Synthesis and Characterization of New Phthalimides Containing 1, 2, 4-triazole and Imine Group

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Abstract

Several new derivatives of 1, 2, 4-triazoles linked to phthalimide moiety were synthesized through following multisteps. The first step involved preparation of 2, 2-diphthalimidyl ethanoic acid [2] via reaction of two moles of phthalimide with dichloroacetic acid. Treatment of the resulted imide with ethanol in the second step afforded 2,2-diphthalimidyl ester[3] which inturn was introduced in reaction with hydrazine hydrate in the third step ,producing the corresponding hydrazide derivative[4] .The synthesized hydazide was introduced in different synthetic paths including treatment with carbon disulfide in alkaline solution then with hydrazine hydrate to afford the new 1,2,4-triazole[10] .Reaction of compound [10] with different aldehydes produced a new Schiff base derivatives[11,12] .Reaction of derivative [4] with different aldehydes produced a new derivatives [5-8].All the synthesized compounds have been characterized by melting points ,FTIR ,^{1}HNMR(some of them) and mass spectroscopy of compound [2]. Derivatives [5, 6, 7, 10, 11, 12] were tested against inhibition of *E-coli, staphyloccus aureus* and were all found to be active. Schem1,2 illustrated the reaction steps.

Keywords: synthesis, imide, 1, 3, 4-triazole, Schiff bases**.**

Introduction

During the last few decades, a considerable attention has been devoted to the synthesis of 1,2,4-triazole derivatives possessing comprehensive bioactivities[1] .for example, a large number of 1,2,4-triazoles have been incorporated into a wide variety of therapeutically interesting drug *candidates* including *anti-inflammatory*[2,3] *antioxidant*[4], *analgesic*[5], *antimicrobial*[6-9], *anticancer*, and *antifungal activities*[10]

Among the bicyclic non aromatic nitrogen heterocycles, phthalimide is interesting functionality due to its wide presence in the natural products and in the pharmacologically active compounds. Compounds containing phthalimide moiety are distinguished by their potent fungicidal action [11, 12]

Schiff bases are also known to have biological activities such as antimicrobial [13], antifungal [14], and antitumor [15] and as herbicider [16].Keeping in view the facts mentioned we thought it is worthwhile to synthesize new Schiff bases containing phthalimide moiety which were predicated to have useful biological activities.

Experimental

Instruments

Ftir Spectra Were Performed On A Shimadzu Ftir 8400 Fourier Transform Infrared Spectrophotometer. ^{1}hnmr Spectra Were Recorded On A Bruker, Ultrashield 300 Mhz Spectrometer And Mass Spectroscopy Were Recorded On A Gcms Qp2010 Ultra(Gas)Chromatograph Mass Spectrometer Shimdzu Japan. Melting Points Were Determined On Gallenkamp Melting Apparatus And Were Uncorrected.

Chemicals

All chemicals were of analytical reagent grade and were used without further purification

-Synthesis of 2, 2- diphthalimidyl ethanoic acid [2] [11]

Phthalimide (1g, 0.007 mole) was dissolved in aqueous potassium hydroxide (0.57g , 0.01 mole) 50mL distilled water then added dichloroacetic acid (0.438g , 0.003 mole). The reaction mixture was heated on sand bath for (4 hrs). The reaction mixture was cooled to room temperature and acidified with dilute HCI to precipitate the acetic acid derivative. The crud product was recrystallized from ethanol.

-Synthesis of ethyl -2, 2- diphthalimidyl ethanoate[3][12]

Methyl -2, 2- diphthalimidyl acetic acid [2] (4.0g, 0.01 mole) was dissolved in ethanol (70mL).concentration sulfuric acid (4mL) was added. The reaction mixture was refluxed for (6 hrs.). After completion of the reaction (monitored by T.L.C), the reaction mixture was poured on to ice-cold water then neutralized with (2%) KOH .The mixture was extracted with ethyl acetate (2X30 mL), combined organic layer dried over magnesium sulphate and the solvent was removed to give compound [2] as a syrup.

