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Full Length Research Paper

Synthesis of Azalactones involving the use of sodium acetate, anhydrous, KPO4 and calcium acetate

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Azalactone 1 can be synthesized via treatment of 4- chlorobenzoyl glycine with p-anisaldehyde in the presence of acetic anhydride and sod Acetate. The behaviour of azalactone 1 towards nitrogen nucleophiles, for example, anthranilic acid, 6-aminopyrimidin-2,4-dione and 2-amino-6-phenyl 1,3,4-thiadiazole can be investigated. Benzoxazone 2 can be treated with hydrazine hydrate, hydroxylamine hydrochloride, and o-phenylenediamine in different reaction conditions to afford quinazolinone derivatives 3, 4, 5, 6. The imidazoloquinazolinone derivative 6 can be treated with hydrazine derivatives. Pyrimidino/thiadiazolopyrimidine 8, 11 can be treated with acetic anhydride to increase their biological activities. The structures of all synthesized compounds were confirmed from analytical as well as spectral data.

Key words: Azalactone, benzoxazinone, quinazolinone, fused pyrimidinopyrimidine, 1,3,4-thiadiazolopyrimidine, Schiff's base.

INTRODUCTION

Azalactones have been used in a wide variety of reactions as precursors for biologically active molecules (Holla et al., 1996; Kidwai et al., 1998), herbicides and fungicides (Jeschkeit et al., 1989; Bakos et al., 1987), pesticide and agrochemical intermediates (Augustin et al., 1988), anti-hypertensives and irradiation with microwaves (Sosale et al., 2007) and in the asymmetric synthesis of amino acids (Chandrasekhar and Karri, 2006). Synthesis of oxazolone involves the condensation of aromatic aldehydes and hippuric acid with a stoichiometric amount of fused sodium acetate in the presence of acetic anhydrides; as the dehydrating agent, this reaction is known as the Erlenmeyer Plöchl reaction (Plöchl, 1893, 1884; Erlenmeyer, 1893; Flavio et al., 2010).

In literature, numbers of methods are reported for the synthesis of azalactones (Carter et al., 1947; Mohammed, 2003; Adolf et al., 1925; Buck and Ide, 1932; Karrer and Bussman, 2004) involving the use of sodium acetate (Clearly et al., 2010), anhydrous zinc chloride (Monk et al., 2000; Mohammed, 2009), alumina (Conway et al., 2009), KPO4 (Clearly et al., 2010: 625) and calcium acetate (Paul et al., 2004). In the recent example, assisted synthesis of azalactone is also reported (Suman et al., 2011; Patil et al., 2011). Many studies have also been focused on the synthesis of benzoxazin-4-one and quinazolin-4-one and their derivatives since they possess significant activities as antifungal (Lopez et al., 2001; Farghaly and Moharram, 1999), antibacterial, and antimiotic anticancer activity. In the present investigation, new benzoxazin-4-one and quinazolin-4-one derivatives were prepared.

EXPERIMENTAL

All melting points were uncorrected. IR spectra were recorded in Pye-Unicam SP 1200 spectrophotometer using KBr Wafer technique. The H^1-NMR spectra were determined on Varian Gemini 200 MHz, using TMS as internal standard (chemical shifts in δ-scale). EI-MS were measured on Shimadzu-GC-MS operating at 70 eV. Elemental analysis was carried out at the Micro-analytical Center at Cairo University. TLC on silica gel plates (Merk 60,F254) was used to monitor the reaction and for testing the purity of the products.

4-(4-Methoxybenzylidene)- 2-(2-chlorophenyl)oxazol-5-one (1)

A mixture of hippuric acid (0.01 mole), naphthaldehyde

(0.01 mole), sodium acetate (anhydrous) (0.03 moles) and acetic anhydride was heated on a water bath for two hours. The reaction mixture was cooled and poured into cold water to separate (1) m.p.166-167°C (60% yield) which was filtered off and crystallized from ethanol. IR (γcm^{-1}):1770 (C=O), 1636 (C=N).Anal.\Cald. for $C_{20}H_{13}NO_2$,(299):C,80.3;H,4.3;N,4.7.Found:C,80.7;H,4.4;N,4.3.

2(Z\E)2-(α-2-chlorobenzamido-β-(4-methoxyphenyl)vinyl)-4H-3,1-benzoxazin-4-one (2)

A mixture of (1) (0.01mole) and anthranilic acid (0.01 mole) was refluxed in 20 ml of acetic acid for 6 h, cooled and poured into cold water.

