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Synthesis of Benzotriazole Derivatives

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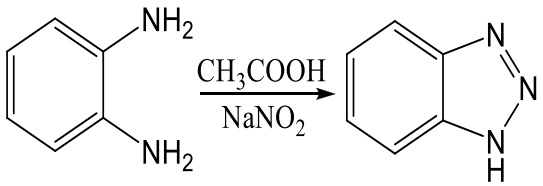
Abstract:
In our present study benzene-1, 2-diamine (1) has been reacted with sodium nitrite in present glacial acetic acid yielded 1H-benzo[d]1,2,3-triazole(2), Which react with 2-chloro-N-phenylacetanide, 2-chloro-N-(4-chlorophenyl)acetamide, methyl 4-(2-chloroacetamido) benzoate, ehtyl4- (2 – chloro acetamido) benzoate, 2-chloro - N - (2-nitrophenyl) acetamide, 2-chloro - N- (4 - nitrophenyl) acetamide, 2-chloro -N- (3-hydroxy phenyl) acetamide, 2-chloro - N- (p-tolyl) acetamide, 2-chloro-N-(3-nitropheny) acetamide and 2-chloro-N-(4-methoxy-3-nitrophenyl) acetamide to give 2-(1H-benzo[d]1,2,3-triazol-1-yl) -N - (phenyl) acetamide (3) , 2-(1H-benzo[d] 1,2,3-triazol-1-yl) - N -(2-chlorophenyl) acetamide (4), 2-(1H-benzo [d] 1,2,3-triazol-1-yl) - N -(4-chlorophenyl) acetamide (5), methyl 4- (2- (1H-benzo[d]1,2,3-triazol-1-yl) acetamido) benzoate (6), ethyl 4-(2-(1H-benzo[d]1,2,3-triazol-1-yl) acetamido) benzoate (7).
All the synthesized compounds were characterized on the basis of melting point, TLC, IR, ^{1}HNMR, ^{13}CNMR and mass spectrometry.

Kay words: benzene-1, 2-diamine, sodium nitrite, glacial acetic acid, 2-chloro- N- (substituted phenyl) acetamide, alkyl 4-(2-chloroacetamido)benzoate, 1H-benzo[d] 1,2,3-triazole, K_2CO_3, DMF.

I. Introduction

Benzotriazoles are lesson of heterocyclic organic compounds having a ring framework containing three nitrogen molecules and intertwined benzene ring appears wide run of organic exercises.
It is synthesized by diazotization process using benzene-1,2-diamine with sodium nitrite and glacial acetic acid[(1,2)]



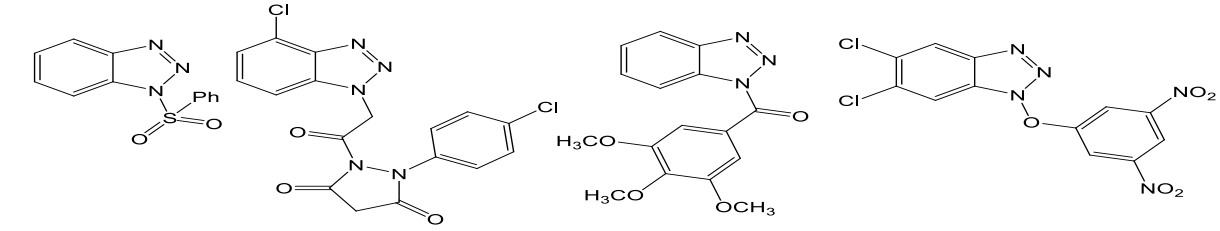
(1) (2)

Physical properties are listed in the below table:	
Molecular formula	**$C_6H_5N_3$**
Molecular weight	**119.1240**
Melting point	**98.5 -100^{0}C**
Nature	**White to brown crystalline powder**
Density	**1.36 g/ cm^3**
Solubility	**g/ 100ml is 2(moderate)**
CAS Registry number	**95-14-7**
UV absorbance	**286 nm**

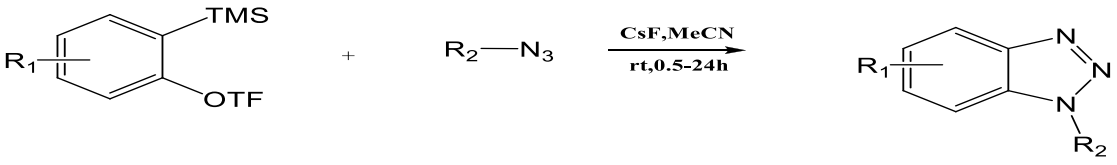
Beside that, amid functional group play a vital role in organic synthesis [3,4]. A large number of natural and synthetic formation possess this functional group. The synthetic chemists are always looking for better methods for formation of amide bond[5-10]. There are extraordinary intrigued of triazole lesson emerging due to their wide utilize in industry and agri business. Benzotriazole and its derivatives have great significance in medicinal chemistry[11]. The consolidation of the Benzotriazole cores is an imperative engineered procedure in medicate revelation. The tall helpful properties of the related drugs have energized the restorative chemists to synthesize the expansive number of novel chemotherapeutic agents[12]. In general, nitrogen and sulfur containing organic compounds and their metal complexes display a wide range of biological activity as antitumor, antibacterial, antifungal and antiviral agents[13]. Benzotriazoles are frequently utilized as erosion inhibitors, radio protectors, and photo stabilizer within the generation of plastic, elastic and chemical fiber 3. In conjunction with these exercises, benzotriazole is additionally critical as a antecedent within the blend of peptides, corrosive azides, planning of 3hydroxymethyl-2,3-dihydrobenzofurans and 3-hydroxymethylbenzofurans[14] N-

Substituted benzotriazoles exist as two isomers:1H- and 2H-substituted. It is by and large concurred that 1H-substituted ruled in strong and arrangement, while the extent of the 2H-tautomer expanded within the gas phase[15]. However, the energy difference between the two isomers is very little[16, 17].

