



(2,4- DIOXO-1,4 - DIHYDRO - 2H - QUINAZOLIN - 3 - YL) - ACETIC ACID HYDRAZIDE: SYNTHESIS AND REACTIONS

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ABSTRACT

Pyrimidines are fused heterocycles that are reported to have a wide variety of biological and pharmaceutical activities. Our study focused on the synthesis of series of quinazolindione derivatives which contain other heterocyclic moieties such as oxadiazole, diazole and triazole. The elucidation of the chemical structures of the newly compounds has been supported by their spectral and physical data.

Keywords: Benzoin, Cyclohexanone, (2, 4-dioxo-1, 4-dihydro-2H-quinazolin-3-yl)-acetic acid hydrazide, Acetic anhydride, Fusion , Phenylisocyanate.

1. INTRODUCTION

Quinazoline and pyrimidine derivatives are incorporated in a wide variety of pharmaceuticals. In addition, Quinazoline and pyrimidine derivatives are attracting important applications in the field of medicinal chemistry; the pyrimidine ring is present in a large number of biological important compounds (Ostrowski *et al.*, 2000). Quinazolines represents one of the most important classes of heterocycles possessing wide spectrum of biological activities. Because of the important applications of pyrimidines and quinazolines derivatives, this encouraged us to carry out the synthesis of new derivatives of quinazolindiones starting from (2,4-dioxo-1,4-dihydro-2H-quinazolin-3-yl)-acetic acid hydrazide **2**. The newly synthesized compounds have been evaluated by their spectral data.

2. EXPERIMENTAL

2.1. Instrumentation

All melting points are uncorrected. The IR spectra (KBr) were recorded on a Shimadzu 408 spectrometer and carried out at the Central lab. of SVU, Egypt. The ¹H NMR spectra were recorded

using 300 MHz Varian EM 390 spectrometer; chemical shifts are reported in ppm with TMS as an internal standard and are given in δ units. Electron impact mass spectra were obtained at 70 eV with Shimadzu GC-MS (QP-2010 plus). ^{13}C NMR spectra were measured on a JEOL ECX instrument 400 MHz in $\text{DMSO-}d_6$ and carried out at Jacobs University Bremen, Germany. Elemental analyses were performed at the Microanalysis Unit - CU, Egypt. The purity of our newly compounds was detected by TLC.

3. SYNTHESIS

(2,4-Dioxo-1,4-dihydro-2H-quinazolin-3-yl)-acetic acid ethyl ester (1)

A mixture of *N*-phenylsulphonyloxyphthalimide (5 g, 0.016 mol) and glycine ethyl ester hydrochloride (2.99 g, 0.02 mol) in pyridine (20 mL) was refluxed for 9 hours. After cooling; the reaction mixture was acidified with cold dilute hydrochloric acid (1:1), and the solid formed was filtered off and dried. The target product was crystallized from benzene to give (2,4-dioxo-1,4-dihydro-2H-quinazolin-3-yl)-acetic acid ethyl ester **1** (Hassan *et al.*, 2013) (3.7 g, 0.15 mol) **1** as a gray crystal. Yield: 3.7 g, 90 %. M.p.: 206-208 °C. FT-IR (KBr, ν , cm^{-1}): 3380 (NH), 1719, 1671 (C=O ' s). ^1H NMR (300 MHz, $\text{DMSO-}d_6$, δ , ppm): 1.1 (t, 3H, CH_3), 4.1 (q, 2H, CH_2), 4.6 (s, 2H, N- CH_2), 7.2-7.9 (m, 4H, arom.), 11.6 (s, 1H, NH). MS (m/z , %): 248 (31.1 %) (M^+). The main fragmentation routes for compound **1** are shown in Scheme 2. Anal. calcd. for $\text{C}_{12}\text{H}_{12}\text{N}_2\text{O}_4$: C, 58.06; H, 4.87; N, 11.28. Found: C, 58.25; H, 4.89; N, 11.35%.

