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Study of Reactions of 2,2-Dichloro-*N*-(Substituted Phenyl) Acetamide With 1H-Benzo[d]1,2,3-Triazole

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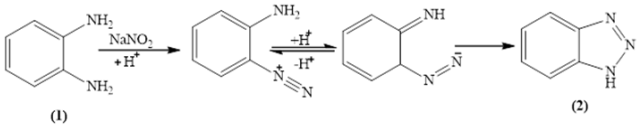
A B S T R A C T

Synthesis of 2,2-dichloro-*N*-(substituted phenyl) acetamid **(a_{1-4})** via treated substituted aniline with dichloroacetyl chloride in the present of anhydrous K_2CO_3 in CH_2Cl_2. The later 1*H*-benzo[d]1,2,3-triazole **(2)** were reacted with 2,2-dichloro-*N*-(4-chloro phenyl) acetamid **(a_1)**, 2,2-dichloro-N-(3-nitro phenyl) acetamid **(a_2)**, 2,2-dichloro-N-(4-methoxy-3-nitrophenyl) acetamid **(a_3)**, 2,2-dichloro-N-(p-tolyl) acetamid **(a_4)** in the present of anhydrous K_2CO_3 in acetone under reflux to give 2-(1*H*-benzo[d]1,2,3-triazol-1-yl)-2-chloro-*N*-(4-chloro phenyl) acetamide **(3)**, 2-(1*H*-benzo[d]1,2,3-triazol-1-yl)-2-chloro-*N*-(3-nitro phenyl) acetamide **(4)**, 2-(1*H*-benzo[d]1,2,3-triazol-1-yl)-2-chloro-*N*-(4-methoxy-3-nitro phenyl) acetamide **(5)** and 2-(1*H*-benzo[d]1,2,3-triazol-1-yl)-2-chloro-*N*-(p-tolyl) acetamide **(6)** respectively.

All the synthesized compounds were characterized on the basis of melting point, TLC, IR, ^{1}HNMR, ^{13}CNMR and mass spectrometry.

1 Introduction

One of the most potent antibacterial medications used either alone or in combination for cancer therapy is heterocyclic compounds with nitrogen atoms (Li et al., 2007). According to a recent study (Biagi et al., 2001), some benzotriazole derivatives formed an intriguing family of heterocycles and became the most quickly developing class of antifungal compounds with the benefit of low toxicity (Scapin et al., 2003), broad range action and good oral bioavailability (Carta et al., 2004). There are two isomers of N-substituted benzotriazoles: 1H and 2H substituted. It is commonly acknowledged that in the solid phase, 1H substitutions predominate, whereas in the gas phase, the proportion of 2H tautomers increase (Catalán et al., 1998). However, there is relatively little energy difference between the two isomers (Wang et al., 2000). On the other hand, o-phenylene diamines and sodium nitrite are cyclocondensed with one another in acetic acid to produce benzotriazoles. The reagents were simply heated together to initiate the reaction. Followed by spontaneous cyclization after the diamine is transformed into derivatives of monodiazonium (Furniss et al., 1989).



(1) (2)

The 1,2,3-benzotrazole has been produced directly through the action of nitrous acid on *o*-phenylene diamine and through the hydrolysis of an acylate or aroylated benzotriazole that has already been produced through the aforementioned procedure. This direct method provides a higher overall yield than methods involving a number of intermediate steps (Khalafi-Nezhad et al., 2007).

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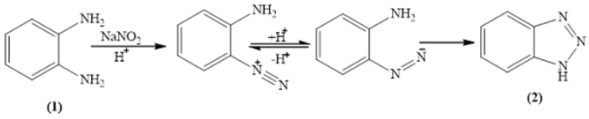


A popular technique for creating amide bonds is to condense an amine or aniline with a carboxylic acid or its derivatives (Chen et al., 2005). The provided techniques are not appropriate for all classes of universally applicable abstract objects. Due to their extensive role in the synthesis of biologically active compounds that exhibit pharmaceutical properties like analgesic activities (Özkay et al., 2011), the synthesized *N* - phenylamides *N* - benzothiazol – 2 – yl acetamides and *N*- (4-pheylthiazol-2-yl) aceamides have attracted significant interest in recent years. HIV-1 reverse transcriptase inhibitor (Chander et al., 2015), anticancer action (Kumar et al., 2014), antibacterial and antituberculosis (Patel et al., 2013), COX-2 inhibitors (Raghavendra et al., 2012) and sp1 receptor agonist (Bolli et al., 2010).

