SYNTHESIS OF 1,2,3-TRIAZOLES BASED ON PHENACYL AZIDE DERIVATIVES *VIA* CLICK CHEMISTRY

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Abstract

Etherification of *n*-hexanol, *n*-heptanol and *n*-octanol with propargyl bromide in the presence of sodium hydroxide in DMF afforded the terminal alkynes (2) a, b and c. Phenacyl bromide, *p*-bromophenacyl bromide and *p*-phenylphenacyl bromide were converted to corresponding azides (4) a, b and c respectively by traditional S_N2 reaction of the mentioned bromides and sodium azide in DMF. The cycloaddition of the propargyl ethers (2) with the prepared organic azides (4) using click conditions gave the target 1,4-disubstituted 1,2,3- triazoles (5)-(7) in good yields. All the synthesized triazoles were characterized by FT-IR while the compounds (5) a,b and c were characterized by ^{1}H NMR and ^{13}C NMR in addition to FT-IR technique.

Key Words: 1,2,3-triazoles, click chemistry, phenacyl azide

تحضير ١,٢,٣-ترايزولات ابتداءً من مشتقات أزيد الفيناسيل بطريقة كيمياء النقرة

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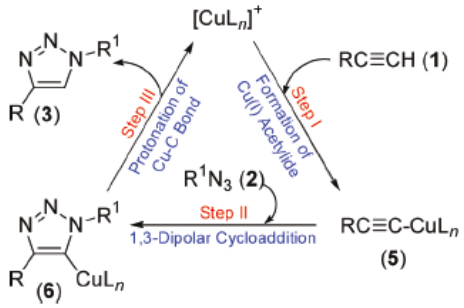
الخلاصة

تضمن البحث تفاعل تكوين الأيثر ل ن-هكسانول، ن-هبتانول و ن-أوكتانول مع بروميد البروبرجيل بوجود هيدروكسيد الصوديوم في ثنائي مثيل فورمأميد أعطى البروبارجيل أيثر (٢) أ، ب، ج. تم تحويل بروميد الفيناسيل، بارا- برومو بروميد الفيناسيل و بارا- فينيل بروميد الفيناسيل الى الأزيدات المقابلة (٤) أ، ب، ج وذلك من خلال تفاعل التعويض ثنائي الجزيئة لمركبات البروميد المذكورة مع أزيد الصوديوم في ثنائي مثيل فورمأميد. الأضافة الحلقية للألكاينات الطرفية (٢) مع الأزيدات العضوية المحضرة (٤) بأستخدام ظروف النقرة تعطي المركبات المنشودة ٤,١-ثنائية التعويض ٣.٢,١-ترايزول (٥-٧) بمنتوج جيد. تم تشخيص جميع الترايزولات المخلقة بواسطة طيف الأشعة تحت الحمراء. بينما المركبات (٥) أ، ب، ج تم تشخيصها بواسطة طيف الرنين النووي المغناطيسي البروتوني والكاربوني بالأضافة الى تقنية طيف الأشعة تحت الحمراء.

الكلمات المفتاحية:-أزيد الفيناسيل، ترايازولات

Introduction

The essential science upon which these advances are based is the synthesis of designed heterocyclic compounds which make it possible to probe sensitively the key events in biology in partnership with structural biology [1]. The original Huisgen 1,3-dipolar cycloaddition between alkyne and azide usually requires higher temperatures and provides a mixture of 1,4- and 1,5-disubstituted 1,2,3- triazoles (Scheme 1) [2].