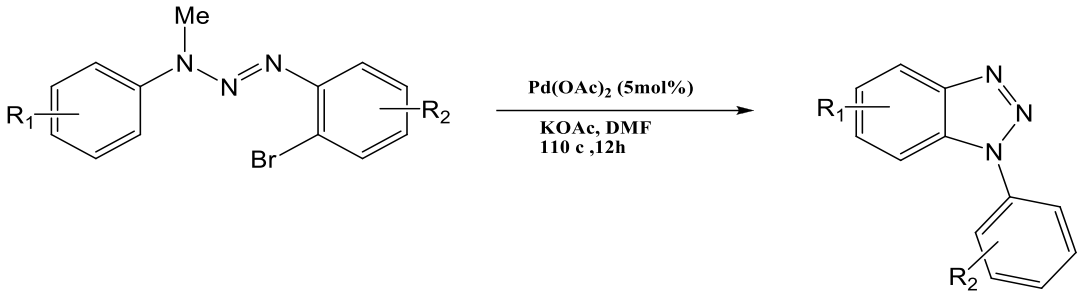
Benzotriazoles are important heterocyclic scaffolds, widely used in medicinal chemistry[18-20], organic synthesis[21-23] and material science.[24,25]Application of benzotriazole derivatives in medicinal chemistry is particularly widespread due to enzyme inhibition through π–π stacking or hydrogen bonding of the triazole unit.[a] For example, antifungal benzotriazole derivatives have been discovered that inhibit the growth of fluconazole-insensitive Cryptococcus neoformans,[b] while halogenated aryloxy-benzotriazoles inhibit isoniazid-resistant. Mycobacterium tuberculosis.[c] In natural blend, benzotriazoles have been utilized as antecedents for the arrangement of other heterocycles such as indoles, carbazoles as well as pyridoacridines,[26-31]and seminal work by Katritzky and co-workers demonstrated their application as auxiliaries for alkylation and benzannulation reactions.[32]



Figure(a) representative example of pharmaceutically active benzotriazole



Figure(b) cycloaddition reaction of benzynes with azides



Figure(c) 1,7-palladium migration-cyclization-dealkylation sequence

II. Materials And Methods

Melting point were decided in open capillary tube on VEEGO (VMP-D) softening point device and are uncorrected.. IR spectro (KBr pellets) were recorded on a SHINADZU FTIR 8400S infrared spectrophotometer. The ^{1}HNMR spectra were determined in DMSO –d_6 at 300 MHz on a BRUKER DP-X300NMR spectrophotometer using TMS as an internal standard. The monitored by TLC using silica gel plates. C13NMR were measured on Bruker 400MHz with internal reference TMS $\delta = 0$. Mass spectra were recorded at 70 ev with a GCMS – QP 1000EX spectrometer.

Synthesis of 2-(1H-benzo[d]1, 2, 3 –triazol-1-yl) –N- (substituedphenyl) acetamide (3, 4, 5) and alkyl 4-(2- (1H-benzo[d]1, 2, 3 –triazol-1-yl) acetamido) benzoate (6, 7).
Equimolar quantity of 2-hloro – N – substituted phenyl) acetamide (0.01mol) and alkyl 4- (2 - chloro acetamido) benzoate (0.01mol) with 1H – benzo[d] 1, 2, 3 –triazole (2) (0.01 mol) in present K_2CO_3 were dissolved in DMF, this mixture was heated on water bath for 24 hrs. the reaction was cooled at room temperature and poured into water (200 ml) with stirring for 15min, the solid obtained was filtrated and finally recrystallized from absolute ethanol.

2-(1H-benzo[d]1, 2, 3-triazol -1-yl)-N-phenylacetamide (3). Yield 63%, m.p. 225-226 ^{0}C . IR ($\bar{\upsilon}_{max}$, cm^{-1}): 3282(NH), 3087(CH- aromatic), 2920(CH- alphatic), 1690(CON) and 1609(C=N). ^{1}HNMR (DMSO, δ_H , ppm):5.7(s, 2H, CH_2CO), 7.1 - 8.1 (m, 9H, aromatic-H) and 10.6 (s, 1H, NH). M/S, m/z (%) = 252(M^+, 19), 118(M^+, C_8H_8NO, 18), 99 (M^+, $C_{12}H_9$, 17) and 56 (M^+, $C_{13}H_{10}NO$, 12). Anal. Calc. for $C_{14}H_{12}N_4O$ (252): C 66.65, H 4.79, N 22.21%, found: C 67.00, H 5.01, N 22.50%.