2 Materials and Method

Melting points were determined in open capillary tube on VEEGO (VMP- D) softening point device and are uncorrected. IR spectra (KBr pellets) were recorded on a SHINADZU FTIR 8400S infrared spectrophotometer. The ^{1}HNMR spectra were determined in DMSO-d6 at 300 MHz on a BRUKER DP-X300NMR spectrophotometer using TMS as an internal standard. The reactions were monitored by thin layer chromatography (TLC). ^{13}CNMR were measured on Bruker 400MHz with internal reference TMS δ = 0. Mass spectra were recorded at 70 ev with a GCMS-QP 1000EX spectrometer. Elemental analyses were carried out at the Microanalytical laboratory of the faculty science, Cairo University, Giza, Egypt.

Synthesis of 2,2-dichloro-*N*-(substituted phenyl) acetamid(a_{1-4}).

To magnetically stirred solution of substituted aniline (0.01 mol) and K_2CO_3 (0.01mol) in CH_2Cl_2 (100ml), cool in an ice bath, dichloroacetyl chloride (0.01mol) was added slowly dropwise. The reaction mixture was stirred at room temperature and monitored by TLC. After the reaction was complete. The solvent was removed under vacuum and ice water(200ml) was added to the residue. The precipitate was filtered washed with water, and finally recrystallized from absolute ethanol.

2,2-Dichloro - *N* - (4 - chlorophenyl) acetamide(a_1).

Yield 75 %, m.p.142 ^{0}C, IR ($\bar{\upsilon}_{max}$, cm^{-1}): 3276 (NH), 3020(CH- aromatic), 2724(CH-aliphatic) and 1677(CON). ^{1}HNMR (DMOS, δH, ppm): 6.6 (s, 1H, CH), 7.6-7.7 (m,4H, aromatic-H) and 10.8(s, 1H, NH). M/S, m/z (%) = 237(M^+,7%), 126(M^+, C_6H_4Cl, 27%), 111(M^+, C_6H_5NCl, 7%), 83(M^+, C_7H_5NClO, 63%) and 76(M^+, $C_2H_2NCl_3O$, 100%). ^{13}CNMR: 67.16 (1C), 121.36 (2C), 128.37(2C), 128.74 (1C) 136.43(1C) and 161.73(1C). Anal. Calc. for C_8H_6ClNO (238): C 40.29, H 2.54, Cl 44.60, N 5,87 %, found: C 40.28, H 2.56, Cl 44.59, N 5.89%.

2, 2-Di chloro - *N* - (3 - nitrophenyl) acetamide (a_2).

Yield 77 %, m.p.109-110 ^{0}C, IR ($\bar{\upsilon}_{max}$, cm^{-1}): 3342 (NH), 3018 (CH- aromatic), 2984(CH-aliphatic) and 1694(CON). ^{1}HNMR (DMOS, δH, ppm): 6.6 (s, 1H, CH), 7.6-8.6 (m,4H, aromatic-H) and 11.2 (s, 1H, NH). M/S, m/z (%) = 248(M^+, 0.2%), 165(M^+, $CHCl_2$, 20%), 137(M^+, C_2HCl_2O, 6%), 91(M^+, $C_2HNCl_2O_3$, 12%) and 76(M^+, $C_2H_2N_2Cl_2O$, 100%).^{13}CNMR: 67.86 (1C), 113.9 (1C), 118.98(1C), 125.67(1C), 130.31 (1C), 138.65(1C), 147.83(1C) and 162.76(1C). Anal. Calc. for $C_8H_6Cl_2N_2O_3$ C 38.58, H 2.43, Cl 28.47, N 11. 25, found: C 38.60, H 2.41, Cl 28.49, N 11.22 %.