Scheme 1: 1,3-dipolar cycloaddition of organic azides to alkynes

Because of the vicinal nitrogen atoms cyclic arrangement, no natural heterocyclic compounds like 1,2,3 triazoles have been isolated. However, this type of structure displays wide spread use. It has been considered as an interesting component in terms of biological activity [3]. New N-alkylaminocyclitols bearing a 1,2,3-triazole system at different positions of the alkyl chain have been prepared as potential G Case pharmacological chaperones using click chemistry approaches [4]. 1-Nonyl-4-[(6-deoxy-1,2:3,4-Di-O-isopropylidene-α-D-galactos-6-yl)oxymethyl]1H-1,2,3 triazole was prepared via click chemistry starting from D-galactose [5]. A number of synthesized sugar triazoles were evaluated for their antitubercular activity against Mycobacterium tuberculosis H37Rv, where one of the compounds displayed mild antitubercular activity in vitro with MIC 12.5 µg/mL [6]. Chiral 1,4-disubstituted-1,2,3-triazoles derivatives have the potential of mimicking the binding mode of known purine analogues have been synthesized [7]. New fluorous-tagged azabis(oxazoline) ligands were prepared using the copper-catalyzed azide-alkyne cycloaddition as ligation method [8]. The synthesis of two novel glycosyl-nucleoside fluorinated amphiphiles (GNFs) derived from the 2H,2H,3H,3H-perfluoro-undecanoyl hydrophobic chain were prepared using a 'double click' chemistry[9].

Experimental Part

Chemicals and Instruments

Chemical reagents and starting materials were obtained from Ajax and Sigma-Aldrich. Infrared spectra were recorded using AVATAR 320 FT-IR. ^{1}H and ^{13}C NMR spectra were recorded using 300 MHz Bruker DPX spectrometers. Silica TLC plates were used with an aluminum backing (0.2 mm, 60 F_{254}). The reactions were monitored by TLC and visualized by development of the TLC plates with an alkaline potassium permanganate dip.

Synthesis of *n*-alkyl propargyl ethers (2) [10]

Alcohol (2.0 mmol) was dissolved in DMF (10 mL) and powdered NaOH pellets (0.32 g, 8.0 mmol) were added. The contents were stirred in a salt-ice bath for 10 min then propargyl bromide (0.25 mL, 2.82 mmol) was added dropwise. The reaction mixture allowed to stir for 24 h, gradually warming to r.t. The reaction mixture was partitioned between Et_2O (30 mL) and water (50 mL) and the aqueous layer extracted with more Et_2O (3 x 30 mL). The combined organic extracts were dried over Na_2SO_4, and evaporated to dryness under reduced pressure. The residue was chromatographed (silica gel, Et_2O: light petroleum 1:7).

Synthesis of phenacyl azide derivatives (4)

To a suspension of sodium azide (2.0 g, 30 mmol) in DMF (30 mL), α-bromoketone (3) (10.0 mmol) was added and heated to 40°C for 1h. After completion of reaction, monitored by TLC (n-hexane:ethyl acetate; ratio = 5:1), the mixture was diluted with water (60 mL) and extracted with EtOAc (3 x 50 mL). The combined organic layer washed with saturated NaCl solution (50 mL), cold water (3 x 50 mL), dried over anhydrous Na_2SO_4 and filtered. Evaporation of solvent afforded the desired azides.

Synthesis of 1,2,3-triazoles (5-7)

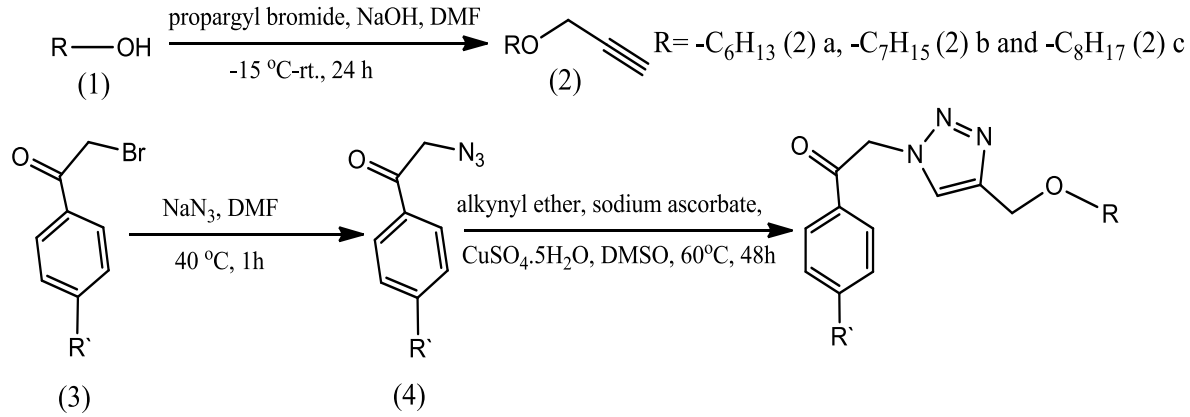
Propargyl ether (alkyne) (1 mmol) and α-azidoketone (4) (1 mmol) were added to a suspension of sodium ascorbate (0.0198 g, 0.10 mmol) and $CuSO_4 \cdot 5H_2O$ (0.0125 g, 0.05 mmol) in DMSO (5 mL). The mixture was heated to 60°C and stirred for 48 h. The reaction mixture was diluted with water (30 mL), extracted with EtOAc (3×25 mL). The combined organic layers were washed with brine (2×20 mL), dried over Na_2SO_4, and evaporated to dryness under reduced pressure. The residue was

