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RESEARCH ARTICLE

# New Diquinazoline Derivatives: Synthesis and Evaluation of Anti-Bacterial Activity

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## Abstract

Oxazine and quinazoline has a very important in organic chemistry especially in hetero cyclic fields. this research consist the preparation of 4H,4'H-2,2'-bibenzo[d][1,3]oxazine-4,4'-dione compound (1) from di acid chloride with 2-aminobenzoic acid in pyridine as solvent to give compound (2) 3,3'-diamino-2,2'-biquinazoline-4,4'(3H,3'H)-dione .compound 2 include free amino group .this compound was reacted with maleic and phthalic anhydride for synthesized of cyclic imide compounds (3,4).another reaction for compound 2 with some substituted aromatic aldehyde for prepared of some novel Schiff bases (5-9) contains quinazoline ring. compound 1 was treated with sulfathiazole and sulfadiazine for synthesized of sulfa compounds contains sulfone amide groups (10,11).compounds (12,14) were prepared from the reaction of 4H,4'H-2,2'-bibenzo[d][1,3]oxazine-4,4'-dione with some primary amine p-aminoacetophenone ,p-amino acetanilide to product of (12,13) but compound 14 was prepared from reaction of of compound 1 with 3-amino-2-phenylquinazolin-4(3H)-one to give compound 14 consist four rings of quinazoline. all synthesized compounds were measured for antibacterial activity and the structures of quinaolines were confirmed by thin layer chromatography, melting points ,Fourier transform infrared, H-NMR, elemental analyzer.

**Keywords:** Dioxazine, Diquinazoline, dicyclicimide,antibaterials.

## Introduction

In heterocyclic chemistry there are many rings and hetero atoms in different positions but this research, the focus was on six membered ring that contains two nitrogen atoms at position 1,3 this compounds are called pyrimidine. Pyrimidine attached with phenyl ring at position 5, 6 called quinazoline .A literature survey of substituted pyrimidine and quinazoline derivatives show versatile activity against many bacterial. Recently there are many researches specialized in preparation of quinazoline derivatives field as well as study the possibility of use in the field of pharmaceutical and industrial research.

A large number of quinazoline are reported as anti-microbial activity [1], anti-acetylcholinesterase activity [2], anti-Plasmodium falciparum, Human Cytomegalovirus, and Leukemia Cells [3], antimalarial, antiprotozoan, diuretics, anticonvulsant, muscle relaxant [4]. Compounds of quinazoline attached with sulfonamide moiety have a high activity against tumor [5, 7], anti-cancer [6], anticipated [8], fighting infectious diseases [9].Immunosuppressive activity [10]. Some new Schiff bases analog with quinazoline are considered as high important therapeutic scaffolds [11, 12] such as anthelmintic and anti-oxidant activities [13], antimicrobial [14], there is another use for these compounds where they are used as corrosion inhibition over alloy steel in acidic media [15], mordant dyes [16].phthalimide attached with quinazoline have biological activity such as antiparkinsons, antiaggregatingactivity [17], anticancer against MCF7-& cells [18].

## Experimental

### Materials

The chemicals were equipped by sigma Aldrich, C.D.H ltd and Scharlu Company.

### Instruments

All melting points for prepared compounds were determined by gallenkamp apparatus.

Checking of purity compounds by thin layer chromatography on alumina with proper solvents. The spots were appeared by vapors of iodine or ultraviolet light. CHNS-O of my compounds was measured on EA-3000(ITALY). Fourier transforms infrared shimadzu 8400. H-NMR on BRUKER 300MHz.all compounds were examined for in vitro bacteria and showed a good result.

**Synthesis of 4H, 4'H-2, 2'-bibenzo[d] [1, 3] oxazine-4, 4'-Dione (1)**

To a solution of 2-amino benzoic acid (0.002) mol in pyridine with some pellet of Na OH, diacid chloride (oxalyl chloride) (0.01) was added in ice bath gradually. The mixture solution was sirred at room temperature 24hrs. The product poured onto crushed ice and added 50 ml saturated solution sodium bicarbonate .the solid product filtration and recrystallized from ethyl alcohol. m.p = 295$^0$C, yield= 75%;Rf=0.4 FT-IR (KBr) C=O (of pyrimidind) at 1766 Cm$^{-1}$ C=N=1601 ,CH Ar at 3076. $^1$H-NMR (300MHz**,** DMSO-d6, ppm) 7.14-8.79 ppm(m,H-aromatic ring). Elemental analysis; calc C, 51.58; H, 1.92; N, 6.68; found C, 50.08; H, 1.32; N, 6.38.