2,2-Dichloro - *N* - (4-methoxy-3 - nitro phenyl) acetamide(a_3).

Yield 70 %, m.p. 97-99 ^{0}C, IR ($\bar{\upsilon}_{max}$, cm^{-1}): 3277(NH), 3016(CH- aromatic), 2974(CH-aliphatic) and 1691(CON). ^{1}HNMR (DMOS, δH, ppm): 3.9(s, 3H, OCH_3), 6.7 (s, 1H, CH), 7.4 (d, 2H, aromatic-H), 7.5(t, 2H, aromatic-H) and 10.8 (s, 1H, NH). M/S, m/z (%) = 278(M^+, 3%), 277(M^+, H, 21%), 231(M^+, HNO_2, 22%), 151(M^+, $CHNCl_2O_2$, 48%), 120(M^+, $C_2H_4NCl_2O_3$, 16%) and 76(M^+, $C_3H_6N_2Cl_2O_4$, 100%). Anal. Calc. for $C_9H_8Cl_2N_2O_4$ C 38.73, H 2.89, Cl 25.41, N 10.04, found: C 38.70, H 2.91, Cl 25.38, N 10.08.

2,2-Dichloro - *N* - (4 –toly) acetamide(a_4).

Yield 79 %, m.p. 159-160 ^{0}C, IR ($\bar{\upsilon}_{max}$, cm^{-1}): 3242(NH), 3060(CH- aromatic), 2928(CH-aliphatic) and 1670(CON). ^{1}HNMR (DMOS, δH, ppm): 2.3(s, 3H, -CH_3), 6.6 (s, 1H, CH), 7.2 (d,2H, aromatic-H), 7.5(t, 2H, aromatic-H) and 10.5 (s, 1H, NH). M/S, m/z (%) = 217(M^+, 16%), 134(M^+, $CHCl_2$, 26%), 106(M^+, C_2HCl_2O, 64%) and 76(M^+, $C_3H_5NCl_2O$, 100%). ^{13}CNMR: 67.30(1C), 115.70(2C), 125.30(2C), 133.68(1C), 134.57(1C) and 161.46(1C). Anal. Calc. for $C_9H_8Cl_2N_2O_4$ C 49.57, H 4.16, Cl 32.51, N 7.34, found: C 49.54, H 4.11, Cl 32.59, N 7.39.

Synthesis of 2-(1H-benzo[d]1,2,3-triazol-1-yl) - 2 - chloro - *N* - (substituted phenyl) acetamide (3, 4, 5, 6)

A mixture of 1H-benzo[d]1,2,3-triazole **(2)** (0.01 mol) and 2,2-dichloro -*N*-(substituted phenyl) acetamide (a_{1-4}) (0.01 mol) in the present of anhydrous K_2CO_3 (0.01 mol) in dry acetone (30ml) was heated under reflux for 24hrs. The solvent was evaporated and the residue was diluted with water. The solid obtained was filtered off, dried and crystallized from DMF.

2-(1H - benzo [d]1,2,3-triazol-1-yl) – 2 - chloro - *N*-(4 - chloro phenyl) acetamide (3).

Yield 65 %, m.p.136-139 ^{0}C, IR ($\bar{\upsilon}_{max}$, cm^{-1}):3278 (NH), 3135(CH- aromatic), 2991(CH- aliphatic) and 1678(CON). ^{1}HNMR (DMOS, δH, ppm): 6.6 (s, 1H, CH), 7.4 -7.7 (m,8H, aromatic-H) and 10.8 (s, 1H, NH). M/S, m/z (%) = 320(M^+, 0.2%), 154(M^+, $C_7H_5N_3Cl$, 6%), 126(M^+, $C_8H_5N_3ClO$, 15%) and 76(M^+, $C_8H_8N_4Cl_2O$, 100%). Anal. Calc. for $C_{14}H_{10}Cl_2N_4O$ C 52.36, H 3.14, Cl 22.08, N 17.45, found: C 52.39, H 3.18, Cl 22.04, N 17.42.