A yellow ppt. was formed, m.p.219 - 220°C (75% yield) and crystallized from benzene. Treatment of the latter compounds (0.01 mole) with acetic anhydride (25 ml) was refluxed at 150 - 170°C using “Water Separator System” for one hour.

The mixture was left under hood system for half an hour, a yellow solid was separated, filtered off and crystallized from pet.ether giving (3) m.p.156 - 157°C (90% yield). IR(γcm^{-1}):3300-3200(NH),1750-1710(C=O),1630-1590(C=N),1130,1150(C-O).Anal\Cald for $C_{24}H_{17}N_2O_4$,Cl (418):C,77.5;H,4.3;N,6.65.Found:C,77.6;H,4.1;N,6.5.

2(Z\E)2-(α-2-chlorobenzamido-β-(4-methoxyphenyl)vinyl)3-amino-4H-3,1-quinazolin-4-one (3)

A solution of (2)(0.01mole) and hydrazine hydrate (0.01 mole) in 50 ml ethanol was refluxed for 3 h. A yellow solid was separated (80% yield), m.p.186 - 188°C and crystallized from dioxane.

IR(γcm^{-1}):3600-3200(NH),1699-1655(C=O),1560(C=N);H^1-NMR(DMSO d_6)δ(ppm):10.9-9(s,2H,enolic form),9.7(s,1H,NH exchangeable with D_2O),8.9-7.4(m,16H,aromatic protons), 3,4(s,3H,OCH_3),4.1(s,2H,NH_2 exchangeable with D_2O).Anal.\Calcd for $C_{24}H_{19}N_4O_3Cl$(432): C,75;H,4.6;N,12.9.Found:C,75.3;H,5;N,12.9.

2(Z\E)2-(α-2-chlorobenzamido-β-(4-methoxyphenyl)vinyl)3-hydroxy-4H-3,1-quinazolin-4-one (4)

A solution of (2)(0.01 mole) and hydroxylamine hydrochloride (0.015 mole) in 30 ml ethyl alcohol was heated under reflux for 3 h. An orange solid was formed, crystallized from benzene (90% yield) and has m.p.164 - 166°C. IR (γcm^{-1}):3700-3200(NH),1689,1645(C=O),1578(C=N).Anal.\Calcd for $C_{24}H_{18}N_3O_4Cl$(433):C,74.8;H,4.4;N,9.7.Found:C,74,6;H4.6;N,9.9.

2-((2-Chlorophenyl-5-methoxyphenyl)-2,5-dihydroxazol-4-yl)-3-amino-4H-3,1-quinazolin-4-one (5a)

A solution of (3)(0.01mole) and hydrazine hydrate (0.01 mole) in 50 ml n-butanol was refluxed for 4 h. The solid was separated (70% yield), m.p.130 - 132°C and crystallized from diethyl ether. IR(γcm^{-1}):3600-3200(NH),1699-1655(C=O),1560(C=N);H^1-NMR(DMSO d_6)δ(ppm):10.9-9(s,2H,enolic form),9.7(s,1H,NH exchangeable with D_2O),8.9-7.4(m,16H,aromatic protons), 3,4(s,3H,OCH_3),4.1(s,2H,NH_2 exchangeable with D_2O).Anal.\Calcd for $C_{24}H_{19}N_4O_3Cl$(432): C,75;H,4.6;N,12.9.Found:C,75.3;H,5;N,12.9.

2-((2-Chlorophenyl-5-methoxyphenyl)-2,5-dihydroxazol-4-yl)-3-hydroxy-4H-3,1-quinazolin-4-one (5b)

A solution of (4)(0.01 mole) and hydroxylamine hydrochloride (0.015 mole) in 30 ml ethyl alcohol was heated under reflux for 3 h. An orange solid was formed, crystallized from benzene (90% yield) and has m.p.165 - 166°C. IR (γcm^{-1}):3700-3200(NH), 1689,1645(C=O),1578(C=N). H^1-NMR(DMSO d_6)δ(ppm):10.9-9(s,2H,enolic form),9.7(s,1H,OH exchangeable with D_2O),8.9-7.4(m,12H,aromatic protons),4.1(m,3H,CH_2CH) 3,4(s,3H, OCH_3),.Anal.\Calcd for $C_{24}H_{18}N_3O_4$ Cl(433):C,74.8;H,4.4;N,9.7. Found: C,74,6;H4.6;N,9.9.