2-(1H-benzo[d]1, 2, 3-triazol -1-yl)-N-(2-chlorophenyl) acetamide (4). Yield 53%, m.p. 183-182 ^{0}C . IR ($\bar{\upsilon}_{max}$, cm^{-1}): 3264(NH), 3060(CH- aromatic), 2920(CH-alphatic), 1668(CON) and 1539(C=N). ^{1}HNMR (DMSO, δ_H , ppm): 5.8(s, 2H, CH_2CO), 7.2 -8.1 (m, 8H, aromatic-H) and 10.2 (s, 1H, NH). M/S, m/z (%) = 286(M^+, 4), 251(M^+, Cl, 11), 132(M^+, C_7H_5ClNO, 11), 104 (M^+, $C_8H_7ClN_2O$, 18), 77(M^+, C_8H_8ClNO, 86), 78(M^+, C_8H_9ClNO, 28), 51(M^+, $C_{10}H_{10}ClN4O$, 100). Anal. Calc. for $C_{14}H_{11}ClN_4O$ (286): C 58.65, H 3.87, Cl 12.37, N 19.54%, found: C 58.90, H 4.05, Cl 11.98, N 19.99%.

2-(1H-benzo[d]1, 2, 3-triazol -1-yl)-N-(4-chlorophenyl) acetamide (5). Yield 67%, m.p. 244-247 ^{0}C . IR ($\bar{\upsilon}_{max}$, cm^{-1}): 3260(NH), 3062(CH- aromatic), 2981(CH-alphatic), 1691(CON) and 1613(C=N). ^{1}HNMR (DMSO, δ_H , ppm): 5.7(s, 2H, CH_2CO), 7.2 -8.1 (m, 8H, aromatic-H) and 10.8 (s, 1H, NH). M/S, m/z (%) = 286(M^+, 2), 104(M^+, $C_8H_7ClN_2O$, 28), 154(M^+, $C_7H_6N_3$, 2), 126 (M^+, $C_8H_6N_3O$, 11), 76(M^+, $C_8H_7ClN_4O$, 32), 77(M^+, $C_8H_8ClN_4O$, 100), 78 (M^+, $C_8H_9ClN_4O$, 36). C^{13}NMR:

50.30(1C), 110.80(1C), 118.98(1C), 120.27(2C), 123.82(2C), 127.32(2C), 128.74(1C), 133.80 (1C), 137.31(1C), 145.07(2C) and 164.50(1C). Anal. Calc. for $C_{14}H_{11}ClN_4O$ (286): C 58.65, H 3.87, Cl 12.37, N 19.54%, found: C 58.90, H 4.03, Cl 12.55, N 19.90%.

Methyl4-(2-(1H-benzo[d]1, 2, 3-triazol-1-yl) acetamido) benzoate (6). Yield 58%, m.p. 251-252 ^{0}C . IR ($\bar{\upsilon}_{max}$, cm^{-1}): 3265(NH), 3069(CH- aromatic), 2878(CH- alphatic), 1700 ($COOCH_3$), 1606(CON) and 1550 (C=N). ^{1}HNMR (DMSO, δ_H , ppm):3.8(s, 3H, $COOCH_3$), 5.7(s, 2H, CH_2CO), 7.4 -8.1 (m, 8H, aromatic-H) and 11.02 (s, 1H, NH). M/S, m/z (%) = 310(M^+, 0.2), 132(M^+, $C_9H_8NO_3$, 15), 119(M^+, $C_{10}H_{11}NO_3$, 9), 104(M^+, $C_{10}H_{11}N_2O_3$, 33), 76(M^+, $C_{10}H_{12}N_4O_3$, 33), 77 (M^+, $C_{10}H_{13}N_4O_3$, 100) and 78 (M^+, $C_{10}H_{14}N_4O_3$,48). C^{13}NMR: 50.049(1C), 51.83(1C), 110.86(1C), 118.86(1C), 123.82 (2C), 124.45 (2C), 127.83 (2C), 130.34 (1C), 133.82 (1C), 142.71 (1C), 145.33 (1C) and 165.66(1C). Anal. Calc. for $C_{16}H_{14}N_4O_3$ (310): C 61.93, H 4.55, N 18.06 %, found: C 62.80, H 4.61, N 18.01 %.

Ethyl4-(2-(1H-benzo[d]1, 2, 3-triazol-1-yl) acetamido) benzoate (7). Yield 61%, m.p. 96-97 ^{0}C . IR ($\bar{\upsilon}_{max}$, cm^{-1}): 3265(NH), 3069(CH- aromatic), 2984(CH- alphatic), 1700 ($COOC_2H_5$), 1605(CON) and 1549(C=N). ^{1}HNMR (DMSO, δ_H , ppm): 1.3(t , 3H, $COOCH_2\underline{CH_3}$), 4.28(q, 2H, $COO\underline{CH_2}CH_3$) 5.7(s, 2H, CH_2CO), 7.4 -8.1 (m, 8H, aromatic-H) and 11.0 (s, 1H, NH). M/S, m/z (%) = 324 (M^+, 0.5), 160(M^+, $C_9H_{10}NO_2$, 1), 132(M^+, $C_{10}H_{10}NO_3$, 8), 119(M^+, $C_{11}H_{13}NO_3$, 6), 104(M^+, $C_{11}H_{14}N_2O_3$, 27), 76(M^+, $C_{11}H_{14}N_4O_3$, 21), 77(M^+, $C_{11}H_{15}N_4O_3$, 100), 78(M^+, $C_{11}H_{16}N_4O_3$, 29). C^{13}NMR: 14.08(1C), 50.47(1C), 60.40(1C), 110.87(1C), 118.65 (1C), 123.82(2C), 124.71(2C), 127.33 (2C), 130.28(1C), 133.82(1C), 142.67(1C), 145.33(1C), 164.95 (1C) and 165.15 (1C). Anal. Calc. for $C_{17}H_{16}N_4O3$ (324): C 62.95, H 4.97, N 17.27 %, found: C 62.98, H 5.04, N 17.33 %.