chromatographed (silica gel, EtOAc/n-hexane 1:6 – 1:2) and the main fraction recrystallized from light petroleum (40-60^{o}C) gave appropriate triazoles.

Results and Discussion

Straight chain alcohols were etherified with propargyl bromide in the presence of NaOH in DMF and gave the n-alkyl propargyl ethers (2) in moderate to good yields. (Table 1) showed the physical properties; (table 2) the summery of FT-IR bands in cm^{-1}.

Phenacyl bromide derivatives (3) were converted to the corresponding phenacyl azide derivatives (4) by S_N2 reaction with sodium azide in DMF, the overall work steps shown in scheme below:



R`=H (3 and 4) a, Br (3 and 4) b and ph (3 and 4)c.

compound	R	R`
5 a	-C_6H_{13}	H
5 b	-C_7H_{15}	H
5 c	-C_8H_{17}	H
6 a	-C_6H_{13}	Br
6 b	-C_7H_{15}	Br
6 c	-C_8H_{17}	Br
7a	-C_6H_{13}	ph
7 b	-C_7H_{15}	ph
7c	-C_8H_{17}	ph

Scheme 2: synthetic route of phenacyl triazoles

FT-IR spectrum of *n*-hexyl propargyl ether **(2)a** showed the following bands cm^{-1}(neat): 3311 (**C-H** acetylenic) stretching, 2927, 2858 **(C-H** aliphatic**)** stretching, 2115 (**C≡C**) stretching, 1461, 1382 **(C-H)** bending, 1101-1056 (**C-O**) stretching. The bands at 3311 and 2115 are very good evidences of formation of the alkyne. FT-IR spectrum of *n*-heptyl propargyl ether **(2)b** (figure 1) showed the following bands cm^{-1}(neat): 3311 (**C-H** acetylenic) stretching, 2927, 2856 **(C-H** aliphatic**)** stretching, 2116 (**C≡C**) stretching, 1466, 1356 **(C-H)** bending, 1265-1104 (**C-O**) stretching. Again the bands at 3311 and 2116 are very good proofs of formation of the terminal alkyne. FT-IR spectrum of *n*-octyl propargyl ether **(2)c** showed the following bands cm^{-1}(neat): 3311 (**C-H** acetylenic) stretching 2927, 2856 **(C-H** aliphatic**)** stretching, 2117 (**C≡C**) stretching, 1466, 1356 **(C-H)** bending, 1265-1104 (**C-O**) stretching. Once more the same scenario with compound **(2)c**.

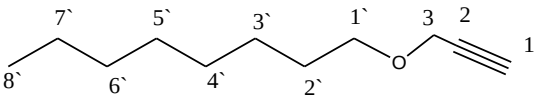


Figure 2: Numbering of carbon atoms in compound (2)c

^{1}H NMR (300 MHz, $CDCl_3$) for **(2)c** figure (3) δ ppm: 0.87 (t, *J* 6.7 Hz, 3H, H8`), 1.31 (m, 10H, H3`-H7`), 1.60 (m, 2H, H2`), 2.40 (t, *J* 2.4

Hz, 1H, H1), 3.49 (t, *J* 6.6 Hz, 2H, H1`), 4.12 (d, *J* 2.4 Hz, 1H, H3).