**Synthesis of 3, 3'-diamino-2, 2'-biquinazoline-4, 4' (3H, 3'H)-dione(2)**

A solution 4H,4'H-2,2'-bibenzo[d][1,3]oxazine-4,4'-dione (0.5g) in ethanol (150 ml)with three drops of DMF hydrazine hydrate 80% was added (2.5ml) the mixture warmed under reflux at 70$^0$C for 36hrs the yellow precipitate product was filtered and recrystallized from isopropanol. M.P= 203 $^0$C, yield=68%,Rf=0.7; IR: $NH_2$(3267 and 3305 Cm$^{-1}$),(C=O amide)at 1697 Cm$^{-1}$ ,(C=N) at 1624 Cm$^{-1}$; $^1$H-NMR,4.72ppm($NH_2$),7.22-8.60(m,H-aromatic rings); Elemental Analysis: calc: C, 62.74; H, 4.61; N, 27.44;found; C, 60.79; H, 4.32; N, 26.32;

**Synthesis of 1, 1'-(4, 4'-dioxo-2, 2'-biquinazoline-3, 3' (4H, 4'H)-diyl) bis (1H-pyrrole-2, 5-Dione) (3)**

Maleic anhydride (0.02mol)dissolved in glacial acetic acid(50ml) in conical flask (250ml) 3,3'-diamino-2,2'-biquinazoline-4,4'(3H,3'H)-dione(0.01mol) in(100ml) of GAA was added the mixture stirred at R.T for 0.5hr and heated under refluxed for 24hrs the reaction was checked by thin layer chromatography cooled and poured the mixture into ice water filtered to give the title compound which was recrystallized from dioxan; M.P=280 dec ,yield=54% ;Rf= 0.7 ,IR; C=O (1743 and 1697Cm$^{-1}$cyclic imide)C=O(1681 Cm$^{-1)}$ ; $^1$H-NMR, H-aromatic ring (m-7.24-7.92)ppm; H-pyrolidindione(dd ,8.07-8.71) Elemental Analysis: calc; C, 60.00; H, 2.52; N, 17.49 found C, 61.13; H, 2.48; N, 17.17

**Synthesis of 2, 2'-(4, 4'-dioxo-2, 2'-biquinazoline-3, 3' (4H, 4'H)-diyl) diisoindoline-1, 3-dione (4)**

3,3'-diamino-2,2'-biquinazoline-4,4'(3H,3'H)-dione (0.01mol) and phthalic anhydride(0.02mol) was mixed in conical flask with GAA (250ml) the mixture product was refluxed for 24hrs and checked by TLC technique the resulting solution was cooled and poured in cold water the white precipitate was filtered and recrystallized from dioxan M.P=246 ,yield=76% ,Rf=0.6, IR; C=O (1743 and 1708Cm$^{-1}$cyclic imide)C=O(1693Cm$^{-1)}$ ; $^1$H-NMR, H-aromatic ring (m-7.28-78.70)ppm; Elemental Analysis: calc C, 66.21; H, 2.78; N, 14.48 found C, 64.98; H, 2.52; N, 14.98.

**Synthesis of 3, 3'-bis (substituted-benzylideneamino)-2, 2'-biquinazoline-4, 4' (3H, 3'H)-Dione (5-9)**

To a solution of 3,3'-diamino-2,2'-biquinazoline-4,4'(3H,3'H)-dione(0.01mol) in ethyl alcohol(50ml) added of substituted aromatic aldehyde(0.02mol) with 5drops of glacial acetic acid the reaction mixture was stirred and refluxed in water bath at 70$^0$C and monitored by TLC for (8-10hrs). The products were filtered and recrystallized from appropriate solvents.