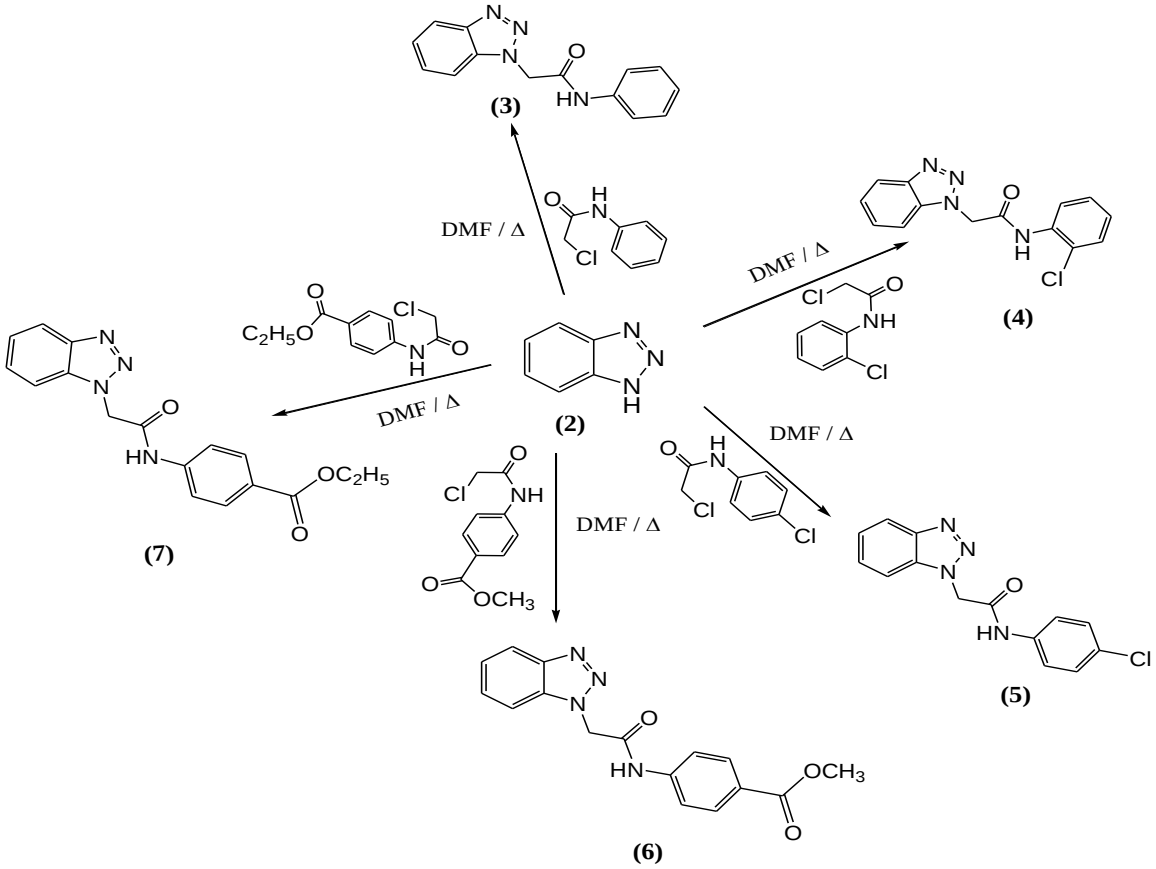
III. Results and Discussion

In the present work, 1H-benzo[d]1, 2, 3-triazole derivatives (3, 4, 5, 6, 7) obtained by reacting1H-benzo[d]1, 2, 3-triazole (3) with 2-chloro-N-(substituted phenyl) acetamide and alkyl 4-(2-chloroacetamido) benzoate in DMF in present K_2CO_3 on water bath for 24 hrs. The structure of compound (3) was confirmed by elemental analysis and the IR spectrum, which showed stretching bands at 3282, 1690 and 1609 cm^{-1}correarance to NH, C=O and C≡N groups respectively. The 1HNMR spectrum revealed the appearance of singlet signal at δ 4.7 ppm attributed methylenic protons, in addition to multiplet signals at δ 7.1-8.1 ppm to night aromatic protons and singlet signal at δ 10.6 ppm (NH). The mass spectrum of (3) showed molecular ion peak at m/z 252.

The structures of both compounds (4) and (5) were characterized by the presence of strong absorption bands of amidic carbonyl group at 1668 cm^{-1} and 1691 cm^{-1} respectively, but absorption bands of (-NH) group of compounds (4) and (5) appeared at 3264 cm^{-1} and 3260 cm^{-1} respectively. The ^{1}HNMR spectrum of compound (4) and (5) showed singlet signal of (-NH) group at δ 10.2 ppm and δ 10.8 ppm respectively, also appeared singlet signal of (- CH_2-) group at δ 5.8 ppm of compound (4), but its showed singlet signal at δ 5.7 ppm of compound (5). The mass spectrum of compounds (4) and (5) showed molecular ion peak at 286 that was consistent with the molecular weight of compounds. ^{13}CNMR spectrum of compound (5) has on single at 164. 50 ppm indicated to carbonyl group, additional to serval single from 110.86 to 145.07 ppm (carbon of aromatic rings), in additional single peak at 50.39 ppm that indicated to (-CH_2-) group.