S_N2 reaction of the phenacyl bromide derivatives **(3)** with sodium azide in DMF afforded phenacyl azide derivatives **(4)** in very good yield. FT-IR spectrum of phnacyl azide **(4)a** (figure 4) showed the following bands cm^{-1}(KBr): 3060 (**C-H** aromatic) stretching 2927, 2862 **(C-H** aliphatic**)** stretching, 2102 (**-N_3**) stretching, 1668 **(C=O)** stretching, 1600, 1490 **(C=C** aromatic**)** stretching, 966-698 **(C-H** aromatic**)** bending oop. FT-IR spectrum of 4-bromophnacyl azide **(4)b** showed the following bands cm^{-1}(KBr): 3090 (**C-H** aromatic) stretching 2925, 2856 **(C-H** aliphatic**)** stretching, 2102 (**-N_3**) stretching, 1660 **(C=O)** stretching, 1595, 1519 **(C=C** aromatic**)** stretching, 970-705 **(C-H** aromatic**)** bending oop. FT-IR spectrum of 4-phenylphenacyl azide **(4)c** showed the following bands cm^{-1}(KBr): 3091 (**C-H** aromatic) stretching 2927, 2868 **(C-H** aliphatic**)** stretching, 2106 (**-N_3**) stretching, 1668 **(C=O)** stretching, 1587 **(C=C** aromatic**)** stretching, 985-661 **(C-H** aromatic**)** bending oop.

Copper (I) catalyzed 1,3-dipolar cycloaddition (click conditions) of terminal alkynes **(2)** with phenacyl azide derivatives gave the targeted 1,2,3-triazoles in good yields. FT-IR spectrum of 2-(4-((hexyloxy)methyl)-1H-1,2,3-triazol-1-yl)-1-phenylethanone **(5)a** showed the following bands cm^{-1}(KBr): 3095 (**C-H** aromatic) stretching 2929, 2862 **(C-H** aliphatic**)** stretching, 1672 **(C=O)** stretching, 1604, 1442 **(C=C** aromatic**)** stretching, 1182, 1093 (**C-O**) stretching 910-661 **(C-H** aromatic**)** bending oop, the disappearance of the azide band at 2102 cm^{-1} and the terminal alkyne bands at 3311 and 2115 cm^{-1} is a very good evidence for the formation of compound **(5)a**.

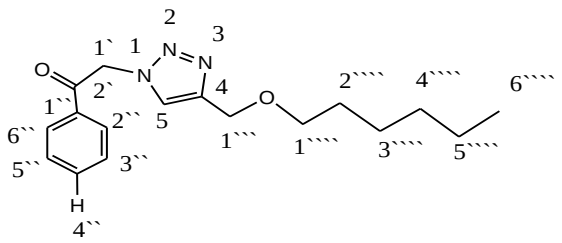


Figure 5: Numbering of compound (5)a

^{1}H NMR (300 MHz, $CDCl_3$) (figure 6) δ ppm: 0.88 (t, *J* 6.6 Hz, 3H, H6````), 1.32 (m, 6H, H3````-H5````), 1.50 (m, 2H, H2````), 3.58 (t, *J* 6.6 Hz, 2H, H1````), 4.46 (s, 4H, H1`, H1```). 7.46-8.00 (m, 6H, H5, H2``-H6``). The presence of the aliphatic signals and the integration of aromatic region to six protons are excellent proofs for the formation of compound **(5)a.** ^{13}C NMR (75 MHz, $CDCl_3$) (figure 7) δ ppm: 14.0-32.8 (6C, C1````-C6````), 62.9 (2C, C1`, C1```), 122.5 (C5), 128.9-134.1 (6C, C1``-C6``), 144.4 (C4) and 191.4 (C2`). The appearance of aromatic and aliphatic signals and fitting them to the structure is excellent evidence for the formation of **(5)a.**