2-(1H-benzo[d]1,2,3 - triazol-1-yl) - 2 - chloro - *N* - (3-nitro phenyl) acetamide (4).

Yield 62 %, m.p. 105-108 ^{0}C, IR ($\bar{\upsilon}_{max}$, cm^{-1}): 3341(NH), 3018(CH- aromatic), 2991(CH-aliphatic) and 1693(CON). ^{1}HNMR (DMOS, δH, ppm): 6.6 (s, 1H, CHCl), 7.6 -8.6 (m,8H, aromatic-H) and 11.1 (s, 1H, NH). M/S, m/z (%) = 331(M^+, 0.2%), 165(M^+, $C_7H_5N_3Cl$, 26%), 137(M^+, $C_8H_5N_3ClO$, 5%), 91(M^+, $C_8H_5N_4ClO_3$, 13%) and 76(M^+, $C_8H_6N_5ClO_3$, 100%). Anal. Calc. for $C_{14}H_{10}ClN_5O_3$ C 50.69, H 3.04, Cl 10.69, N 21.11, found: C 50.63, H 3.08, Cl 10.71, N 21.15.

2 - (1H-benzo[d]1,2,3-triazol -1-yl) - 2 - chloro -*N* - (4 - methoxy - 3 - nitro phenyl) acetamide (5). Yield 70 %, m.p. 100-105 ^{0}C, IR($\bar{\upsilon}_{max}$, cm^{-1}): 3276(NH), 3016(CH- aromatic), 2990(CH-aliphatic) and 1691(CON). ^{1}HNMR (DMOS, δH, ppm): 3.9(s, 3H, $-OCH_3$), 6.7 (s, 1H, CHCl), 7.3-7.5 (m,7H, aromatic-H) and 10.8 (s, 1H, NH). M/S, m/z (%) = 361(M^+, 10%), 331(M^+, CH_3O, 27%), 195(M^+, $C_7H_7N_2O_3$, 15%), 78(M^+, $C_9H_9N_5ClO_4$, 13%) and 77(M^+, $C_9H_8N_5ClO_4$, 36%). Anal. Calc. for $C_{15}H_{12}ClN_5O_4$ C 49.80, H 3.34, Cl 9.80, N 17.69, found: C 49.84, H 3.36, Cl 9.75, N 17.67.

2-(1H-benzo[d]1,2,3-triazol-1-yl)-2-chloro - *N* - (p - tolyl) acetamide (6).

Yield 78 %, m.p.166-169 ^{0}C, IR ($\bar{\upsilon}_{max}$, cm^{-1}): 3242(NH), 3060(CH- aromatic), 2937(CH-aliphatic) and 1670(CON). ^{1}HNMR (DMOS, δH, ppm): 2.2 (s, 3H, CH_3), 6.6 (s, 1H, CHCl), 7.2 -7.5 (m,8H, aromatic-H) and 10.8 (s, 1H, NH). M/S, m/z (%) = 300(M^+, 0.2%), 134 (M^+, $C_7H_5N_3Cl$, 5%), 106(M^+, $C_8H_5N_3ClO$, 9%), 77(M^+, $C_9H_9N_4ClO$, 29%) and 76 (M^+, $C_9H_{10}N_4ClO$, 100%) Anal. Calc. for $C_{15}H_{13}ClN_4O$ C 49.80, H 3.34, Cl 9.80, N 17.69, found: C 49.83, H 3.31, Cl 9.84, N 17.66.