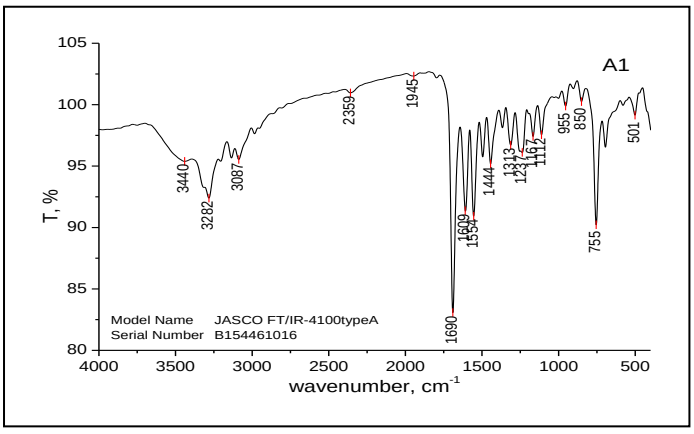
The structure of compounds (6) and (7) were confirmed via ^{1}HNMR spectrum which revealed singlet signals of amidic group at δ 11.0 ppm, also singlet signal of (-$COOCH_3$) group at δ 3.8 ppm for compound (6), but its

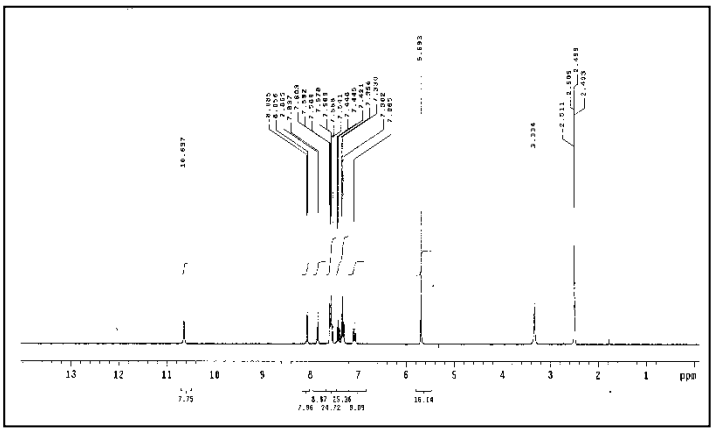
appeared triplet and quartet signals of compound (7) at δ 1.3 ppm and δ 4.28 ppm for ($COOCH_2\underline{CH_3}$) and($COO\underline{CH_2}CH_3$) groups respectively. In other side its appeared methylene group of compounds (6) and (7) at δ 5.7 ppm. IR spectrum of compounds (6) and (7) showed strong absorption bands of amidic and ester groups at 3265 cm^{-1} and 1700 cm^{-1} respectively. The mass spectrum of compounds (6) and (7) revealed m/z 310 and m/z 324 which corresponding to the molecular formula $C_{16}H_{14}N_4O_3$ and $C_{17}H_{16}N_4O_3$ respectively. ^{13}CNMR spectrum of compounds (6) and (7) appeared carbonyl groups of amide (-CO-) at 165.66 and 165.15 ppm respectively and carbonyl group of ester for both compounds appeared at 164 ppm, also (-CH2-) group observed at 51.83 ppm and 50.47 ppm respectively. In addition two peaks at 60.40 ppm and 14.08 ppm correspond to the ethyl group of ester for compound (7) whereas present on peak at 50.49 ppm of compound (6) indicated to methyl group of ester.



SCHEME 1

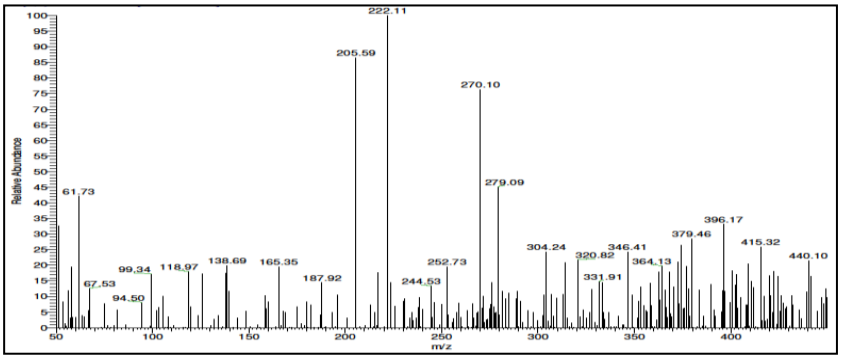
Spectral data of compounds , spectral data of compound (3)





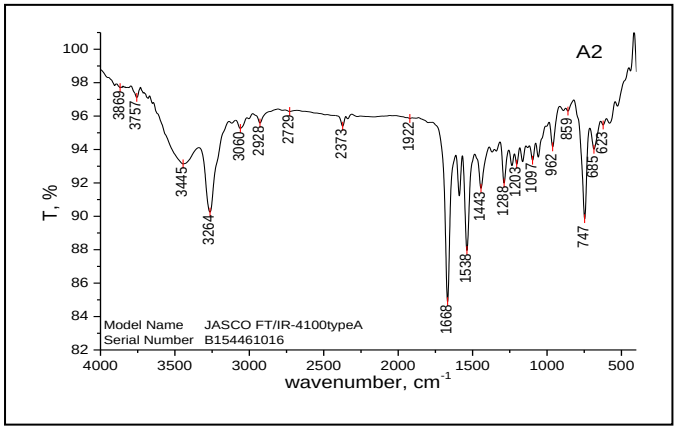
IR spectrum (cm^{-1}) of compound (3)

^{1}HNMR spectrum of compound (3)