FT-IR spectrum of 2-(4-((heptyloxy)methyl)-1H-1,2,3-triazol-1-yl)-1-phenylethanone **(5)b** (figure 8) showed the following bands cm^{-1}(KBr): 3100 (**C-H** aromatic) stretching, 2923, 2854 **(C-H** aliphatic**)** stretching, 1679 **(C=O)** stretching, 1581, 1442 **(C=C** aromatic**)** stretching, 1170, 1066 (**C-O**) stretching 929-740 **(C-H** aromatic**)** bending oop, the disappearance of the azide band at 2102 cm^{-1} and the terminal alkyne bands at 3311 and 2116 cm^{-1} is a very good evidence for the formation of compound **(5)b**. The same system of numbering of compound **(5)a** was used to number compound **(5)b** then utilized it in the explanation of NMR spectra. ^{1}H NMR (300 MHz, $CDCl_3$) (figure 6) δ ppm: 0.86 (t, *J* 6.7 Hz, 3H, H7````), 1.27 (m, 8H, H3````-H6````), 1.53 (m, 2H, H2````), 3.59 (t, *J* 6.7 Hz, 2H, H1````), 4.46 (s, 4H, H1`, H1```). 7.46-8.00 (m, 6H, H5, H2``-H6``). The presence of the aliphatic signals and the integration of aromatic region to six protons are excellent proofs for the formation of compound **(5)b.** ^{13}C NMR (75 MHz, $CDCl_3$) (figure 7) δ ppm: 14.1-32.6 (7C, C1````-C7````), 63.0 (2C, C1`, C1```), 122.6 (C5), 128.9-134.1 (6C, C1``-C6``), 144.4 (C4) and 191.4 (C2`). The appearance of aromatic and aliphatic signals and fitting them to the structure is excellent evidence for the formation of **(5)b.**

FT-IR spectrum of 2-(4-((octyloxy)methyl)-1H-1,2,3-triazol-1-yl)-1-phenylethanone **(5)c** showed the following bands cm^{-1}(KBr): 3331 (moisture), 2925, 2856 **(C-H** aliphatic**)** stretching, 1676**(C=O)** stretching, 1604 **(C=C** aromatic**)** stretching, 1184, 1029 (**C-O)** stretching 952-698 **(C-H** aromatic**)** bending oop, the disappearance of the azide band at 2102 cm^{-1} and the terminal alkyne bands at 3311 and 2117 cm^{-1} is a very good evidence for the formation of compound **(5)c**. ^{1}H NMR (300 MHz, $CDCl_3$) (figure 9) δ ppm: 0.86 (t, *J* 6.6 Hz, 3H, H8````), 1.28 (m, 10H, H3````-H7````), 1.53 (m, 2H, H2````), 3.59 (t, *J* 6.6 Hz, 2H, H1````), 4.46 (s, 4H, H1`, H1```). 7.47-8.00(m,

6H, H5,H2``-H6``). ^{13}C NMR (75 MHz, $CDCl_3$) figure (9) δ ppm: 14.2-32.9 (8C, C1````-C8````), 63.0 (2C, C1`, C1```), 122.6 (C5), 129.0-134.1 (6C, C1``-C6``), 144.4 (C4) and 191.4(C2`).

FT-IR spectrum of 1-(4-bromophenyl)-2-(4-((hexyloxy)methyl)-1H-1,2,3-triazol-1-yl)ethanone **(6)a** showed the following bands cm^{-1}(KBr): 3095 (**C-H** aromatic) stretching , 2927, 2862 **(C-H** aliphatic**)** stretching, 1668**(C=O)** stretching, 1602, 1452 **(C=C** aromatic**)** stretching, 1045 (**C-O**) stretching 925-698 **(C-H** aromatic**)** bending oop, the disappearance of the azide band at 2102 cm^{-1} and the terminal alkyne bands at 3311 and 2115 cm^{-1} is a very good evidence for the formation of compound **(6)a.**

