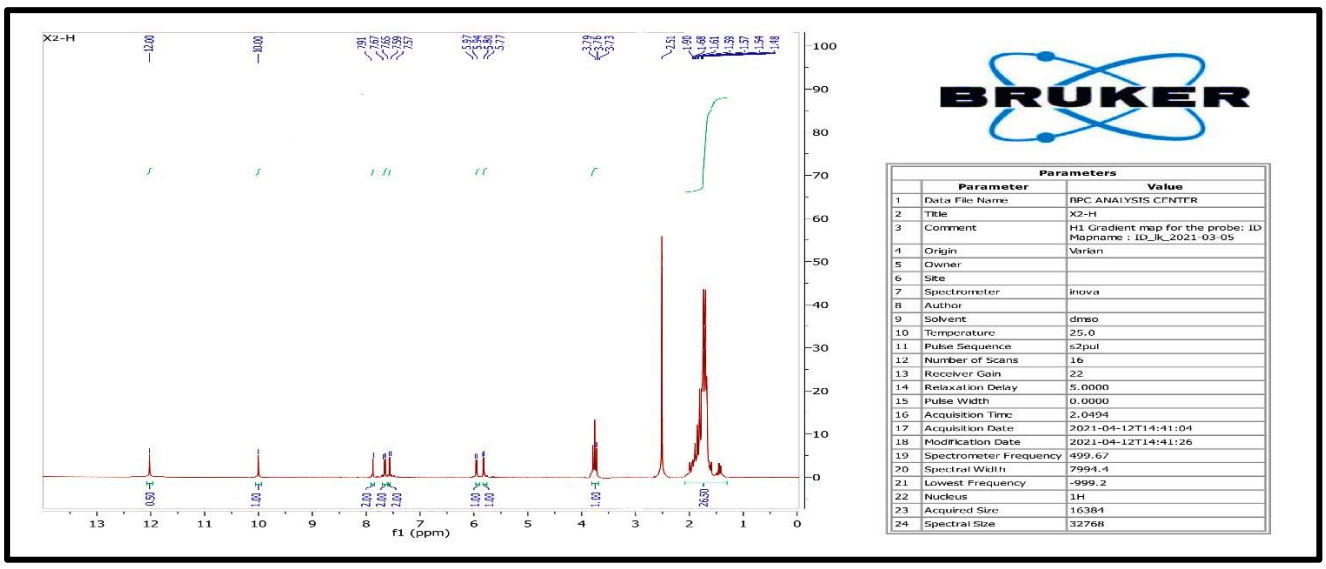
2	Title	X1-H
3	Comment	H1 Gradient map for the probe: ID Mapname : ID_lk_2021-03-05
4	Origin	Varian
5	Owner	
6	Site	
7	Spectrometer	inova
8	Author	
9	Solvent	dmso
10	Temperature	25.0
11	Pulse Sequence	s2pul
12	Number of Scans	16
13	Receiver Gain	22
14	Relaxation Delay	5.0000
15	Pulse Width	0.0000
16	Acquisition Time	2.0494
17	Acquisition Date	2021-04-12T14:41:04
18	Modification Date	2021-04-12T14:41:26
19	Spectrometer Frequency	499.67
20	Spectral Width	7994.4
21	Lowest Frequency	-999.2
22	Nucleus	1H
23	Acquired Size	16384
24	Spectral Size	32768

Figure (8): H-NMR for B1



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5	Owner	
6	Site	
7	Spectrometer	inova
8	Author	
9	Solvent	dmso
10	Temperature	25.0
11	Pulse Sequence	s2pul
12	Number of Scans	16
13	Receiver Gain	22
14	Relaxation Delay	5.0000
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17	Acquisition Date	2021-04-12T14:41:04
18	Modification Date	2021-04-12T14:41:26
19	Spectrometer Frequency	499.67
20	Spectral Width	7994.4
21	Lowest Frequency	-999.2
22	Nucleus	1H
23	Acquired Size	16384
24	Spectral Size	32768