3 Results and Discussion

1H-benzo[d]1,2,3-triazole **(2)** was synthesized according to the reported method according to the reported method (Bashir et al., 2021) by diazotizing benzene 1,2-diamine with sodium nitrite and glacial acetic acid. However, by reacting substituted aniline with dichloroacetyl chloride in CH_2Cl_2, 2, 2-dichloro-*N*-(substituted phenyl) acetamide **(a_{1-4}) (scheme 1)** was produced. Strong absorption bands of the amidic carbonyl group were seen in compounds **(a_1)** and **(a_2)** at 1677 cm^{-1} and 1674 cm^{-1} respectively. However, absorption bands of the (-NH-) group were present in compounds **(a_1)** and **(a_2)** at 3276 cm^{-1} and 3342 cm^{-1}. The ^{1}HNMR spectra of compound **(a_1)** and compound **(a_2)** revealed singlet signals of the (-NH-) group at 10.8 ppm and 11.2 ppm, as well as singlet signals of the (-CHCl-) group at 6.6 ppm of both compounds **(a_1)** and **(a_2) (figure 1, 3)**. The chemical formulas $C_8H_6NCl_3O_3$ and $C_8H_6N_2Cl_2O_3$, respectively, were disclosed by the mass spectra of a_1 and a_2, which showed m/z 237 and 248 respectively. The compounds **(a_1)** and **(a_2)** showed amidic (-CONH-) carbonyl groups in their ^{13}CNMR spectra at 76.16 ppm and 67.86 ppm, respectively, as well as multiple signals (aromatic ring carbons) at 121.26 ppm and 128.37 ppm of compounds **(a_1)**. However, compound **(a_2)** showed a single peak from 113.9 ppm to 147.83 ppm that indicated its aromatic ring carbons **(figure 2, 4).**