**3, 3'-bis (4-methoxybenzylideneamino)-2, 2'-biquinazoline-4, 4' (3H, 3'H)-dione (5)**

m.p:271,yield 86%, Rf= 0.9 IR(Cm$^{-1}$): 1697(C=O), 1639(C=N), 3068(=CH imine), 3248(CH aromatic),H-NMR(ppm): 3.75($OCH_3$), 7.03-8.57 (m,H-aromtic ring), 9.96 and 10.32(CH=N), Elemental Analysis: calc:C, 69.06; H, 4.35; N, 15.10,found lemental Analysis: C, 69.23; H, 4.27; N, 15.67.

**3, 3'-bis (-4-methylbenzylideneamino)-2, 2'-biquinazoline-4, 4' (3H, 3'H)-dione** (6)

m.p:300,yield 66%, Rf= 0.4 IR(Cm$^{-1}$): 1697(C=O), 1641(C=N), 3070(=CH imine), 3249(CH aromatic),H-NMR(ppm): 2.36($CH_3$),

7.28-8.68 (m,H-aromtic ring), 9.07 and 9.36(CH=N), Elemental Analysis: C, 73.27; H, 4.61; N, 16.02,found C, 73.27; H, 4.61; N, 16.02.

**3, 3'-bis (-2-methylbenzylideneamino)-2, 2'-biquinazoline-4, 4' (3H, 3'H)-dione (7)**

m.p:291,yield 56%, Rf= 0.5 IR($Cm^{-1}$): 1697(C=O), 1641(C=N), 3068(=CH imine), 3238(CH aromatic),H-NMR(ppm): 2.47($CH_3$), 7.29-8.76 (m,H-aromtic ring), 10.08 and 10.38(CH=N), Elemental Analysis: C, 73.27; H, 4.61; N, 16.02,found C, 73.87; H, 4.75; N, 16.00.

**3, 3'-bis (-benzylideneamino)-2, 2'-biquinazoline-4, 4' (3H, 3'H)-dione (8)**

m.p:321, yield 45%, Rf= 0.7 IR ($Cm^{-1}$): 1693(C=O), 1639(C=N), 3066(=CH imine), 3244(CH aromatic), H-NMR (ppm): 7.34-8.54 (m,H-aromtic ring), 9.07 and 9.29(CH=N). Elemental Analysis: C, 72.57; H, 4.06; N, 16.93, found C, 72.09; H, 4.34; N, 16.67.

**3, 3'-bis (-5-chloro-2-hydroxybenzylideneamino)-2, 2'-biquinazoline-4, 4' (3H, 3'H)-Dione (9)**

m.p:269, yield 75%, Rf= 0.8 IR ($Cm^{-1}$): 1691(C=O), 1643(C=N), 3057(=CH imine), 3244(CH aromatic), H-NMR (ppm): 12.82(s,OH),6.96-8.65 (m,H-aromtic ring), 10.35 and 10.41(CH=N). Elemental Analysis: C, 60.31; H, 3.04; Cl, 11.87; N, 14.07, found C, 60.54; H, 3.25; N, 14.89.

**Synthesis of 4, 4'-(4, 4'-dioxo-2, 2'-biquinazoline-3, 3' (4H, 4'H)-diyl) bis (N-(substituted-2-yl) benzenesulfonamide) (10, 11)**

A solution of amino compound (2) (0.01mol) in glacial acetic acid (30ml) was added to another solution contains from sulfathiazole ,sulfadiazine (0.02mol) the reaction mixture was stirred and refluxed for 15-17 hrs. The reaction mixture was checked by TLC. The mixture poured onto 50ml of ice water with stirred 30minutes after this time kept this solution at room temperature 24hrs and solid product was filtered and recrystallized from appropriate solvents.