Figure (9): H-NMR for B2

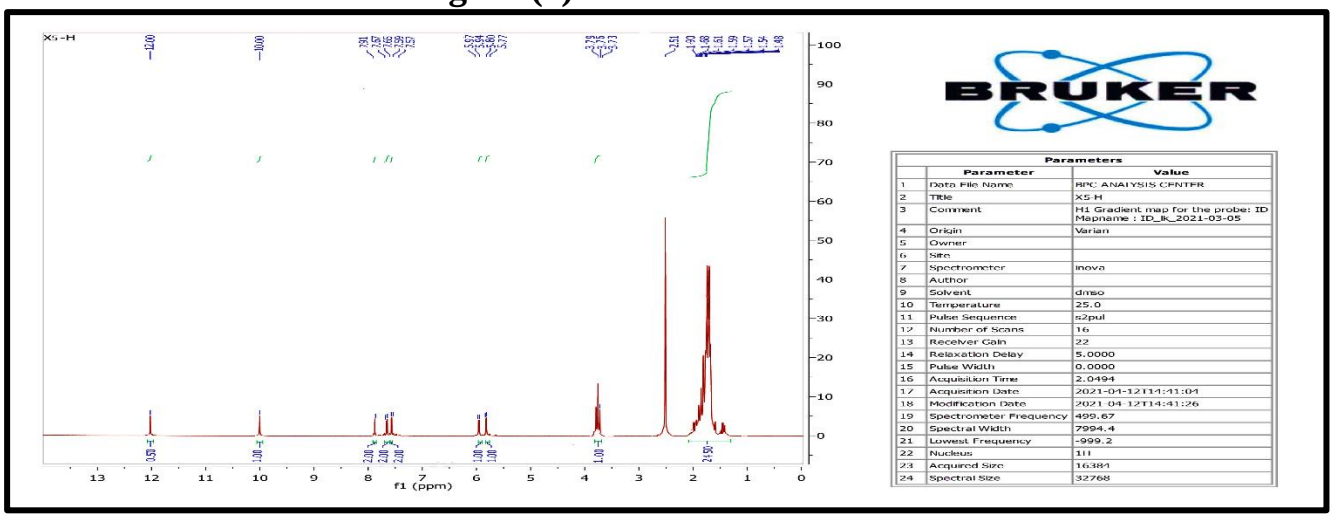

Figure (10): H-NMR for B3

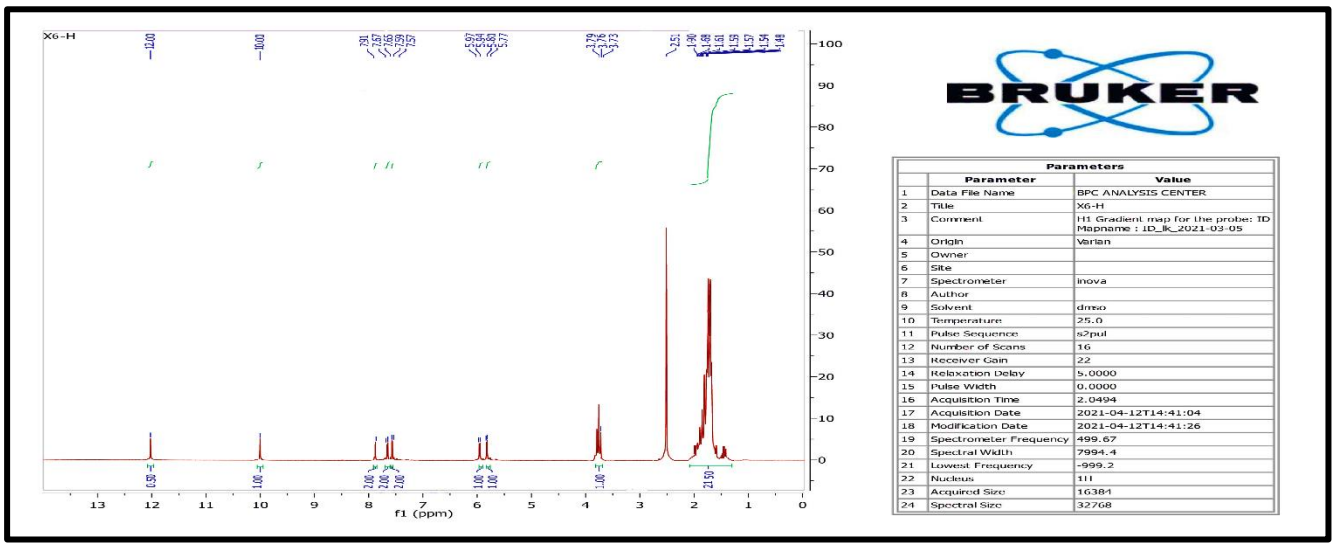