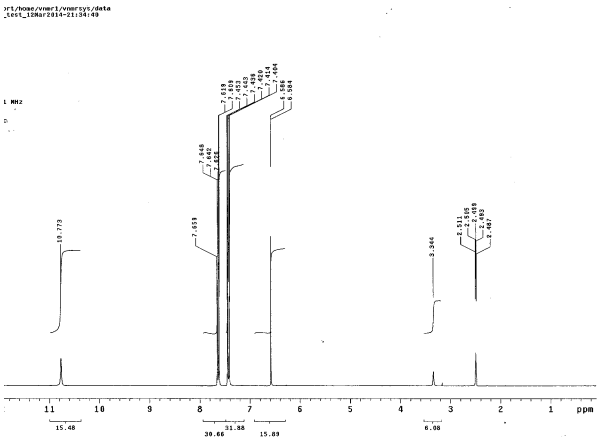


Figure (1): ^{1}HNMR spectrum of **(a_1)**

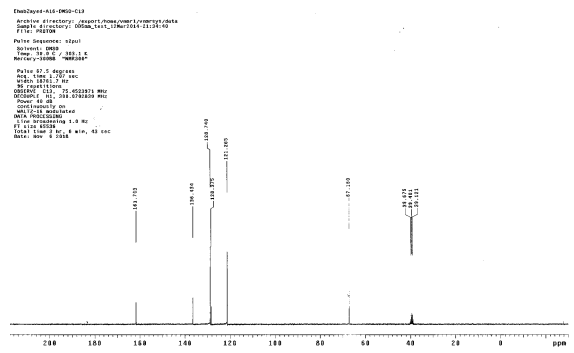


Figure (2): ^{13}CNMR spectrum of **(a_1)**

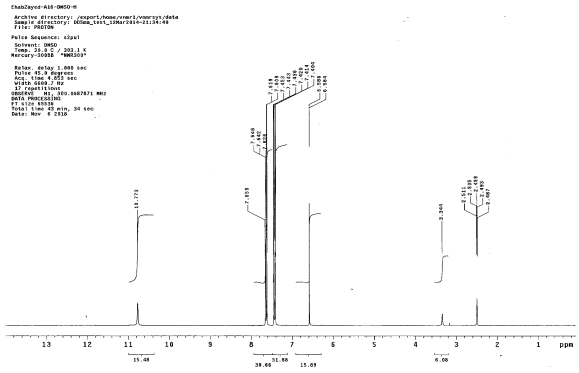


Figure (3): ^{1}HNMR spectrum of **(a_2)**

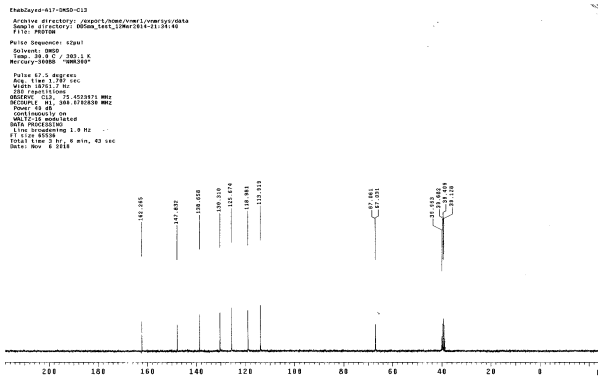


Figure (4): ^{13}CNMR spectrum of **(a_2)**

The ^{1}HNMR spectrum of compounds **(a_3)** and **(a_4)** revealed singlet signals of the amidic group at 10.71 ppm and 10.54 ppm, respectively, as well as singlet signals of the (- CHCl-) group at 6.71 ppm and 6.56 ppm. Compound **(a_3)** also showed a singlet signal of the methoxy group at 3.85 ppm, but compound **(a_4)** showed a singlet signal of the methyl group at 2.27 ppm **(figure 5)** and **(figure 6)**. Amidic groups have substantial absorption bands in the infrared spectrum of compounds **(a_3)** and **(a_4)** at 3277 cm^{-1} and 3242 cm^{-1}, however, amidic carbonyl groups exhibit absorption bands at 1691 cm^{-1} and 1670 cm^{-1}. The molecular ion peak at m/z 287 and m/z 217 in the mass spectrum of **(a_3)** and **(a_4)** was compatible with the molecular weight of compounds. The ^{13}CNMR spectrum of compound **(a_4)** spectrum revealed the anticipated number of carbonyl amidic, methyl, and (-CHCl-) signals at 161.40 ppm, 133.68 ppm, and 67.30 ppm **(figure 7)**. Both the (-CHCl-) group and the carbonyl amidic group were present in both compounds **(a_3)** and **(a_4)**, according to the mass spectrum and elemental analyses **(a_4)**.

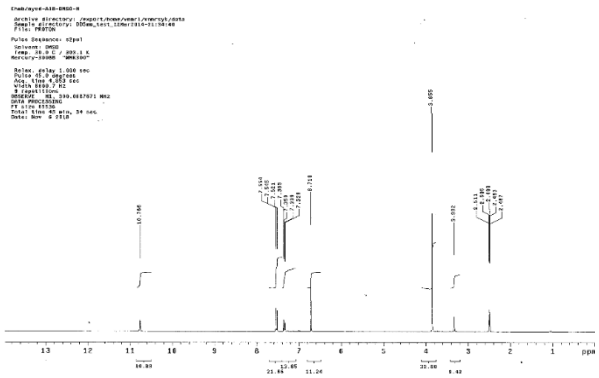


Figure (5): ^{1}HNMR spectrum of **(a_3)**

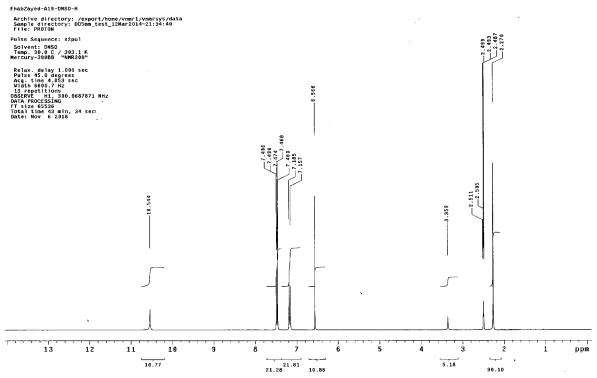


Figure (6): ^{1}HNMR spectrum of **(a_4)**

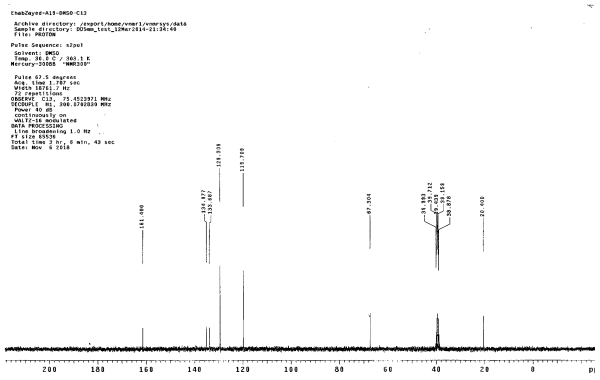


Figure (7): ^{13}CNMR spectrum of (**a$_4$**)