**4, 4'-(4, 4'-dioxo-2, 2'-biquinazoline-3, 3' (4H, 4'H)-diyl) bis (N-(thiazol-2-yl) benzenesulfonamide) (10)**

m.p:294,yield 61%, Rf= 0.6 IR($Cm^{-1}$): 1678(C=O),1146 and 1327 ($SO_2$) 3263 (NH),, H-NMR (ppm): 12.21 (s,NH),7.24-8.71 (m,H-aromtic ring), 6.54 and 6.94 (d,d CH thiazole). Elemental Analysis: C, 53.25; H, 2.89; N, 14.61; S, 16.73, found C, 53.79; H, 2.79; N, 14.98; S, 16.81

**4, 4'-(4, 4'-dioxo-2, 2'-biquinazoline-3, 3' (4H, 4'H)-diyl) bis (N-(pyrimidin-2-yl) benzenesulfonamide) (11)**

m.p:287,yield 79%, Rf= 0.5 IR($Cm^{-1}$): 1668(C=O),1126 and 1379($SO_2$) 3296(NH),,H-NMR(ppm): 15.20(s,NH),6.31-8.60 (m,H-aromtic ring). Elemental Analysis: C, 57.14; H, 3.20; N, 18.51; S, 8.47; found C, 56.98; H, 3.09; N, 18.21; S, 8.35.

**Synthesis of 3, 3'-substituted-2, 2'-biquinazoline-4, 4' (3H, 3'H)-dione (12-14)**

A solution of 4H,4'H-2,2'-bibenzo[d][1,3]oxazine-4,4'-dione(0.01mol) in acetic acid (50ml) was hated at $50^0$C when the solubility of compound(1) in acetic acid was added of substituted aromatic amine (0.02mol) the temperature mixture was raised at the reflux point. The mixture was monitored by TLC technique .finally transform of mixture reaction to refrigerator for 24hrs filtered of solution mixture and recrystallized from appropriate solvents.

**3,3'-bis(4-acetylphenyl)-2,2'-biquinazoline-4,4'(3H,3'H)-dione(12)**

m.p:299,yield 51%, Rf= 0.5 IR($Cm^{-1}$): 1678(C=O quinazoline),1662(C=O ketone) 3294(s,CH),,H-NMR(ppm): 2.56(s,CH3),7.23-8.70 (m,H-aromtic ring), Elemental Analysis: C, 72.99; H, 4.21; N, 10.64,found Elemental Analysis: C, 73.07; H, 4.25; N, 10.61.

**N,N'-(4,4'-(4,4'-dioxo-2,2'-biquinazoline-3,3'(4H,4'H)-diyl)bis(4,1-phenylene) )diacetamide(13)**

m.p:287,yield 67%, Rf= 0.4 IR($Cm^{-1}$): 1697(C=O quinazoline),1666(C=O amide) 3313(s,NH),,H-NMR(ppm): 12.80(s,NH),7.34-8.67 (m,H-aromtic ring),2.04(s,$CH_3$). Elemental Analysis: C, 69.06; H, 4.35; N, 15.10, found Elemental Analysis: C, 69.51; H, 4.32; N, 15.23.

**14** m.p:310,yield 65%, Rf= 0.8 IR($Cm^{-1}$): 1678(C=O quinazoline),1662(C=O ketone) 3294(s,CH),,H-NMR(ppm): 6.94-8.61 (m,H-aromtic ring), Elemental Analysis: C, 72.32; H, 3.59; N, 15.33,found C, 72.45; H, 3.63; N, 15.42.