Figure (11): H-NMR for B4

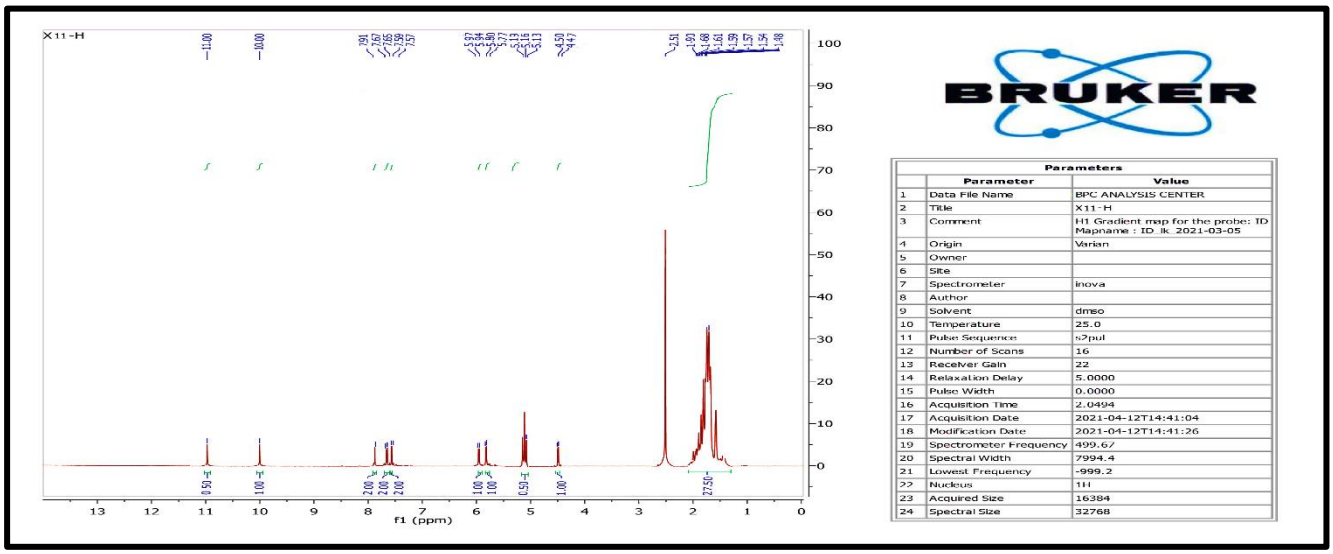

Figure (12): H-NMR for B5

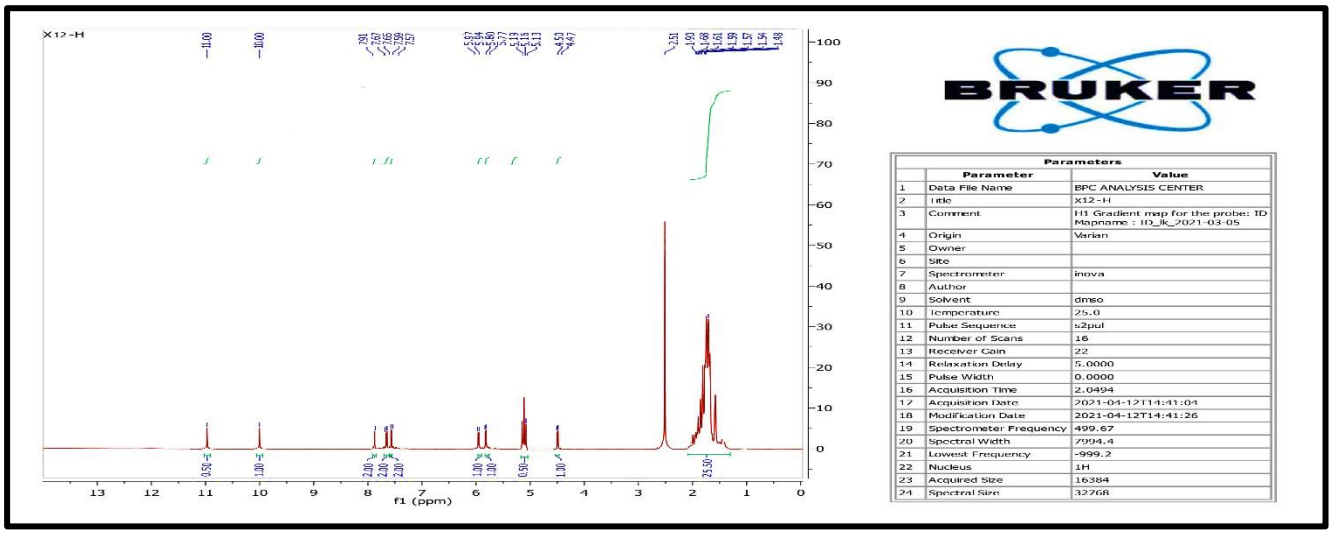