Boiling acetone with anhydrous K_2CO_3 and a mixture of 2, 2-dichloro-*N*-(substituted phenyl) acetamide **(a$_{1-4}$)** and 1H-benzo[d]1,2,3-triazole **(2)** produced 2, 2 - (1H-benzo[d]1,2,3- triazol-1-yl) -2 - chloro- *N* - (substituted phenyl) acetamide **(3, 4, 5, 6) (scheme 2)**. The ^{1}HNMR spectra of compounds **(3)** and **(4)** showed aromatic protons as well as (-CHCl-) protons as singlet signals at 6.6 ppm for both compounds and 10.80 ppm and 11.1 ppm for compounds **(3)** and **(4)** respectively **(figure 8, 9)**. In addition to the distinctive absorption band of the carbonyl amidic group at 1678 cm^{-1} and 1693 cm^{-1}, respectively, the IR spectrum of **(3)** and **(4)** also revealed the presence of NH group absorption bands at 3278 cm^{-1} and 3341 cm^{-1}. The structures of **(3)** and **(4)** were further supported by their mass spectra, which revealed fragmentation and subsequent elimination to produce the most stable base peaks (M^+, $C_8H_8NCl_2O$) and (M^+, $C_8H_6N_5ClO_3$) respectively.

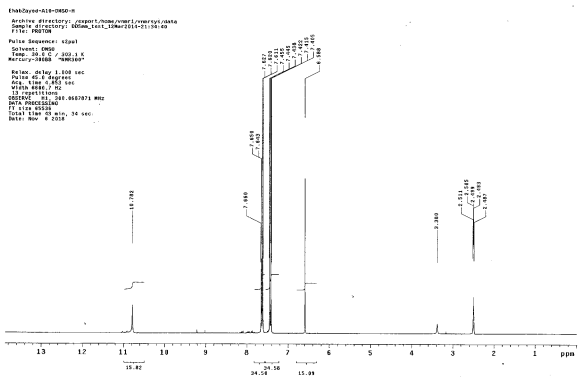


Figure (8): ^{1}HNMR spectrum of **(3)**

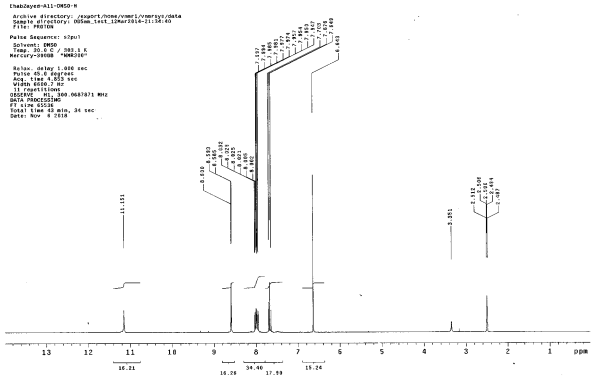


Figure (9): ^{1}HNMR spectrum of **(4)**

Its spectral research led to the inference of the structures of **(5)** and **(6)**. As a result, the (-NH-) group was found to have an absorption band at 3276 cm^{-1} and 3242 cm^{-1} in the IR spectrum of compounds **(5)** and **(6)**, whereas the (-CONH-) group was found to have an absorption band at 1691 cm^{-1} and 1670 cm^{-1} respectively. The ^{1}HNMR spectra of compounds **(5)** and **(6)** also revealed a characteristic singlet signal of (-NH-) at 10.78 ppm and 10.54 ppm, as well as singlet signals at 6.71 ppm and 6.56 ppm for (-CHCl-) group and singlet at 3.85 ppm for methoxy protons of compound **(5)**, as well as the methyl protons at 2.27 ppm of compound **(6) (figure 10, 11)**. The structures were also validated by the mass spectra of compounds **(5)** and **(6)**, which displayed their molecular ion peaks as base peaks.