2(Z\E)-(4-Methoxyphenyl-1-benzo[d]-imidazo[1,2-c]quinazolin-6-yl)ethen-1-yl-4-cholorobenzamide (6)

A mixture of (2) (0.01 mole) and o-phenylenediamine (0.01 mole) was fused in an oil bath at 150 - 160°C for about 4 h. The obtained brown solid was crystallized from pet.ether m.p.178 - 180°C (85% yield). IR(γ cm^{-1}):3652-3250(NH),1657(C=O),1597(C=N).^{1}H-NMR(DMSO d_6)δ(ppm): 10.1(s,1H, NHexchangeable with D_2O)8.2-7.1(m,20 H,aromatic protons), 3,4(s,3H,OCH_3),2.5(s,1H,CH=C).MSm|z(%):494$M^{+\cdot}$(23%) ,272(25%),151(32%),145(36%)105(34%),68(79)56(100). Anal.\Calcd for $C_{30}H_{21}N_4O_2Cl$,(500): C,80.8;H,4.5;N,11.4 .Found :C,80.7;H,4.7;N,11.2.

2-(3-(4-Chlorophenyl)-6-(4-methoxyphenyl)-1,2,4-triazin-5-yl)benzo[d]-imidazo[1,2-c]quinazoline(7)

A solution of (6)(0.01mole) and hydrazine hydrate/phenylhydrazine (0.01 mole) in 50 ml ethanol was refluxed for 3 h. A yellow solid was separated (70% yield), m.p.166 - 168°C and crystallized from dioxane. IR(γ cm^{-1}):3652-3250(NH),1657(C=O),1597(C=N). ^{1}H-NMR(DMSO d_6)δ(ppm) :10.1(s,1H,NH exchangeable with D_2O) 8.2-7.1(m,16H, aromatic protons),3,4(s,3H,OCH_3), 2.8(s,3H,CH_2CH). MS m|z(%):494 $M^{+\cdot}$(23%), 272(25%),

151(32%), 145(36%)105(34%),68(79)56(100).Anal.\ Calcd for $C_{30}H_{23}N_6OCl$,(514): C,78.8; H,4.9;N,17.4.Found :C,78.7;H,4.9;N,17.2.

N-((2-Methoxyphenyl)1,2,3,4,6-pentahydro-4,5,7-trioxo-pyrimidino[4,3-b]pyrimidin-3-yl)-4-chlorobenzamide(8)

A mixture of (1)(0.01 mole) and 6-aminopyrimidin-2,4-dione (0.01 mole) was refluxed in 20 mL of acetic acid for 5 h, cooled and poured into cold water. The solid ppt. was formed, m.p.232 - 234°C (75% yield) and crystallized from ethanol. m.p.122 - 124°C (75% yield). IR(γcm^{-1}):3300-3200(NH),1670-1710(C=O),1630-1590(C=N),1130,1150(C-O).Anal\Cald for $C_{23}H_{17}N_4O_4Cl$ (428):C,77.5;H,4.3;N,10.65.Found:C,77.6;H,4.1;N,10.5.

N-((1,7-Diacetyl-2-methoxyphenyl)-4,5-dioxo-2,3,4-trihydro pyrimidino [4,3-b] pyrimidin-3-yl)-4-chlorobenzamide (9)

Treatment of compound 8 (0.01 mole) with acetic anhydride (25 mL) was refluxed using “Water bath System” for 1 h.

The mixture was left under hood system for half an hour. The solid was separated, filtered and crystallized from pet.ether (80-100). m.p.96 - 98°C (75% yield). IR(γcm^{-1}):1750-1710(C=O),1630-1590(C=N),1130,1150(C-O).Anal\Cald for $C_{27}H_{21}N_4O_6Cl$ (512):C,84.5;H,4.7;N,5.65.Found:C,84.6;H,4.6;N,5.5.

N-((1,7-Diacetyl-2-Methoxyphenyl)1,2,3,4,6-pentahydro-4,5dioxo-oxazolo[4,5-d]pyrimidino[4,3-b]pyrimidin-4-yl)-4-chlorobenzamide (10)

Treatment of compound 8 (0.01 mole) with boiling acetic anhydride (25 ml) was refluxed for 3 h. The mixture was left under hood system for half an hour. The solid was separated, filtered and crystallized from toluene. IR(γcm^{-1}):3300-3200(NH),1670-1710(C=O),1630-1590(C=N),1130,1150(C-O).Anal\Cald for $C_{23}H_{15}N_4O_3Cl$ (410): C,79.5;H,4.0; N,11.65. Found: C,79.6;H,4.1;N,11.58.