(2,4-dioxo-1,4-dihydro-2H-quinazolin-3-yl)-acetic acid hydrazide (2)

To a solution of (2,4-Dioxo-1,4-dihydro-2H-quinazolin-3-yl)-acetic acid ethyl ester **1** (2.5 g, 0.01 mol) in absolute ethanol (30 mL), 5 mL of hydrazine hydrate was added; the reaction mixture was refluxed for 8 hours. The reaction mixture was allowed to cool and the separated product was filtered and dried. Crystallization of the crude product with ethanol and acetic acid, afforded **2** as a white crystal. Yield 1.6 g, 62 %, m.p. > 300 °C. FT-IR (KBr, cm^{-1}): 3296 (NH), 3196 (NH_2), 1714, 1665 (C=O ' s). ^1H NMR spectrum: (300 MHz, $\text{DMSO-}d_6$): 4.5 (s, 2H, CH_2); 4.2 (s, 2H, NH_2); 7.1-7.9 (m, 4H, arom.); 9.2 (s, 1H, NH); 11.5 (s, 1H, NH). MS (m/z , %): 234 (20.1 %) (M^+). Anal. calcd. for $\text{C}_{10}\text{H}_{10}\text{N}_4\text{O}_3$: C, 51.28; H, 4.30; N, 23.92. Found: C, 51.49; H, 4.31; N, 23.98 %.

(2,4-Dioxo-1,4-dihydro-2H-quinazolin-3-yl)-acetic acid (2-hydroxy-1,2-diphenylethylidene)-hydrazide (3)

A mixture of (2,4-dioxo-1,4-dihydro-2H-quinazolin-3-yl)-acetic acid hydrazide **2** (0.6 g, 0.0025 mol) and benzoin (0.65 g, 0.003 mol) in ethanol (20 mL) and few drops of acetic acid was refluxed for 12 hours. The reaction mixture was allowed to stand overnight and the separated product was filtered and dried. Crystallization of the crude product with benzene and ethanol, afforded **3** as a grey crystal. Yield 0.5 g, 50 %, m.p. = 206 °C. FT-IR (KBr, cm^{-1}): 3290 (NH), 1700, 1665 (C=O ' s). ^1H NMR spectrum: (300 MHz, $\text{DMSO-}d_6$): 4.8 (s, 2H, CH_2); 5.01 (s, 1H, CH); 7.1-7.9 (m, 14H, arom.); 11.2 (s, 1H, NH); 11.5 (s, 1H, NH); 11.5 (s, 1H, OH). MS (m/z , %): 428 (0.13 %) (M^+). Anal. calcd. for $\text{C}_{24}\text{H}_{20}\text{N}_4\text{O}_4$: C, 67.28; H, 4.71; N, 13.08. Found: C, 67.36; H, 4.72; N, 13.27 %.

(2,4-Dioxo-1,4-dihydro-2H-quinazolin-3-yl)-acetic acid cyclohexylidene-hydrazide (4)

To a solution of compound **2** (0.5 g, 0.002 mol) in ethanol (20 mL), (0.6 mL, 0.006 mol) of cyclohexanone was added; the reaction mixture was refluxed for 10 hours. After cooling; the reaction mixture was cool and the separated product was filtered and dried. Crystallization of the crude product with benzene and ethanol, afforded **4** as a white crystal. Yield 0.5 g, 74 %, m.p. = 290 °C. FT-IR (KBr, cm^{-1}): 3203 (NH), 1715, 1660 (C=O' s). ^1H NMR spectrum: (300 MHz, DMSO-d_6): 1.5-2.5 (m, 10H, 5CH_2); 4.8 (s, 2H, CH_2); 7.2-7.9 (m, 4H, arom.); 10.5 (s, 1H, NH); 11.4 (s, 1H, NH). MS (m/z , %): 314 (29.81 %) (M^+). Anal. calcd. for $\text{C}_{16}\text{H}_{18}\text{N}_4\text{O}_3$: C, 61.14; H, 5.77; N, 23.92. Found: C, 61.44; H, 5.78; N, 24.03 %.

Dimethylamino-acetic acid N'-[2-(2,4-dioxo-1,4-dihydro-2H-quinazolin-3-yl)-acetyl]-hydrazide (5)

Chloroacetyl chloride (0.5 mL, 0.004 mol) was added dropwisely with stirring in ice bath to a solution of compound **2** (0.5 g, 0.002 mol) in 20 mL DMF. The reaction mixture was vigorously stirred for 2 hours. The solid formed was poured into cold water, and then the solid formed was filtered off and dried. The target product was crystallized from dioxane to give compound **4** as yellowish-white precipitate. Yield 0.5 g, 75 %, m.p. = 180 °C. FT-IR (KBr, cm^{-1}): 3200 (NH), 1739, 1638 (C=O' s). ^1H NMR spectrum: (300 MHz, DMSO-d_6): 2.7 (s, 3H, CH_3); 2.8 (s, 3H, CH_3); 4.1 (s, 2H, CH_2); 4.6 (s, 2H, CH_2); 7.1-7.9 (m, 4H, arom.); 10.3 (s, 2H, 2 NH); 11.5 (s, 1H, NH). MS (m/z , %): 319 (47.89 %) (M^+). Anal. calcd. for $\text{C}_{14}\text{H}_{17}\text{N}_5\text{O}_4$: C, 52.66; H, 5.37; N, 21.93. Found: C, 52.72; H, 5.38; N, 22.18 %.