FT-IR spectrum of 1-(4-bromophenyl)-2-(4-((heptyloxy)methyl)-1H-1,2,3-triazol-1-yl)ethanone **(6)b** showed the following bands cm^{-1}(KBr): 3090 (**C-H** aromatic) stretching , 2922, 2854 **(C-H** aliphatic**)** stretching, 1662**(C=O)** stretching, 1602, 1556 **(C=C** aromatic**)** stretching, 1180, 1082 (**C-O**) stretching 979-613 **(C-H** aromatic**)** bending oop, the disappearance of the azide band at 2102 cm^{-1} and the terminal alkyne bands at 3311 and 2116 cm^{-1} is a very good evidence for the formation of compound **(6)b.**

FT-IR spectrum of 1-(4-bromophenyl)-2-(4-((octyloxy)methyl)-1H-1,2,3-triazol-1-yl)ethanone **(6)c** showed the following bands cm^{-1}(KBr): 2922, 2854 **(C-H** aliphatic**)** stretching, 1662**(C=O)** stretching, 1614, 1581 **(C=C** aromatic**)** stretching, 1168, 1068 (**C-O**) stretching 977-617 **(C-H** aromatic**)** bending oop, the disappearance of the azide band at 2102 cm^{-1} and the terminal alkyne bands at 3311 and 2117 cm^{-1} is a very good evidence for the formation of compound **(6)c.**

FT-IR spectrum of 2-(4-((hexyloxy)methyl)-1H-1,2,3-triazol-1-yl)-1-(biphenyl-4-yl)ethanone **(7)a** showed the following bands cm^{-1}(KBr): 3031 (**C-H** aromatic) stretching , 2925, 2858 **(C-H** aliphatic**)** stretching, 1676 **(C=O)** stretching, 1602, 1556 **(C=C** aromatic**)** stretching, 1184, 1112 (**C-O**) stretching 950-696 **(C-H** aromatic**)** bending oop.

FT-IR spectrum of 2-(4-((heptyloxy)methyl)-1H-1,2,3-triazol-1-yl)-1-(biphenyl-4-yl)ethanone **(7)b** (figure 10) showed the following bands cm^{-1}(KBr): 2927, 2858 **(C-H** aliphatic**)** stretching, 1674 **(C=O)** stretching, 1585 **(C=C** aromatic**)** stretching, 1172, 1070 (**C-O**) stretching 952-763 **(C-H** aromatic**)** bending oop.

FT-IR spectrum of 2-(4-((octyloxy)methyl)-1H-1,2,3-triazol-1-yl)-1-(biphenyl-4-yl)ethanone **(7)c** showed the following bands cm^{-1}(KBr): 3031 (**C-H** aromatic) stretching , 2923, 2854 **(C-H** aliphatic**)** stretching, 1685 **(C=O)** stretching, 1583, 1556 **(C=C** aromatic**)** stretching, 1172, 1068 (**C-O**) stretching 983-761 **(C-H** aromatic**)** bending oop.

Appendix (1)

Table 1: some of the physical properties of the prepared compounds

Comp.	Physical state	bp^{o}C	mp^{o}C	R_f	Eluent	Yield %
2a	Yellow oil	88-90	-	0.79	*n*-hexane/EtOAc9:1	77
2b	Pale yellow oil	93-96	-	0.79	*n*-hexane/EtOAc9:1	58
2c	Yellow oil	100-102	-	0.78	*n*-hexane/EtOAc9:1	63
4a	Yellow solid	-	77-79	0.37	*n*-hexane/EtOAc2:1	81
4b	Yellow solid	-	123-125	0.36	*n*-hexane/EtOAc2:1	75
4c	Yellow solid	-	131-133	0.36	*n*-hexane/EtOAc2:1	83
5a	White solid	-	118-120	0.33	*n*-hexane/EtOAc1:1	74
5b	White solid	-	123-125	0.32	*n*-hexane/EtOAc1:1	79
5c	White solid	-	132-134	0.32	*n*-hexane/EtOAc1:1	71
6a	Pale yellow solid	-	177-180	0.35	*n*-hexane/EtOAc1:2	70
6b	Pale yellow solid	-	182-185	0.34	*n*-hexane/EtOAc1:2	73
6c	Pale yellow solid	-	196-199	0.32	*n*-hexane/EtOAc1:2	75
7a	Yellow solid	-	220-223	0.30	*n*-hexane/EtOAc1:2	77
7b	Yellow solid	-	228-231	0.31	*n*-hexane/EtOAc1:2	69
7c	Yellow solid	-	239-242	0.31	*n*-hexane/EtOAc1:2	70