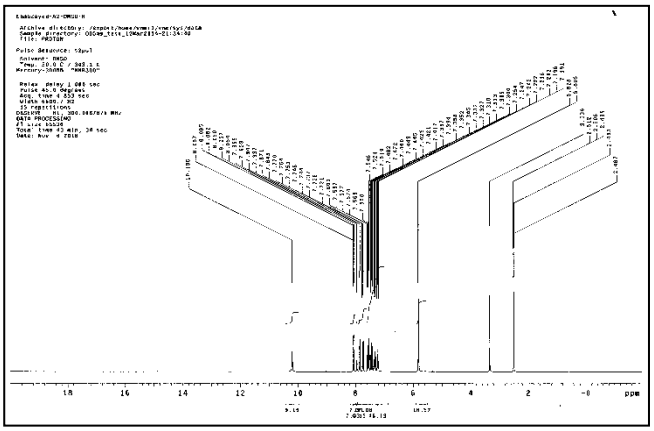


Mass spectrum of compound (3)

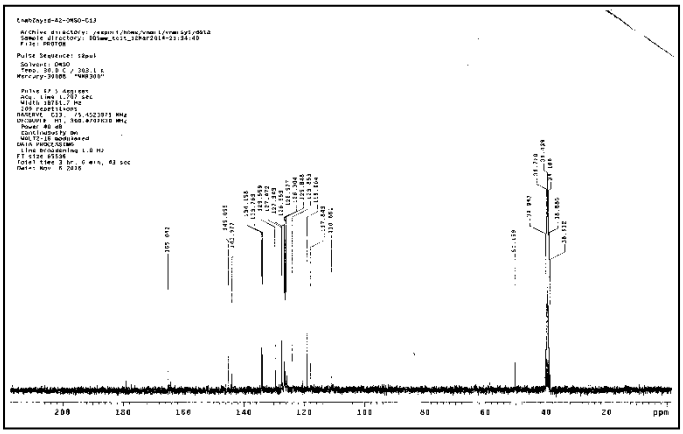
1) spectral data of compound (4)



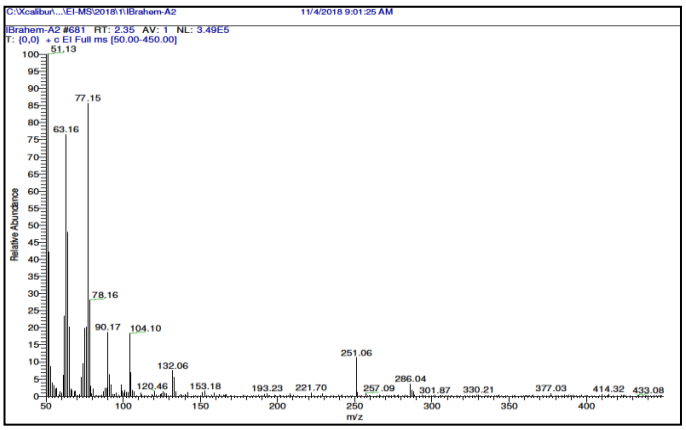
IR spectrum(cm-1) of compound(4)



^{1}HNMR spectrum of compound (4)

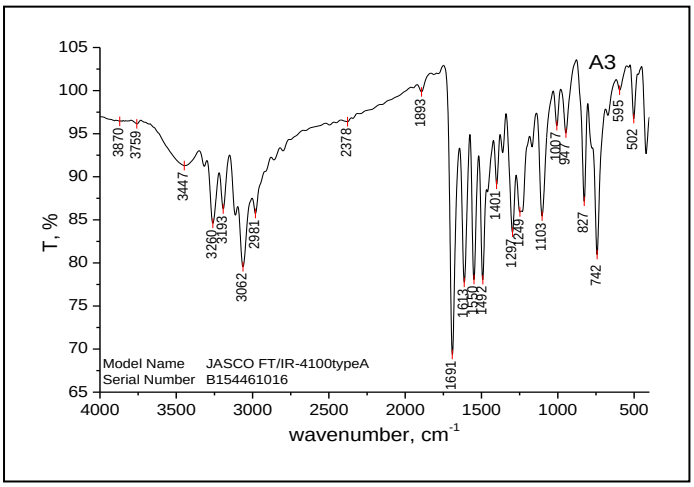


^{13}CNMR spectrum of compound (4)

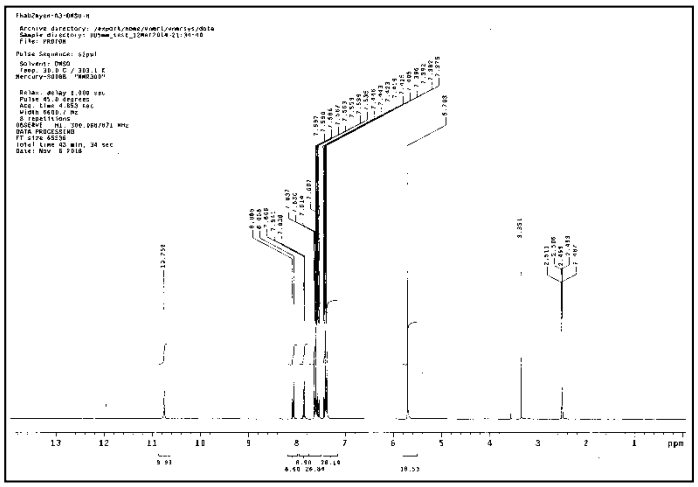


Mass spectrum of compound (4)