(2,4-Dioxo-1,4-dihydro-2H-quinazolin-3-yl)-acetyl azide (6)

To a cold suspension of compound **2** (1 g, 0.004 mol) in acetic acid (30 mL), a cold solution of sodium nitrite (0.88 g, 0.01 mol) in water (10 mL) was added dropwise with stirring. Stirring was continued for 2 hours at room temperature. The solid product thus formed was filtered, washed with water, dried and recrystallized from ethanol to afford compound **6** as white crystals. Yield 0.7 g, 70 %, m.p. = 110 °C. FT-IR (KBr, cm^{-1}): 2200 (N_3), 1739, 1638 (C=O' s). ^1H NMR spectrum: (300 MHz, DMSO-d_6): 4.1 (s, 2H, CH_2); 7.1-7.9 (m, 4H, arom.); 10.3 (s, 1H, NH). MS (m/z , %): 245 (5.3 %) (M^+). Anal. calcd. for $\text{C}_{10}\text{H}_7\text{N}_5\text{O}_3$: C, 48.99; H, 2.88; N, 28.56. Found: C, 49.08; H, 2.89; N, 28.88 %.

3-(5-Phenyl-[1,3,4]oxadiazol-2-ylmethyl)-1H-quinazoline-2,4-dione (7)

A mixture of compound **2** (0.5 g, 0.002 mol) and benzoic acid (0.2 g, 0.0016 mol) and 0.5 mL of phosphorous oxy chloride was refluxed for 4hours. After cooling, the reaction mixture was poured into ice-water and made basic by adding saturated solution of sodium bicarbonate. The precipitate was collected by filtration, washed with cold water and recrystallized from ethanol to give compound **7** as brown crystals. Yield 0.4 g, 60 %, m.p. > 300 °C. FT-IR (KBr, cm^{-1}): 3100 (NH), 1740, 1630 (C=O' s). MS (m/z , %): 320(17.9 %) (M^+). Anal. calcd. for $\text{C}_{17}\text{H}_{12}\text{N}_4\text{O}_3$: C, 63.75; H, 3.78; N, 17.49. Found: C, 63.78; H, 3.8; N, 17.70 %.

1-[(2,4-Dioxo-1,4-dihydro-2H-quinazolin-3-yl)-methylcarbonyl]-4-phenylaminourea (8)

A mixture of compound **2** (0.4 g, 0.002 mol) and phenylisocyanate (0.25 ml, 0.002 mol) in dry pyridine (20 mL) was refluxed for 8 hours. The reaction mixture was allowed to stand overnight and the separated product was filtered and dried. Crystallization of the crude product with dioxane, afforded **8** as a white crystal. Yield 0.5 g, 69 %, m.p. = 290 °C. FT-IR (KBr, cm^{-1}): 3198 (NH), 3196 (ν NH_2), 1740, 1637 ($\text{C}=\text{O}$ s). ^1H NMR spectrum: (300 MHz, DMSO-d_6): 4.5 (s, 2H, CH_2); 7.1-7.9 (m, 9H, arom.); 9.2 (s, 1H, NH); 10.2 (s, 1H, NH); 10.5 (s, 1H, NH); 11.5 (s, 1H, NH). MS (m/z , %): 353 (4.68 %) (M^+). Anal. calcd. for $\text{C}_{17}\text{H}_{15}\text{N}_5\text{O}_4$: C, 57.79; H, 4.28; N, 19.82. Found: C, 57.88; H, 4.29; N, 20.03%.