N-((2-Methoxyphenyl)1,2,3,4,6-pentahydro-4,5,7-trioxo-pyrimidino[4,3-b]pyrimidin-3-yl)-4-chlorobenzamide(11)

A mixture of (1)(0.01 mole) and 2-amino-6-phenyl-1,3,4-thiadiazole (0.01 mole) was refluxed in 20 ml of acetic acid for 5 h, cooled and poured into cold water. The solid ppt. was formed, m.p.232 - 234°C (75% yield) and crystallized from ethanol. m.p.122 - 124°C (75% yield). IR(γcm^{-1}): 3300-3200(NH),1670-1710(C=O),1630-1590(C=N),1130,1150(C-O).Anal\Cald for $C_{27}H_{19}N_4O_2SCl$ (476):C,82.5;H,4.3;N,10.65.Found:C,82.6;H,4.1;N,10.5.

N-((1,7-Diacetyl-2-Methoxyphenyl)1,2,3,4,6-pentahydro-4,5dioxo-oxazolo[4,5-d]pyrimidino[4,3-b]pyrimidin-4-yl)-4-chlorobenzamide (12)

Treatment of compound 8 (0.01 mole) with boiling acetic anhydride (25 ml) was refluxed for 3 h. The mixture was left under hood system for half an hour. The solid was separated, filtered off and crystallized from toluene. IR(γcm^{-1})1630-1590(C=N), ^{1}H-NMR(DMSO d_6)δ(ppm) 8.2-7.1(m,13H, aromatic protons),5.2 (s, 1H, methine proton), 3,4(s,3H,OCH_3), .Anal\Cald for $C_{27}H_{17}N_4OSCl$ (458): C,83.8;H,4.0; N,11.65. Found: C,83.6;H,4.1;N,11.58.

RESULTS AND DISCUSSION

From the importance of azalactone, poly electrophilic center has been considered. In this study, the behaviour of azlactone 1 was reported towards nitrogen nucleophiles to aim the broading synthesis (El-Hashash et al., 2011, 2012) with hope of getting new compounds of anticipated biological activities. When azalactone 1 was allowed to react with anthranilic acid, it afforded benzoxazin-4-one 2. Treatment of benzoxazinone 2 with hydrazine hydrate affording the quinazolin-4-one 3 following the reaction sequence is depicted in Figure 1.

Previously (D’Rozario et al., 1981), it was reported that the 4H-3,1-benzoxazinone derivative gave the corresponding 4H-3,1- quinazolinone, when it reacted with hydroxylamine hydrochloride. Thus, in this study’s case, when benzoxazinone 2 was treated with hydroxyl amine hydrochloride, the 3-hydroxy quinazolin-4-one derivative 4 was obtained. Electrocyclization can be formed in quinazolinone derivatives 3 and 4. When they were allowed to react in high boiling point solvent, treatment of benzoxazone 2 with hydrazine hydrate in boiling butanol and/or hydroxylamine in boiling pyridine for 4 h, they afforded the corresponding 2-oxazoloquinazolinone derivatives 5. As such, the reaction promoted the thermochemical which allowed [4+2] cyclization (Figure 2).

It has been reported (El-Hashash et al., 2011) that the condensation of 2-aryl(alkyl)benzoxazinone with o-phenylenediamine gave the corresponding 2-aryl-3-hetaryl-4H3,1 quinazolinones; however in this study, by fusion of benzoxazone **2** with o-phenylenediamine, the hetorocyclic benzoimidazole derivatives **6** was formed. Furthermore on treating 5 with hydrazine hydrate or phenyl hydrazine in n- butanol, the corresponding 2-triazino imidazoloquinazolinone derivatives 7 a, b were respectively obtained (Figure 3).

On the other hand, when azalactone 1 was allowed to react with 6-amino1,2,3,4-tetrahydropyrimidin-2,4-dione, it afforded pyrimidino-pyrimidine 8 the opportunity to be investigated with electrophilic reagents. When compound 8 reacted with acetic anhydride in mild condition, it resulted to diacetyl derivative 9. But boiling compound 7