Figure (13): H-NMR for B6

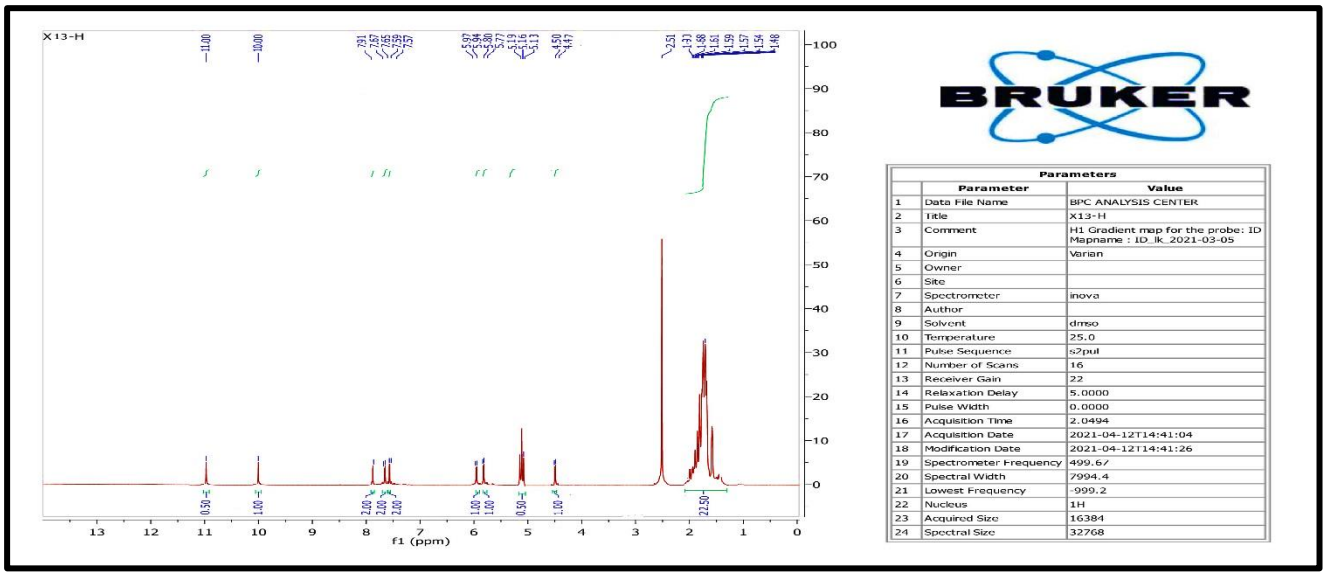

Figure (14): H-NMR for B7

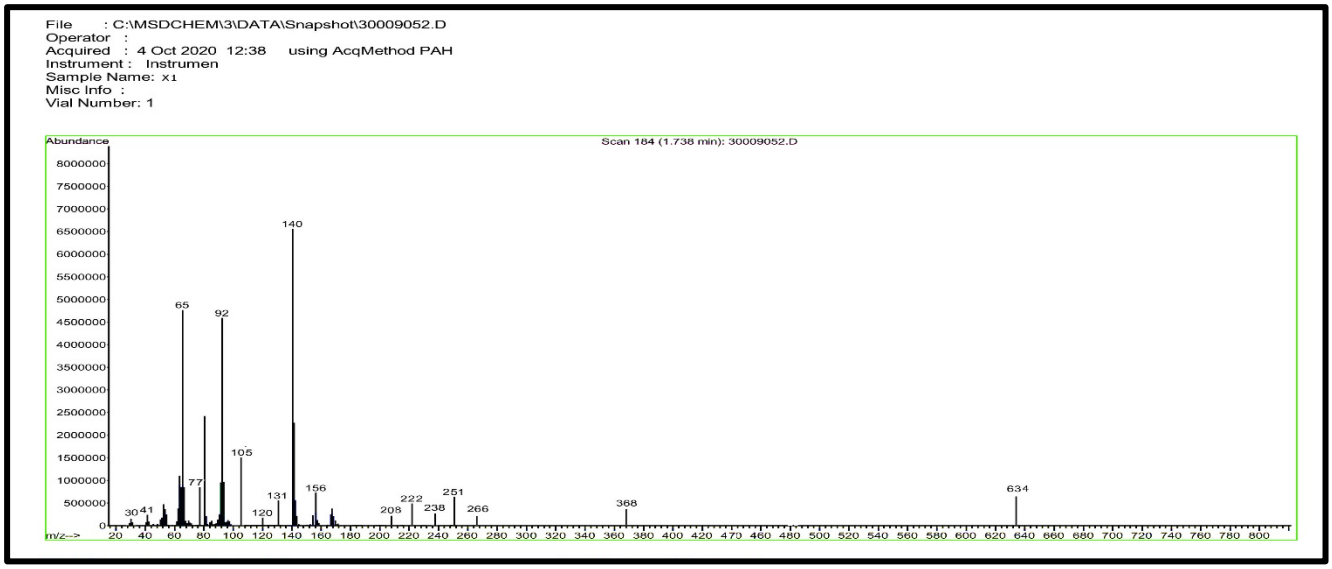