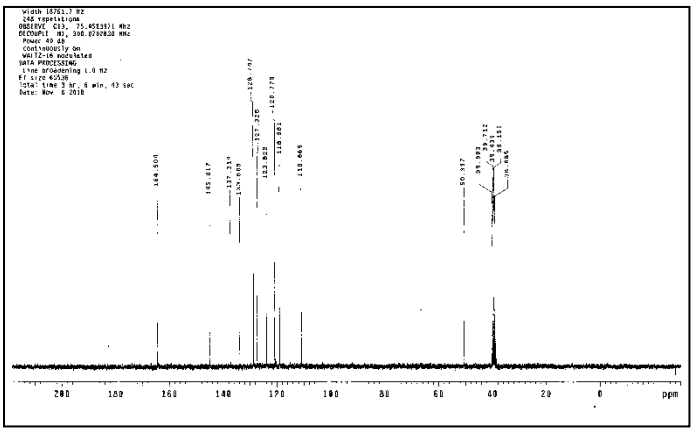
2) spectral data of compound (5)



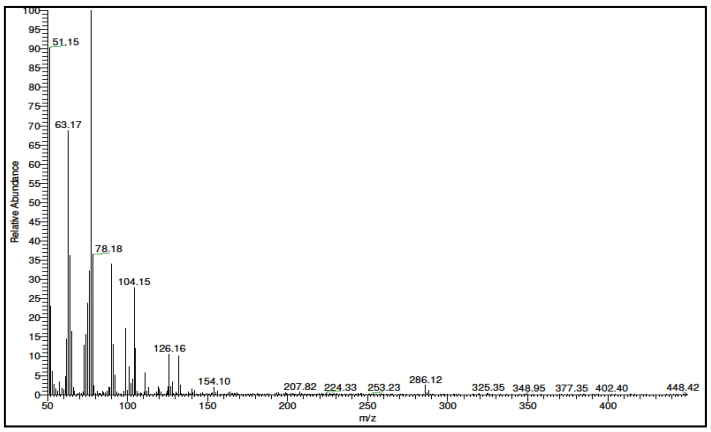
IR spectrum(cm^{-1}) of compound (5)



^{1}HNMR spectrum of compound (5)

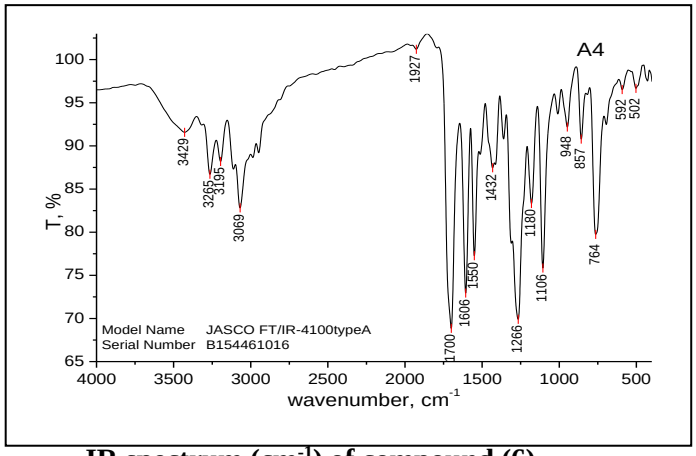


^{13}CNMR spectrum of compound (5)

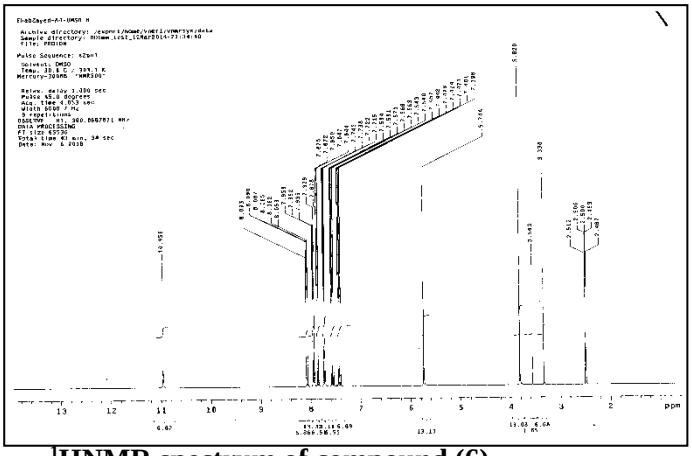


Mass spectrum of compound (5)

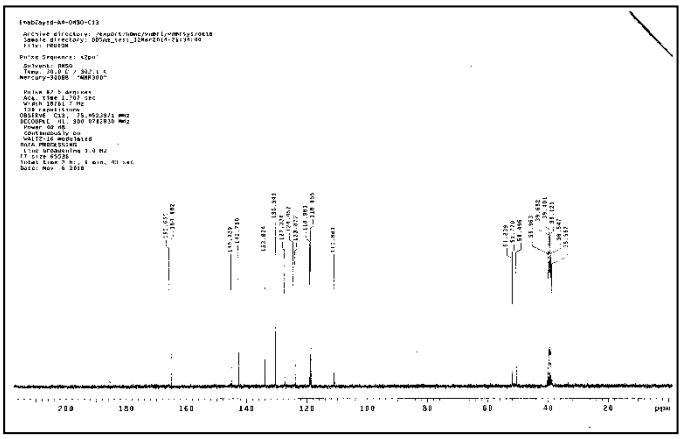
3) spectral data of compound (6)