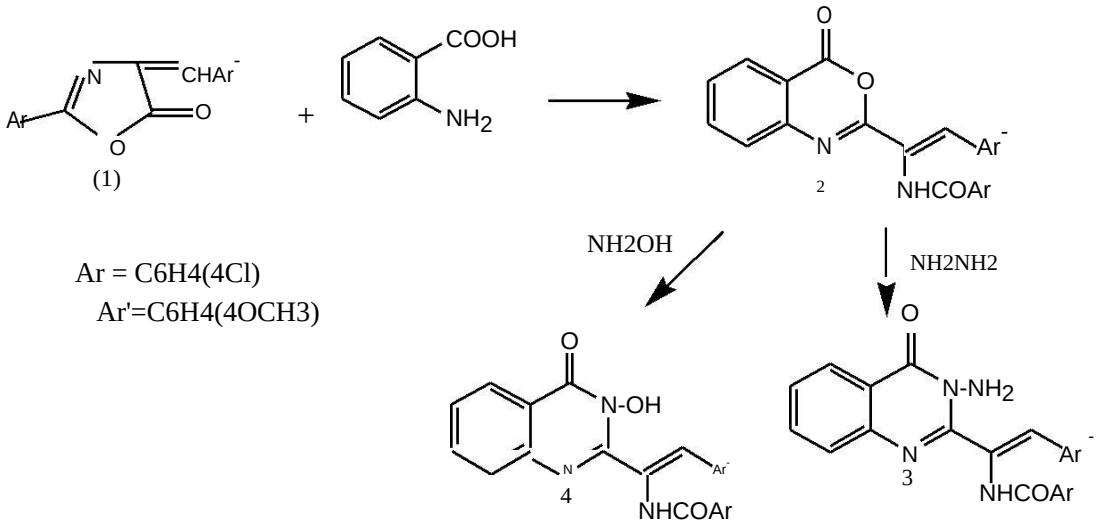

Figure 1. Synthetic pathway for compounds 2,3 and 4.

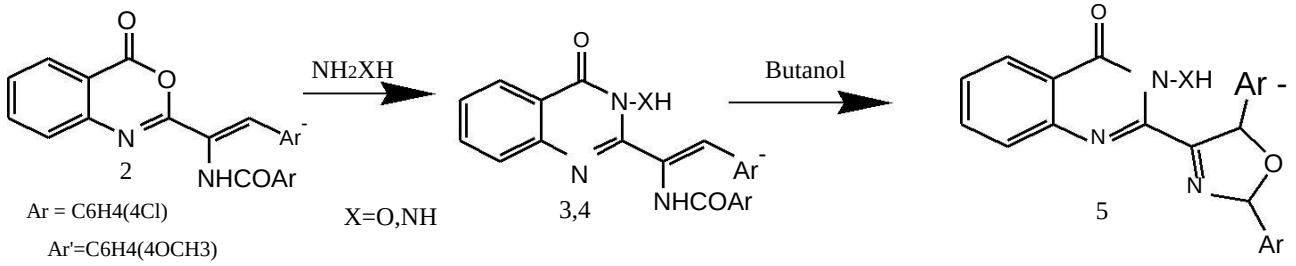

Figure 2. Synthetic pathway for compound 5.

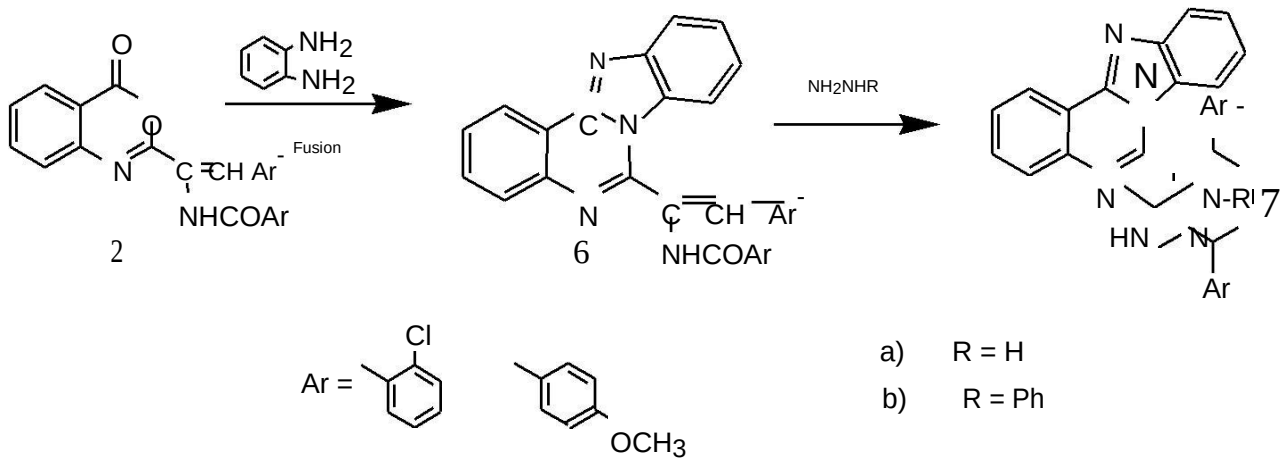

Figure 3. Synthetic pathway for compounds 6 and 7.

with acetic anhydride for 3 h, it resulted to oxazolo derivative 9 (Figure 4).