3-(5-Oxo-6,7-dihydro-5H-pyrrolo[2,1-c][1,2,4]triazol-3-ylmethyl)-1H-quinazoline-2,4-dione (9)

Heating of the hydrazide **2** (1 g, 0.004 mol) with phthalimide (0.42 g, 0.004 mol) in DMF (20 mL) under reflux for 12 hours gave after cooling a solid product which was filtered off and crystallized from benzene to give compound **9** as yellow crystals. Yield 0.5 g, 79 %, m.p. > 300 °C. FT-IR (KBr, cm^{-1}): 3200 (NH), 1700, 1625 ($\text{C}=\text{O}$ s). MS (m/z , %): 297 (1.9 %) (M^+). Anal. calcd. for $\text{C}_{14}\text{H}_{11}\text{N}_5\text{O}_3$: C, 56.57; H, 3.73; N, 23.56. Found: C, 56.77; H, 3.75; N, 23.85 %.

(2,4-Dioxo-1,4-dihydro-2H-quinazolin-3-yl)-acetic acid N'-(2-oxo-2-phenyl-ethyl)-hydrazide (10)

A mixture of compound **2** (0.5 g, 0.002 mol) and bromoacetophenone (0.51 g, 0.005 mol) in ethanol (20 mL) was refluxed for 10 hours. The reaction mixture was allowed to cool and the separated product was filtered and dried. Crystallization of the crude product with acetic acid to give compound **10** as a white crystal. Yield 0.48 g, 64 %, m.p. = 254 °C. FT-IR (KBr, cm^{-1}): 3304 (NH), 1741, 1642 ($\text{C}=\text{O}$ s). ^1H NMR spectrum: (300 MHz, DMSO-d_6): 4.1 (s, 2H, CH_2); 4.6 (s, 2H, CH_2); 7.1-7.9 (m, 4H, arom.); 10.2 (s, 1H, NH); 11.5 (s, 2H, 2NH). MS (m/z , %): 352 (0.02 %) (M^+). Anal. calcd. for $\text{C}_{18}\text{H}_{16}\text{N}_4\text{O}_4$: C, 61.36; H, 4.58; N, 15.90. Found: C, 61.46; H, 4.59; N, 16.18 %.

Acetic acid N'-[2-(2,4-dioxo-1,4-dihydro-2H-quinazolin-3-yl)-acetyl]-hydrazide (11a) and 3-(5-Methyl-[1,3,4]oxadiazol-2-ylmethyl)-1H-quinazoline-2,4-dione (11b)

A solution of compound **2** (1g, 0.004 mol) with acetic anhydride (20 mL) was heated under reflux for 12 hours. After cooling; the reaction mixture was poured into crushed ice to give white precipitate which collected by filtration then crystallized from benzene to give mixture of compound **11a** as open form and compound **11b** as a cyclic form which confirmed by the ^1H -NMR, MS and ^{13}C NMR spectral data that will be shown. Separation of this mixture using column chromatography, HPLC and preparative TLC was not possible owing to their comparable R_F values. Yield 0.8 g, 66 %, m.p. = 196 °C. FT-IR (KBr, cm^{-1}): 3196 (NH), 1722, 1693 ($\text{C}=\text{O}$ s). ^1H NMR spectrum: (300 MHz, DMSO-d_6): 2.2 (s, 3H, CH_3 , open form); 2.5 (s, 3H, CH_3 , cyclic form); 4.9 (s, 2H, CH_2); 5.0 (s, 2H, CH_2); 7.2-7.9 (m, 4H, arom.); 10.3 (s, 1H, NH); 10.7 (s, 1H,

NH); 11.5 (s, 1H, NH); 11.6 (s, 1H, NH). MS (m/z , %): 276 (1.02 %) (M^+) and 258 (18.26 %). Anal. calcd. for $C_{12}H_{12}N_4O_4$: C, 52.17; H, 4.38; N, 20.28. Found: C, 52.30; H, 4.39; N, 20.39 %.

3-(5-Methyl-[1,3,4]oxadiazol-2-ylmethyl)-1H-quinazoline-2,4-dione (11b)

Compound **11b** was obtained from compound **11a** (0.5 g) which underwent to ring closure fusion in oil bath at 200 °C for 4 hours to give compound **11b** as a white crystal. Yield 0.3 g, 60 %, m.p. = 230 °C. FT-IR (KBr, cm^{-1}): 3193 (NH), 1715, 1655 (C=O' s). 1H NMR spectrum: (300 MHz, DMSO- d_6): 2.6 (s, 3H, CH_3); 4.2 (s, 2H, CH_2); 7.1-7.9 (m, 4H, arom.); 11.5 (s, 1H, NH). MS (m/z , %): 258 (100 %) (M^+). Anal. calcd. for $C_{12}H_{10}N_4O_3$: C, 55.81; H, 3.90; N, 21.70. Found: C, 55.92; H, 3.92; N, 21.91 %.