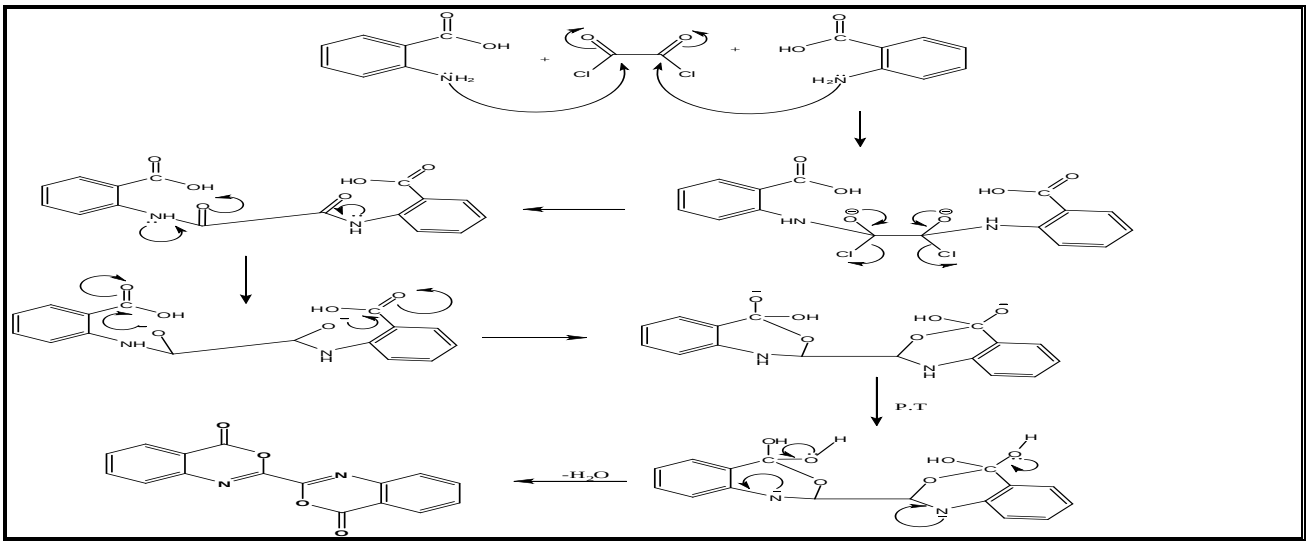
 

### Antibacterial Activity

The fourteenth novel compounds were tested for their some bacterias against *Staphylococcus aureus, Escherichia Coli, Bacillus subtilis, Pseudomonas aeruginosa* at a 100µg/ml, DMSO was used as solvent for tested compounds and was used as a negative control. Ciprofloxacin and Ketoconazole at concentration of 100 µg/mL in dimethylsulfoxide were used as positive control. After incubation period, the growth inhibition zones diameters were carefully measured in mm.

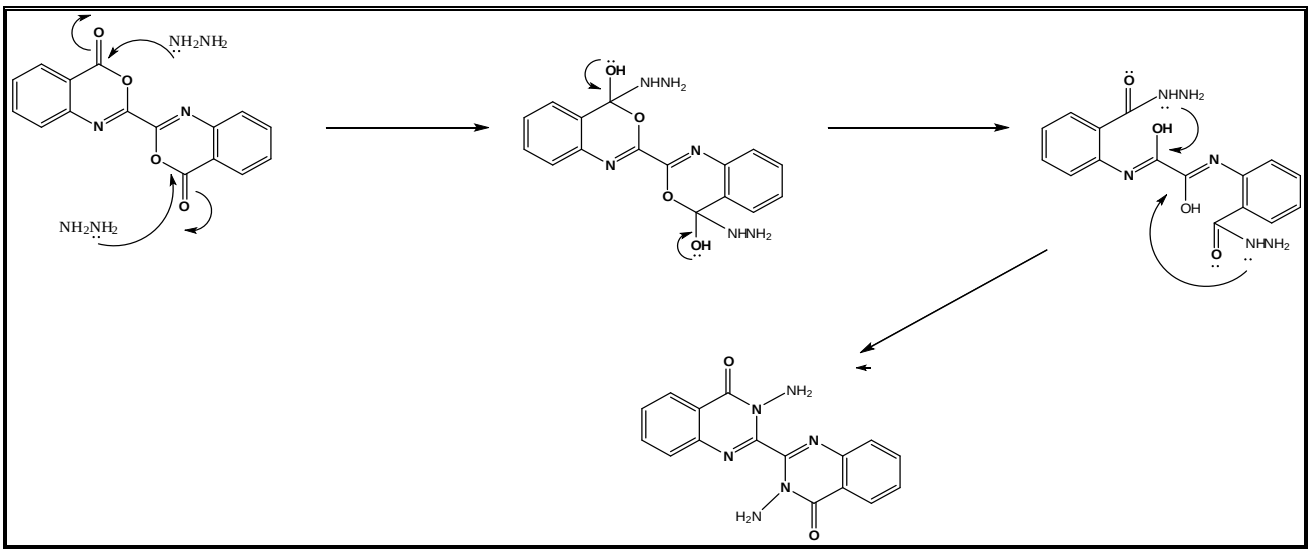
## Results and Discussion

Condensation of 2-oxo-2chloroacetylchloride with 2-aminobenzoic acid afforded corresponding 4H, 4'H-2, 2'-bibenzo[d] [1, 3] oxazine-4, 4'-dione all mechanism steps showed below in scheme (1)