Figure (15): Mass spectral for B1

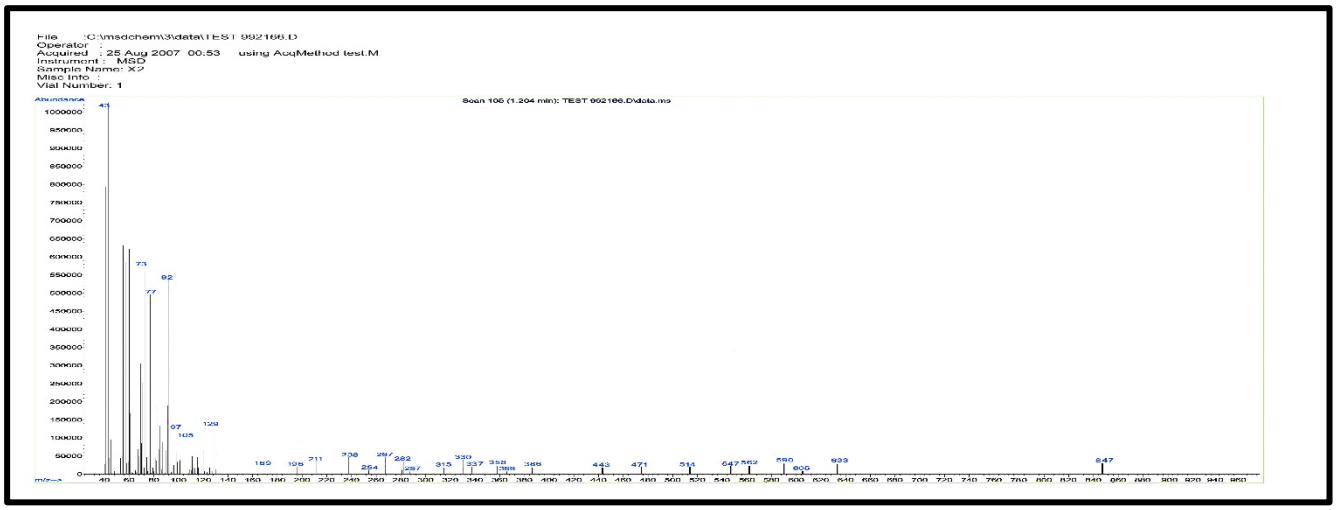

Figure (16): Mass spectral for B2

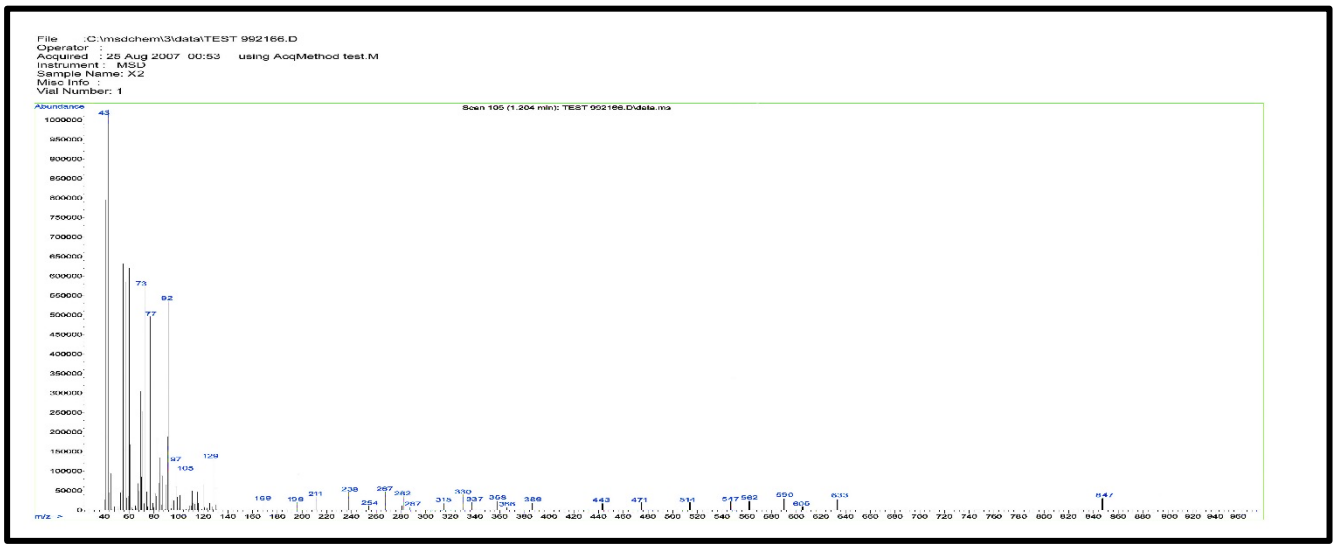