3-[2-(3,5-Dimethyl-pyrazol-1-yl)-2-oxo-ethyl]-1H-quinazoline-2,4-dione (12)

(2,4-dioxo-1,4-dihydro-2H-quinazolin-3-yl)-acetic acid hydrazide **2** (0.6 g, 0.0025 mol) was added to acetyl acetone (0.5 mL) in absolute ethanol (30 mL) in presence of few drops of Piperidine, the reaction mixture was heated under reflux for 10 hours, the solvent was evaporated under vacuo, the resulting formed solid was collected and crystallized from benzene and ethanol to give compound **12** as yellow crystals. Yield 0.5 g, 79 %, m.p. = 244 °C. FT-IR (KBr, cm^{-1}): 3145 (NH), 1715, 1624 (C=O' s). 1H NMR spectrum: (300 MHz, DMSO- d_6): 1.7 (s, 3H, CH_3); 2.0 (s, 3H, CH_3); 4.8 (s, 2H, CH_2); 6.3 (s, 1H, CH); 7.2-7.9 (m, 4H, arom.); 11.4 (s, 1H, NH). ^{13}C NMR (400 MHz, DMSO- d_6 , δ , ppm): 16.5, 26.3, 43.2, 52.4, 90.9, 114, 115.7, 123.8, 128, 135.7, 140, 150.5, 156.1, 162.3, 164.2. MS (m/z , %): 298 (11.60 %) (M^+). Anal. calcd. for $C_{15}H_{14}N_4O_3$: C, 60.40; H, 4.73; N, 18.78. Found: C, 60.44; H, 4.74; N, 18.98 %.

4. RESULTS AND DISCUSSION

In conjugation to our studies (Hassan *et al.*, 2010; Hassan *et al.*, 2013) on the synthesis and investigation of the chemical and biological activities of quinazoline derivatives, the development of expedient methods for the synthesis of new quinazoline derivatives, were implemented, through, the reaction of (2,4-dioxo-1,4-dihydro-2H-quinazolin-3-yl)-acetic acid hydrazide **2** with different reagents in different conditions. Firstly, (2,4-Dioxo-1,4-dihydro-2H-quinazolin-3-yl)-acetic acid ethyl ester **1** resulted from the reaction between glycine ethyl ester hydrochloride and *N*-phenylsulphonyloxypthalimide (scheme 1). Scheme 2 outlines the main fragmentation routes for compound **1**. The starting compound **2** namely (2,4-dioxo-1,4-dihydro-2H-quinazolin-3-yl)-acetic acid hydrazide was prepared by treatment of (2,4-Dioxo-1,4-dihydro-2H-quinazolin-3-yl)-acetic acid ethyl ester **1** (Hassan *et al.*, 2013) with hydrazine in absolute ethanol and reflux for 8 hours (scheme 1). As detailed in scheme 3, treatment of compound **2** with benzoin gave compound **3**, which identified as (2,4-Dioxo-1,4-dihydro-2H-quinazolin-3-yl)-acetic acid (2-hydroxy-1,2-diphenyl-ethylidene)-hydrazide (scheme 3). Hydrazide **2** underwent facile condensation reaction with cyclohexanone in ethanol giving (2,4-Dioxo-1,4-dihydro-2H-quinazolin-3-yl)-acetic acid cyclohexylidene-hydrazide **4** (scheme 3). The reaction of compound **2** with chloroacetyl chloride gave dimethylamino-acetic acid *N'*-[2-(2,4-dioxo-1,4-dihydro-2H-quinazolin-3-yl)-acetyl]-hydrazide **5** (scheme 3). Treatment of compound **2** with sodium nitrite in acetic acid yielded (2,4-