Table 2: Some of the important FT-IR stretching of the synthesized compounds in cm-1

Comp.	C-H acetylenic	C-H aromatic	C-H aliphatic	C≡C	$-N_3$	C=O	C=C
2a	3311	-	2927, 2858	2115	-	-	-
2b	3311	-	2927, 2856	2116	-	-	-
2c	3311	-	2927, 2856	2117	-	-	-
4a	-	3060	2927, 2862	-	2102	1668	1600, 1490
4b	-	3090	2925, 2856	-	2102	1660	1595, 1519
4c	-	3091	2927, 2868	-	2106	1668	1587
5a	-	3095	2929, 2862	-	-	1672	1581, 1442
5b	-	3100	2923, 2854	-	-	1679	1604, 1442
5c	-	-	2925, 2856	-	-	1676	1604
6a	-	3095	2927, 2862	-	-	1668	1602, 1452
6b	-	3090	2922, 2854	-	-	1662	1602, 1556
6c	-	-	2922, 2854	-	-	1662	1614, 1581
7a	-	3031	2925, 2858	-	-	1676	1602, 1556
7b	-	-	2927, 2858	-	-	1674	1585
7c	-	3031	2923, 2854	-	-	1685	1583, 1556

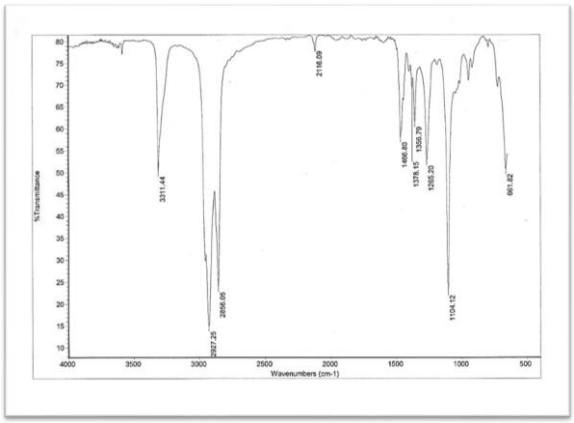


Figure 1: FT-IR spectrum of compound (2)b

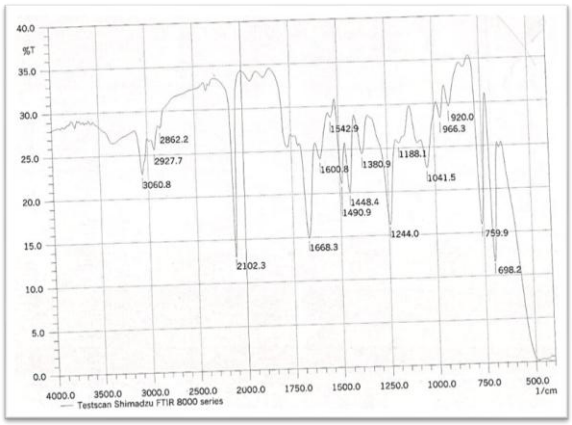


Figure 4: FT-IR spectrum of compound (4)a

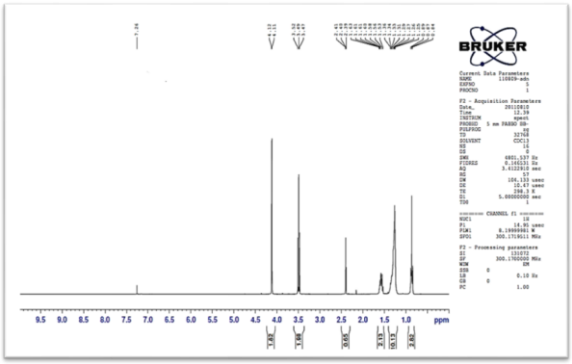


Figure 3: 1H NMR spectrum of compound (2)c

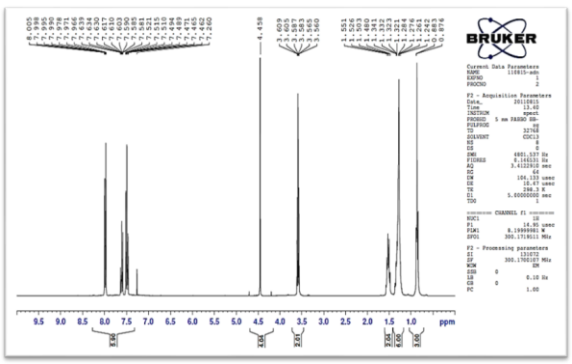


Figure ٦ :1H NMR spectrum of compound (5)a

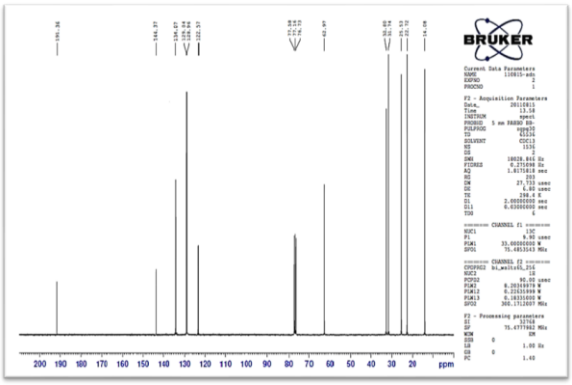


Figure 7: 13C NMR spectrum of compound (5)a

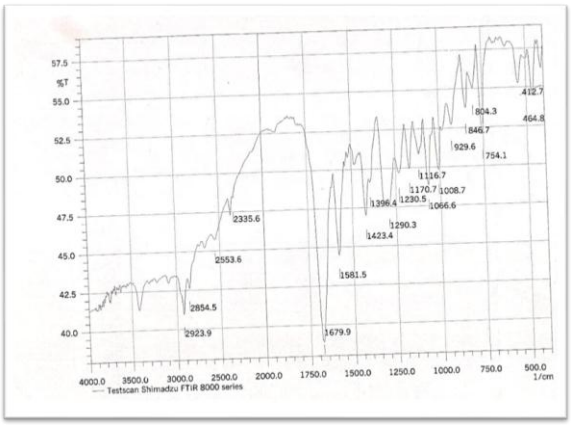


Figure 8: FT-IR spectrum of compound (5)b

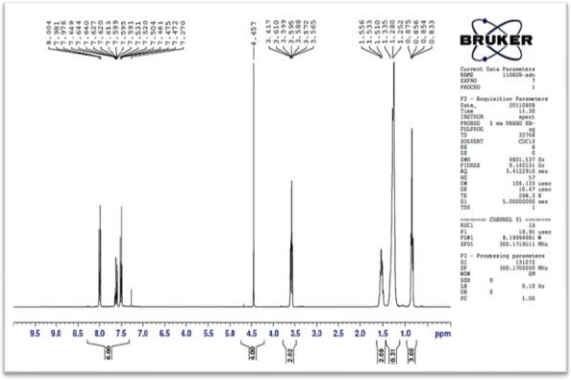


Figure 9: 1H NMR spectrum of compound (5)c

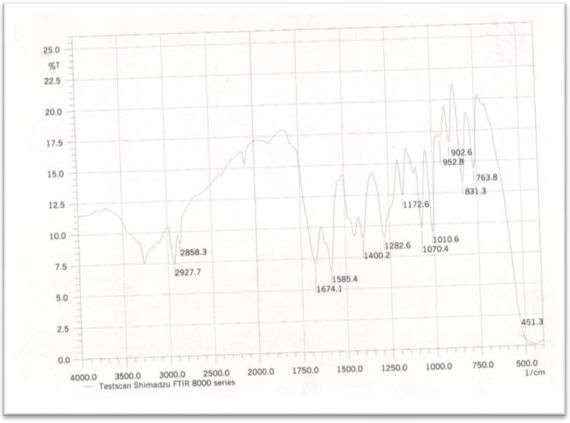


Figure 10: FT-IR spectrum of compound (7)b

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