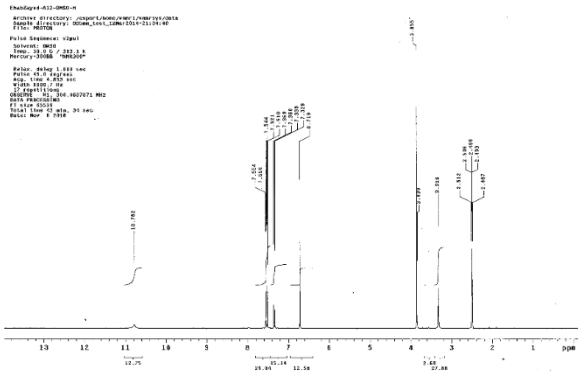


Figure (10): ^{1}HNMR spectrum of **(5)**

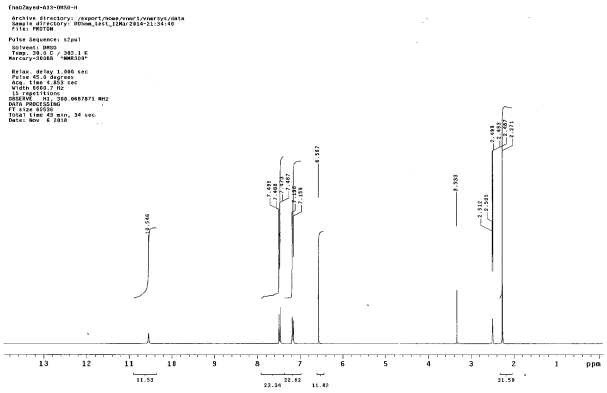
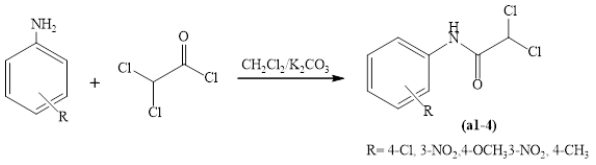
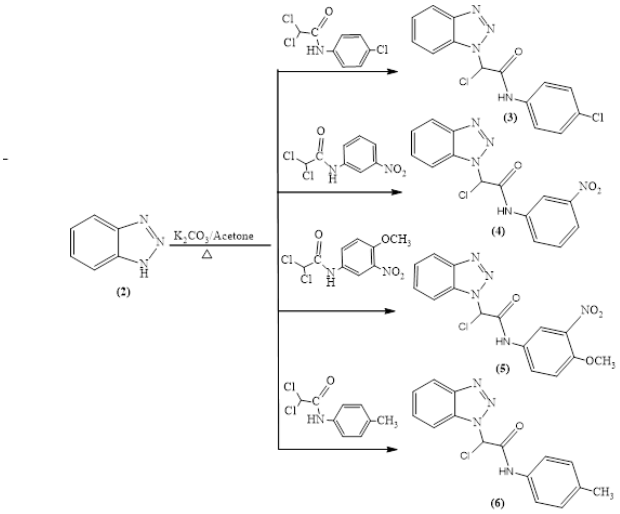


Figure (11): ^{1}HNMR spectrum of **(6)**



Scheme 1



Scheme 2

4 Conclusions

In this study we are reported synthesis of different heterocyclic derivatives from benzotriazole nucleus via reaction 1H-benzo[d]1,2,3-triazole **(2)** with 2,2-dichloro-N- (substituted phenyl) acetamid **(a_{1-6})** to give 2-(1H-benzo[d]1,2,3-triazol -1-yl) - 2 - chloro - *N*-(substituted phenyl) acetamide **(3 - 6)** in the present anhydrous K_2CO_3 in acetone. Furthermore, 2,2-dichloro-*N*-(substituted phenyl) acetamid **(a_{1-6})** were synthesized by reacting substituted aniline with dichloroacetyl chloride in the present of anhydrous K_2CO_3 in CH_2Cl_2. These derivatives confirmed from spectral data analysis (IR, ^{1}HNMR, ^{13}CNMR, Mass spectrometry).

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