Dioxo-1,4-dihydro-2H-quinazolin-3-yl)-acetyl azide **6** as shown in scheme 3. In scheme 4, we notice that, (5-Phenyl-[1,3,4]oxadiazol-2-ylmethyl)-1H-quinazoline-2,4-dione **7** was obtained by reaction of hydrazide **2** with benzoic acid in presence of phosphorous oxychloride. Scheme 5, shows the mechanism (Abdullah and Waldron, 2004) for obtain compound **7**. According to the scheme 5, the intermediate (II) will be formed through the reaction of acid chloride (I) with hydrazide **2**. The reaction proceeds through an ordinary nucleophilic substitution of the electron pair of N-2 atom of the hydrazide to the acid chloride carbonyl group. Finally, cyclodehydration process of the intermediate (II) will take place by its reaction with phosphorous oxychloride to give the oxadiazole. Also, (2,4-dioxo-1,4-dihydro-2H-quinazolin-3-yl)-acetic acid hydrazide, **2**, was subjected to the reaction with phenylisocyanate to give 1-[(2,4-Dioxo-1,4-dihydro-2H-quinazolin-3-yl)-methylcarbonyl]-4-phenylaminourea **8** (scheme 4). Treatment of hydrazide **2** with succinimide in DMF gave (5-Oxo-6,7-dihydro-5H-pyrrolo[2,1-c][1,2,4]triazol-3-ylmethyl)-1H-quinazoline-2,4-dione **9** (scheme 4). Scheme 6 outlines the synthetic pathway used to synthesis of compound **9**. Heating under reflux a mixture of compound **2** with bromo acetophenone afforded (2,4-Dioxo-1,4-dihydro-2H-quinazolin-3-yl)-acetic acid N'-(2-oxo-2-phenyl-ethyl)-hydrazide **10** (scheme 4). In scheme 7, Refluxing of compound **2** with acetic anhydride give mixture of acetic acid N'-[2-(2,4-dioxo-1,4-dihydro-2H-quinazolin-3-yl)-acetyl]-hydrazide **11a** as open form and 3-(5-Methyl-[1,3,4]-oxadiazol-2-ylmethyl)-1H-quinazoline-2,4-dione **11b** as cyclic form. The elucidation of the chemical structure of mixture **11a** and **11b** was corroborated by its spectroscopic data as in experimental section. Next, fusion of this mixture in oil bath at 200 °C gives compound **11b**. Our research work was finally extended to study the reaction of hydrazide **2** with acetyl acetone in ethanol and the presence of Pipridine under reflux gave 3-[2-(3,5-Dimethyl-pyrazol-1-yl)-2-oxo-ethyl]-1H-quinazoline-2,4-dione **12** (scheme 7). The structures of the obtained compounds were confirmed by FT-IR, NMR and MS.

5. CONCLUSIONS

In the present work, our study focused on the utility of hydrazide as a key intermediate for the synthesis of several derivatives of quinazolidione. We have used simple and convenient methods for synthesis of oxadiazole, diazole and triazole moieties attached to pyrimidine ring. The structures of the obtained compounds were elucidated by spectral and physical data.

6. ACKNOWLEDGMENT

The authors are grateful to the spirit of Prof. Dr. Youssef Hassan Ebeed, Faculty of Science, South Valley University, and Ibrahim Mohamed Ismael for his help in analysis of some samples.