**Scheme 1:**

The structure of compound (1) has been proved by IR, H-NMR and elemental analyzer. Fourier transform of 4H,4'H-2,2'-bibenzo[d][1,3]oxazine-4,4'-dione showed a new bands in region (1766 ,1601) $cm^{-1}$ due to of carbonyl (cyclic ester),imine group in oxazine ring respectively and disappearance of (OH) carboxyl group in anthranilic acid.$^1$H-NMR(DMSO-$d_6$) spectra appeared signals at 7.14-8.79 ppm multiplate signals for protons of aromatic ring. Compound (1) was treated with 80% of hydrazine hydrate to give compound (2) IR spectra of this compound showed a good evidence for prove that the reaction between compound 1 with $NH_2NH_2$ to give diquinazoline derivative is band at 1697$cm^{-1}$ due to of carbonyl in quinazoline ring and two bands at 3305and 3207 $cm^{-1}$ for symmetric and asymmetric of primary amine. Proton NMR showed signal at 4.72ppm single peak of $NH_2$ group and multi signals at 7.22-8.60 for protons in benzene rings. The suggested mechanism for the synthesized of compound 2 is shown below in Scheme (2).




**Scheme 2:**

Compound 2 was treated with cyclic anhydride (maleic and phthalic) anhydride for prepared of cyclic imide contains diquinazoline derivatives compound (3, 4). FT-IR of compound (3, 4) exhibited characteristic absorbtion bands at region range (1743, 1708, 1681) of (C=O) of cyclic imide and quinazoline rings. Other proof is disappearance of amino group in compound 2 and appearance of carbonyl cyclic imide.