Figure (17): Mass spectral for B3

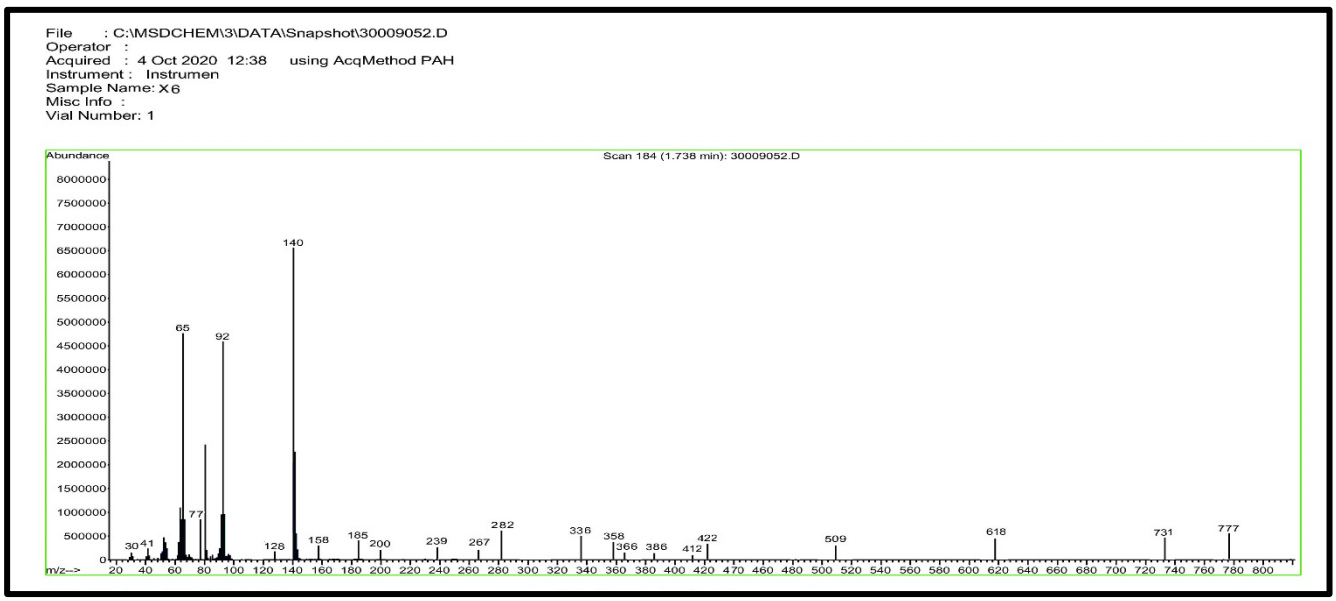

Figure (18): Mass spectral for B4