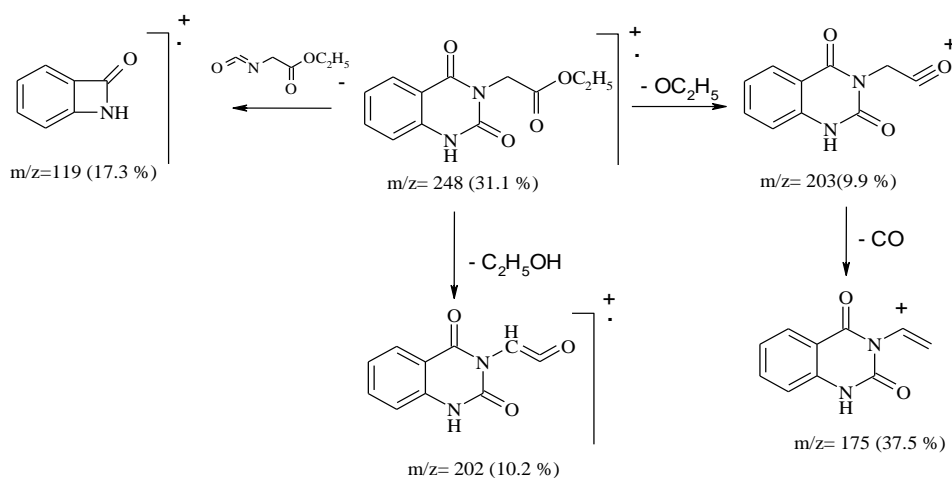
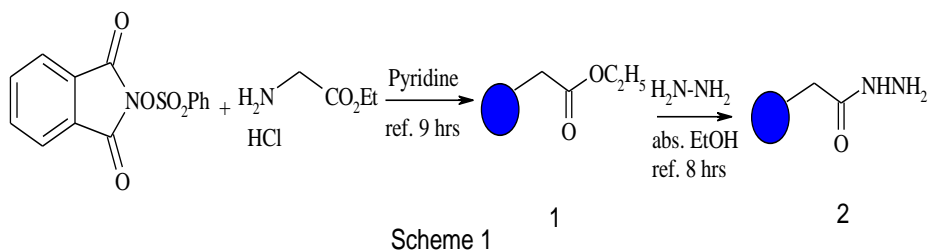
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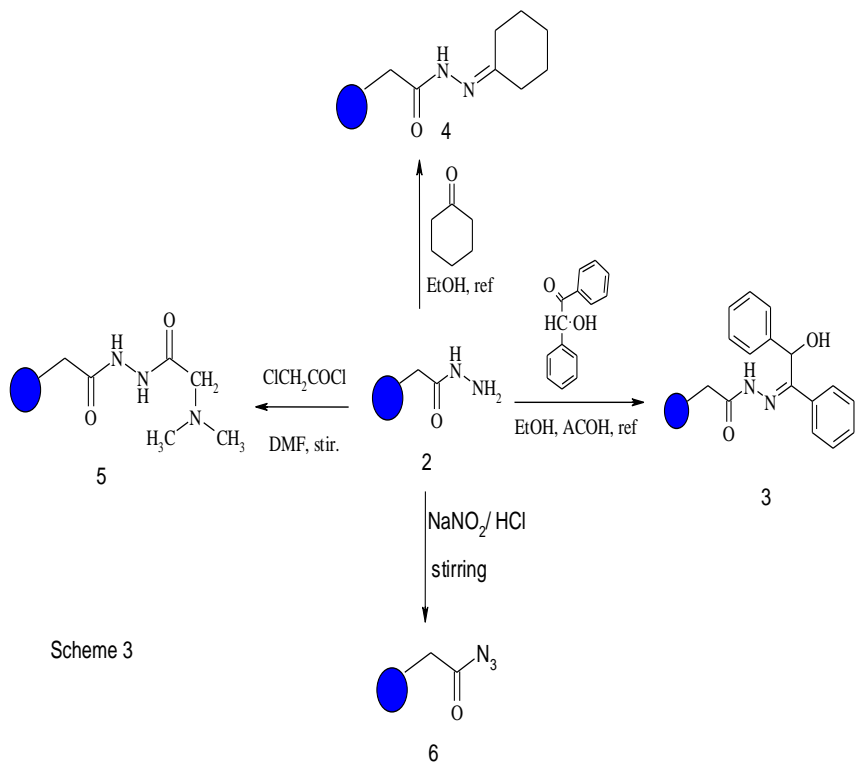
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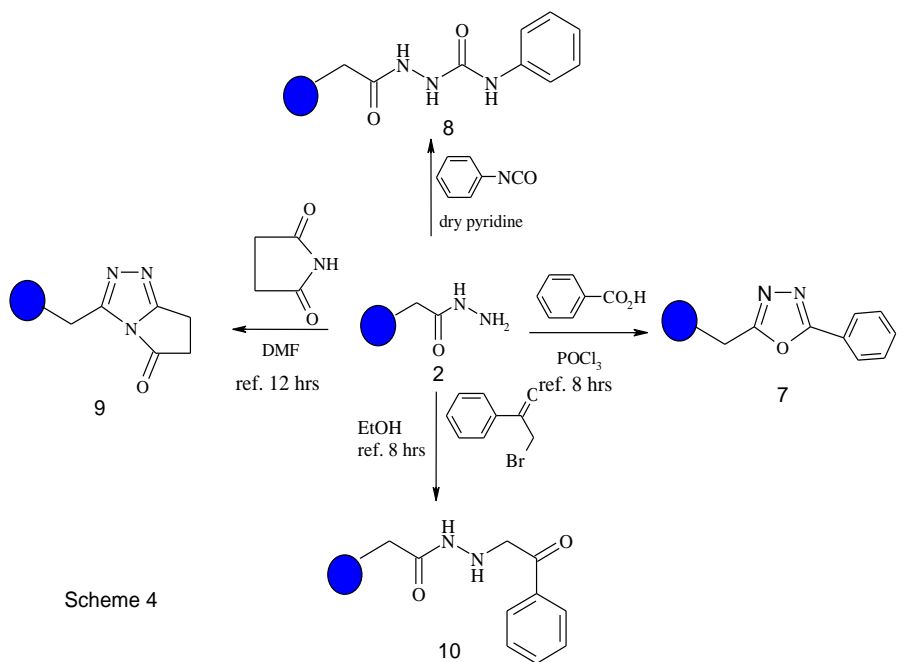
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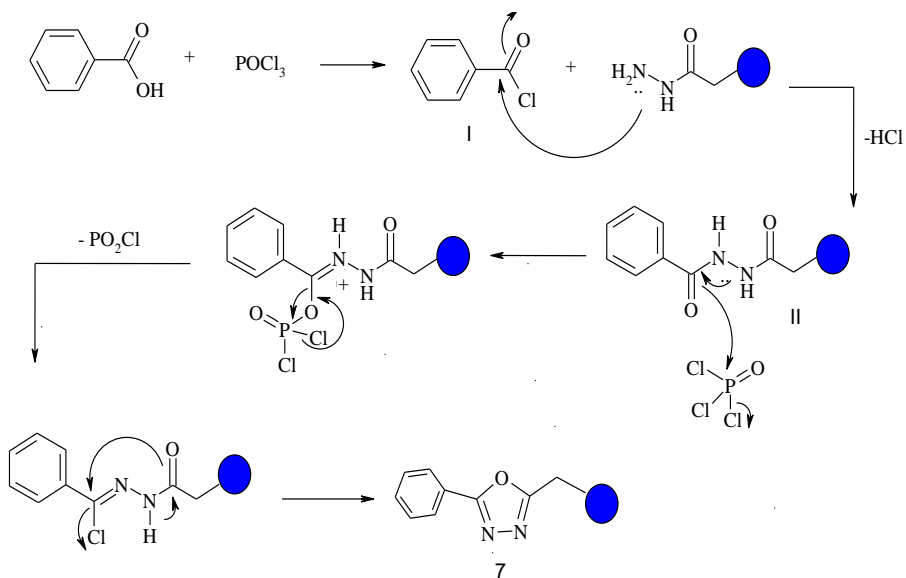