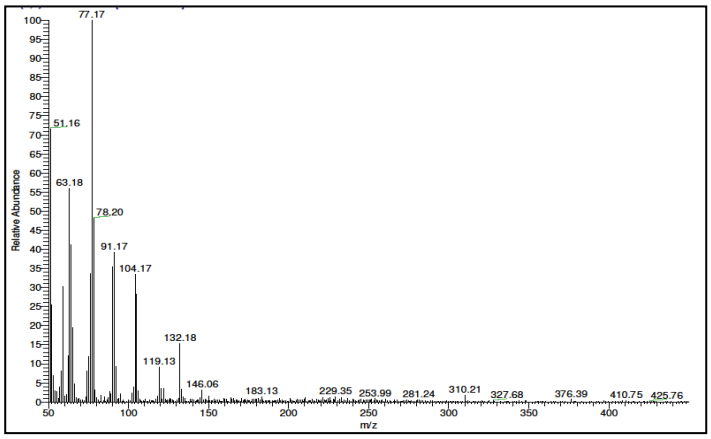
IR spectrum (cm^{-1}) of compound (6)



^{1}HNMR spectrum of compound (6)

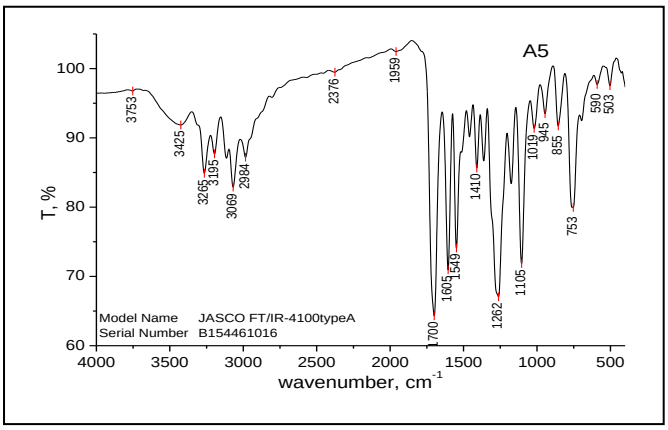


^{13}CNMR spectrum of compound (6)

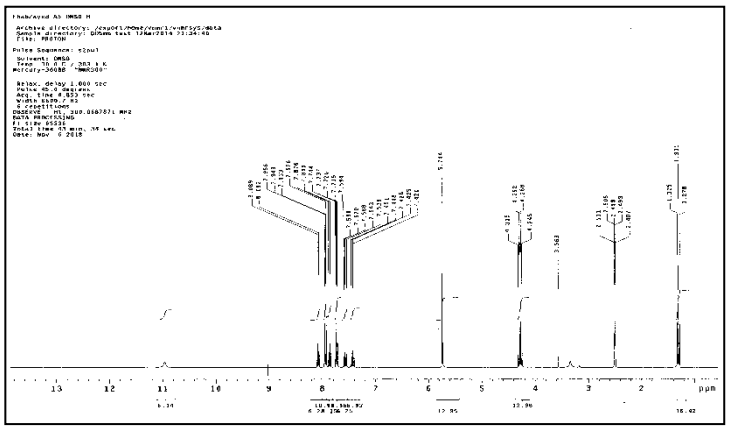


Mass spectrum of compound (6)

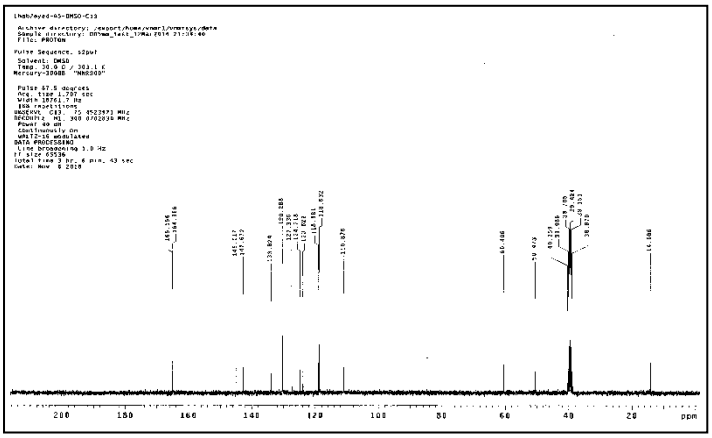
4) spectral data of compound (7)



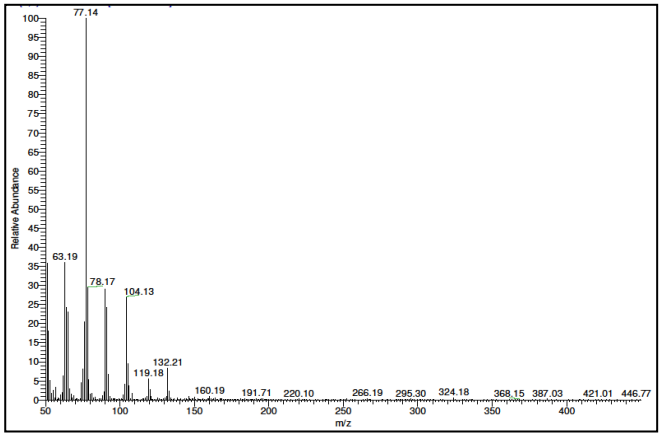
IR spectrum (cm^{-1}) of compound (7)



^{1}HNMR spectrum of compound (7)



^{13}CNMR spectrum of compound (7)



Mass spectrum of compound (7)

In Conclusion, Benzotriazole derivations have gained considerable importance in medicinal chemistry, due to their broad spectrum as antiviral, antibacterial, anticancer, etc. agents, their synthesis has become of great interest. We moreover deliver spots on the science of

the target particle as imminent antiviral drugs.

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