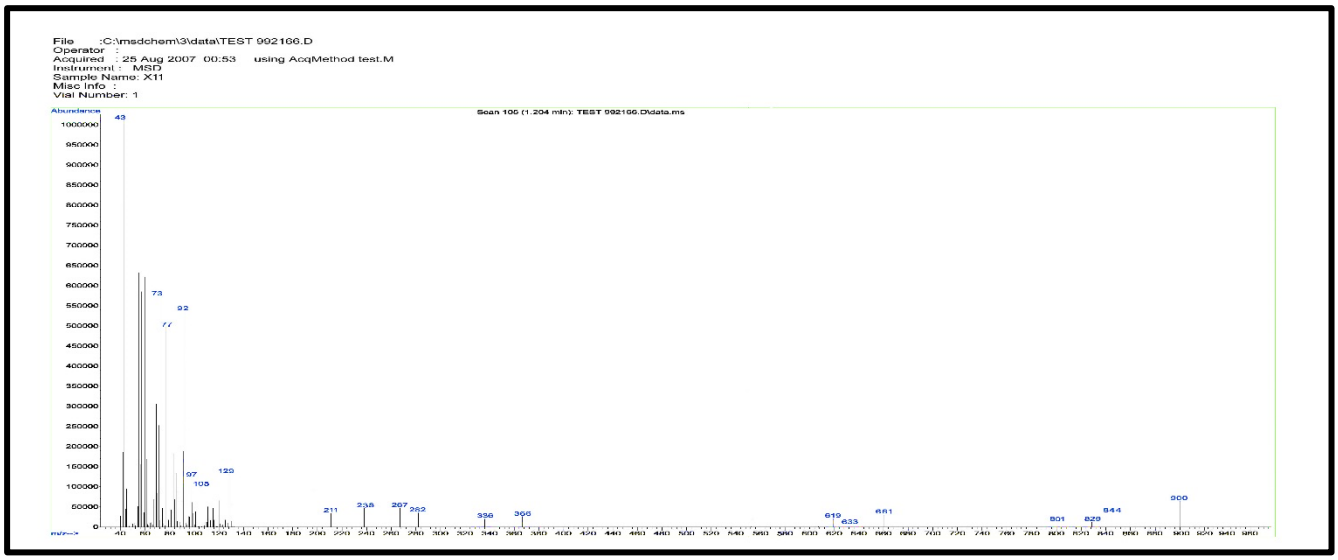

Figure (19): Mass spectral for B5

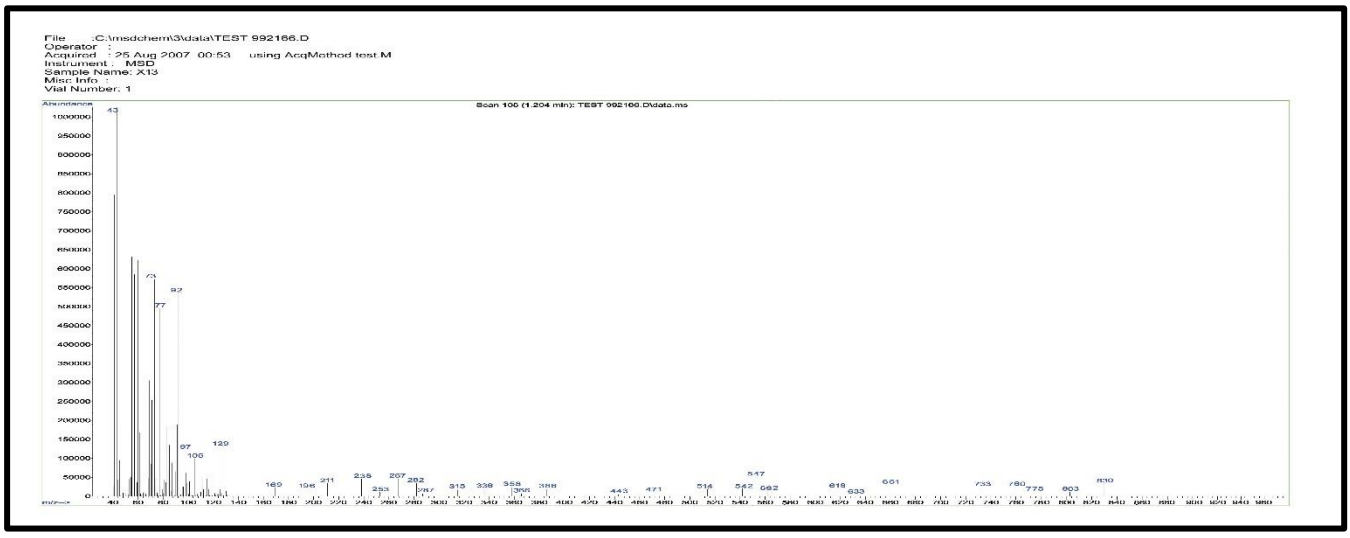

Figure (20): Mass spectral for B6