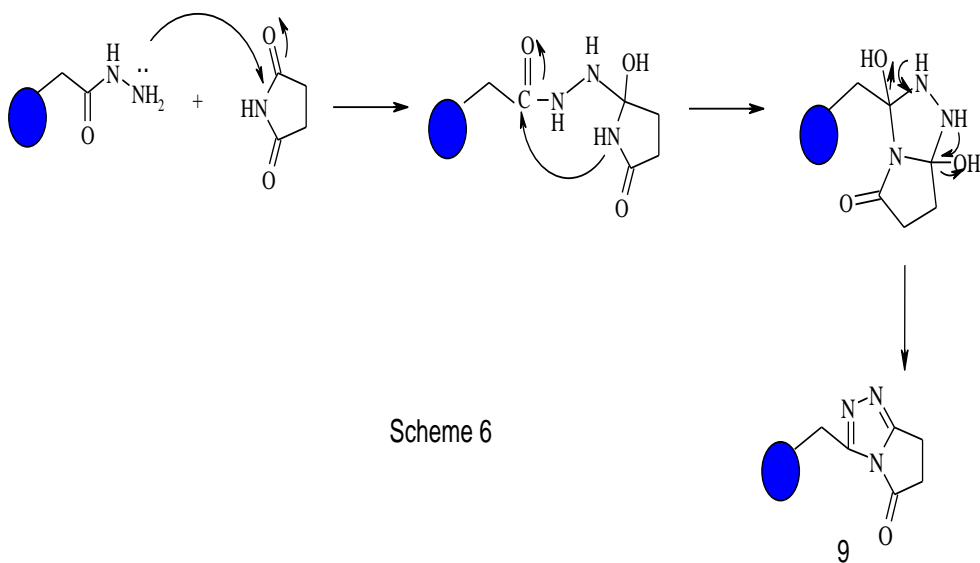
Scheme 3



Scheme 4



Scheme 5



Scheme 6