Similarly, azalactone 1 was allowed to react with 2-amino-5-phenyl-1,3,4-thiadiazole in boiling ethanol, and it afforded ihiadiazolopyrimidine derivative 17 via aza-Micheal addition, followed by heterocycle interconversion

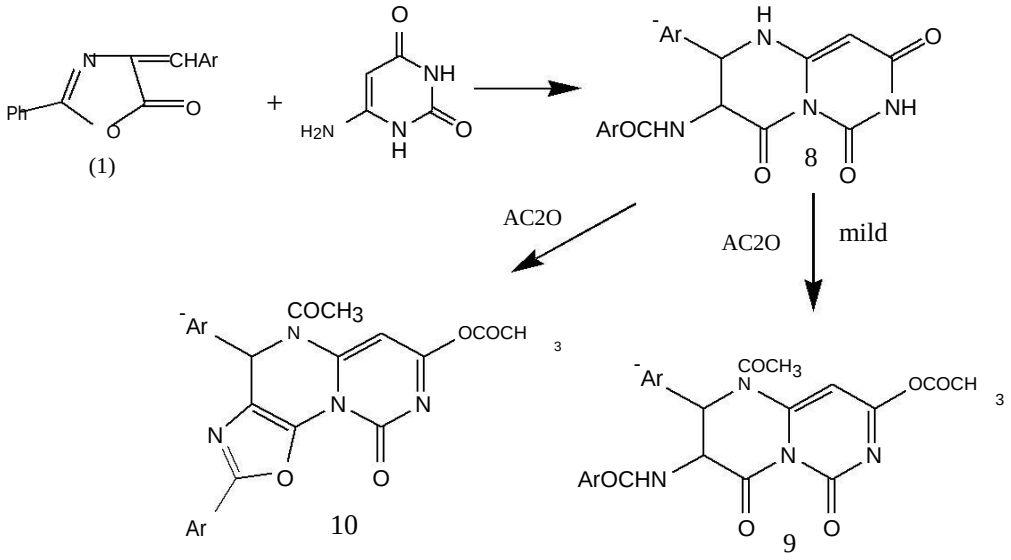

Figure 4. Synthetic pathway for compounds 8,9 and 10.

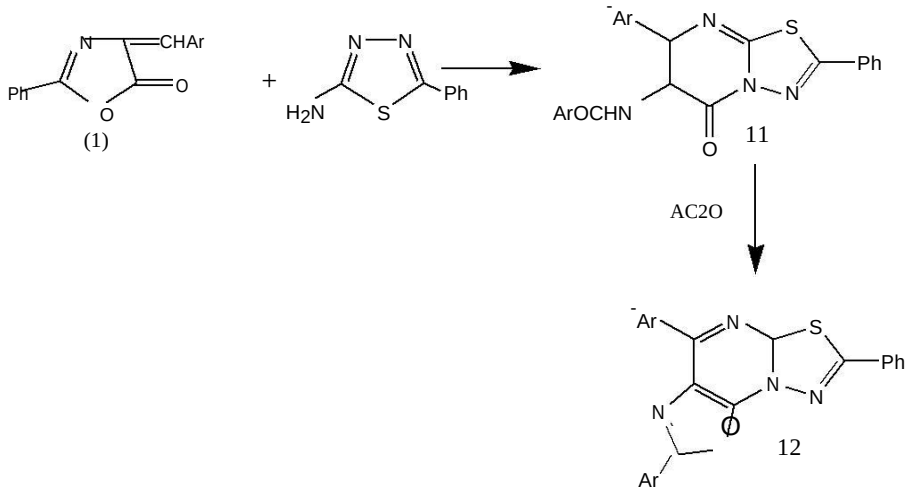

Figure 5. Synthetic pathway for compounds 11 and 12.

(Figure 5). The product of 17 can be elucidated chemically when treated with acetic anhydride, and it resulted to oxazolo derivative 18 which can be supported by spectra and microanalytical data. IR spectrum cannot reveal any carbonyl group, and 1H-NMR confirmed methine proton of thioimidine moiety.

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