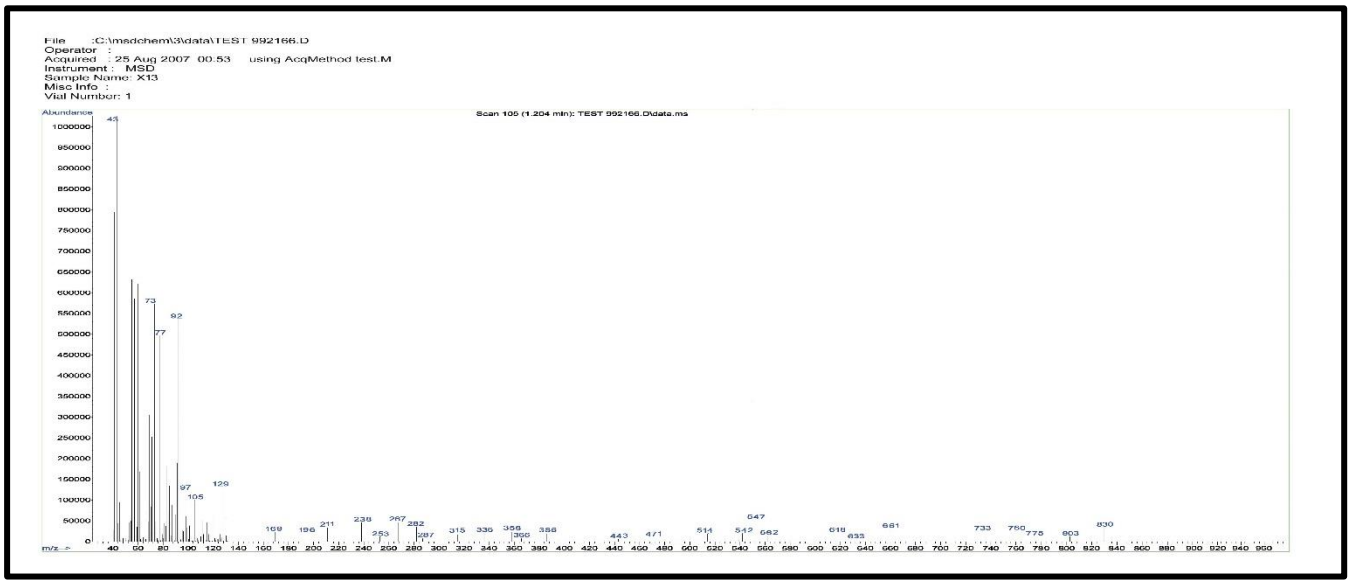

Figure (21): Mass spectral for B7

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