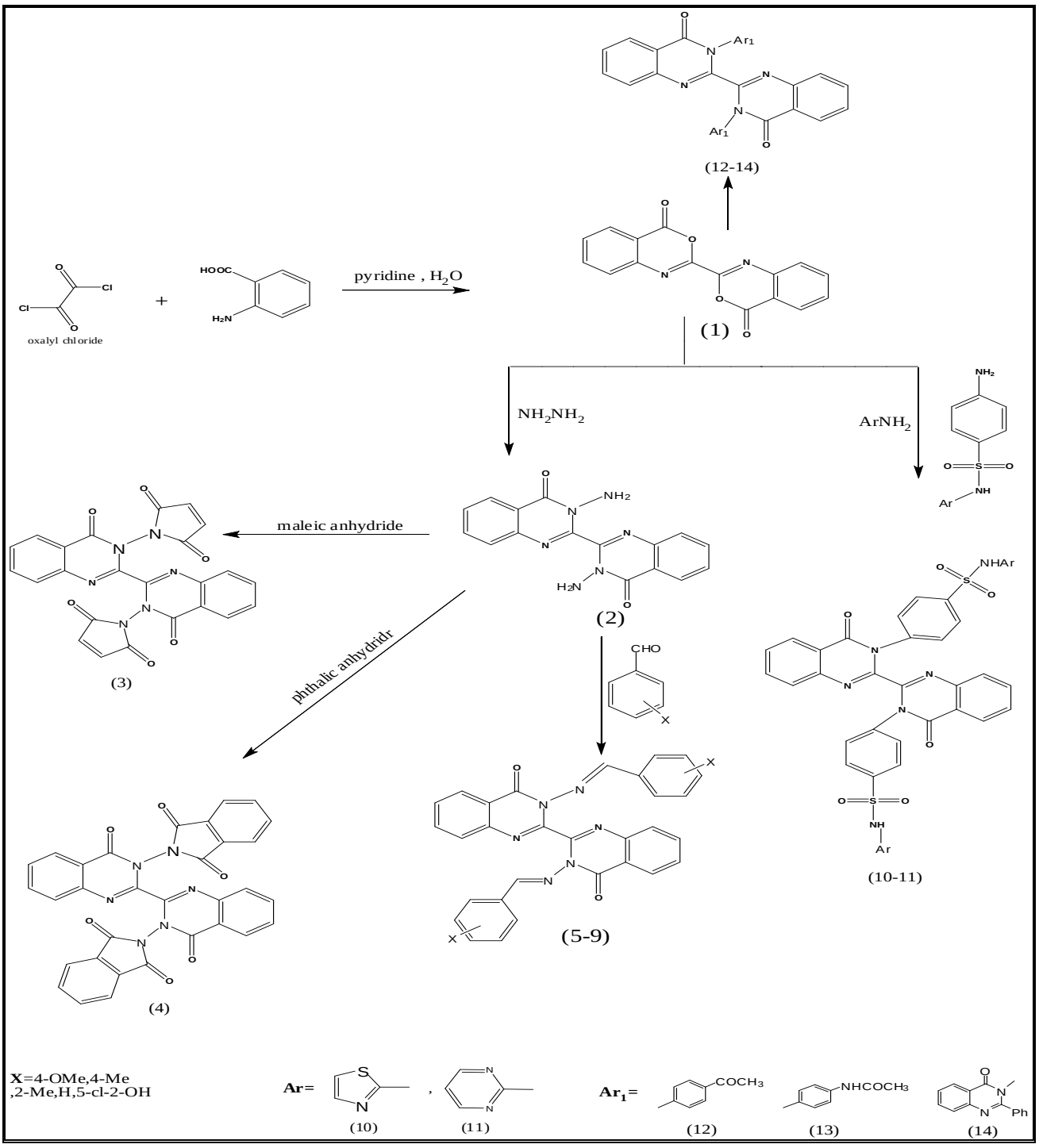
$^1$H-NMR (DMSO-$d_6$) of compound (3, 4) showed disappearance in region 4.72ppm signal of amino signal in compound2 this is a good evidence for prepared of cyclic imide (3,4). Compound 2 is precursor for synthesis of some new Schiff bases of qunazoline derivatives by reaction with some substituted aromatic aldehyde to give compounds (5-9). The IR of these compound showed a new bands (1639-1643) for imine (C=N) and disappearance bands of amino group .proton NMR appeared new signals (9.07-10.38) ppm due to of proton schiff bases and disappearance signals at 4.72ppm for ($NH_2$).compounds (10-11) were synthesized by the reaction of compound 1 with sulfathiazole, sulfadiazine this compounds is very important in in pharmaceutical fields because these compounds contains sulfonamide groups. The FT-IR of these compounds showed disappearance of carbonyl ester in oxazine ring and appeared a new band at (1668-1678) $cm^{-1}$ for carbonyl in cyclic amide in quinazoline rings ,bands at (1126,1146) and (1327,1379) $cm^{-1}$ for ($SO_2$) .H-NMR showed anew peak at (12.21 and 15.20) for NH in sulfonamide moiety and two peaks in compound 10 at (6.54 ad 6.94)ppm for protons thiazole ring.

Compounds (12-14) were prepared by the reaction of compound 1 with some amino derivatives p-amino acetophenone, p-amino acetanilide, amino quinazoline derivative respectively, the FT-IR of compounds 12 appeared band at 1678 $cm^{-1}$ for (C=O) in quinazoline ring and new band at 1662 $cm^{-1}$ for acetophenone and disappearance band of carbonyl ester in oxazine ring compound 13 showed bands at 1666, 3313 $cm^{-1}$ for (C=O) and (NH) respectively.

## Antimicrobial Activity

**Table 1: Show the results for evaluation of antibacterial for all synthesized compounds In vitro anti-microbial activity of synthesized compounds expressed as inhibition zones (mm)**

| No. of compounds | Staphylococcus aureus | Escherichia Coli | Bacillus subtilis | Pseudomonas aeruginosa |
|---|---|---|---|---|
| 1 | 12 | 12 | - | - |
| 2 | 10 | 13 | 10 | - |
| 3 | 7 | 9 | 10 | - |
| 4 | 9 | 8 | 9 | |
| 5 | - | - | - | 9 |
| 6 | - | 14 | – | – |
| 7 | 15 | 14 | – | – |
| 8 | - | 13 | – | – |
| 9 | - | 12 | - | - |
| 10 | 11 | - | - | 8 |
| 11 | 27 | 13 | - | - |
| 12 | 11 | - | - | 8 |
| 13 | 27 | 13 | - | - |
| 14 | 10 | 12 | - | 14 |

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