Synthesis of 2,2 diphthalimidyl ethanoic hydrazide [4] [17,18]

Compound [3] (1.8g, 0.004 mole) and hydrazine hydrate (4.0mL) were dissolved in (25mL) of ethanol. The reaction mixture was refluxed for (20 hrs). The precipitate which separated on cooling was filtered and recrystallized from ethanol to give compound [3]

Diphthalimidyl ethanoic hydrazone methyl substituted phenyl (5-8) [19]

Compound [4] (0.01 mol.) was dissolved in 50 mL of absolute ethanol. Appropriate aldehyde (0.01mol.) was added gradually, 2-3 drops of glacial acetic acid was added. The reaction mixture was refluxed for (7 hrs.) After completion of the reaction (monitored by T.L.C) and cooled, the product was precipitated filtered off and recrystallized from appropriate solvent, Table-1. showed the physical properties for Schiff base derivatives (4-7)

Synthesis of 2,2 diphalimido methyl xanthate [9] [20]

Compound [4] (3.8g , 0.05 mole) was dissolved in solution of potassium hydroxide (0.6g , 0.01 mole) in ethanol (100mL) and the reaction mixture was stirred for 1 hrs.at room temperature. Then carbon disulfide (3.8g, 0.05mole) was added slowly at 0-5 c. The reaction mixture was stirred for overnight at room temperature. The xanthate [13] was filtered, washed with ether.

Synthesis of 3-(2,2- diphthalimidyl – methyl) -4- amino – 1,2,4- triazole -5- thione [10] [20]

Compound [9] (3.4g, 0.01) was dissolved in (40mL) of water. Then excess amount of hydrazine hydrate was added. The mixture was refluxed for 20 hrs. The reaction was cooled, and then neutralized with 10 % HCl. The separated crude produced was filtered, dried and recrystallized from ethanol.

Synthesis of 3-(2,2-diphthalimidyl – methyl) -4- arylidenimino 1,2,4- triazole -5-thione [11-12] [19]

Compound [10] (0.6, 0.01 mole) was dissolved in 250 mL of methanol. Appropriate aldehyde (0.02 moles) was added gradually and (2-3) drops of glacial acetic acid were added. The mixture was refluxed for (16 hrs.). On cooling the separated solid was filtered dried and recrystallized from methanol .Table (1) showed the physical properties for compounds (2-12).derivatives. While the tables (2 and 3) include the structures and nomenclatures for compounds (2-12).

Result and Discussion

Chemistry

The present work is directed towards synthesis of new heterocyclic derivatives of phthalimides Containing 1, 2, 4-triazole and imine group derivatives. Performing this target was achieved through following multi step synthesis which its steps are outlined in scheme (1) and (2). The first step involved the synthesis of 2,2-diphthalimidyl ethanoic acid which was prepared via reaction of two moles of phthalimide potassium salt [1] with dichloroacetic acid according to Gabriel Synthesis.

The structure of the white crystals of compound (2) was assigned on the basis of mass and FTIR spectral data. The spectrum of electron ionization mass spectroscopy (EIMS) for compound2, 2- diphthalimidyl ethanoic acid figure (1) displayed the molecular ion at m/z= 350 also the base peak appeared at m/z=166. In addition of these, the other fragments were in agreement with the suggested structure of compound 2, 2- diphthalimidyl ethanoic acid. Furthermore, the patron of these fragments and the rearrangements which appeared are in

agreements with most literatures that study the mass fragmentations of the substituted phthalamide ([16-18]). (Scheme 3) dedicated the most important fragments. In EIMs losing molecular of CO or 2CO beside the CO_2 from phathalmide attached with acetic acid are widely known [18, 19]. In our compound the molecular ion appeared as exactly expect ($M^{\cdot +}$), however in some literatures the molecular ion was either ($M^{\cdot +}$-CO) and ($M^{\cdot +}$-CO_2) [4] or $M^{\cdot +}$-N_2 [20]instead of $M^{\cdot +}$.

The FTIR spectrum showed clear absorption bands at (3400-2500) cm^{-1} due to v (O-H) acid, v (1693) cm^{-1} , (1770) cm^{-1} due to vasy and vsy (C=O) imide respectively,(1687) cm^{-1}due to v (C=O)acid. And the mass spectrum supported this compound. Synthesis of compound 4 involved two steps; in the first one compound [2] was converted to its ester derivatives [3] by the reaction of with ethanol in the presence of sulphric acid. The resulted ester [3] was introduced in reaction with hydrazine hydrate in ethanol absolute under reflux condition in the second step producing the desired hydrazide derivative [4].

FTIR Spectrum of compound [3] showed disappearance of absorption bands due to v (C=O) carboxylic acid at (1687) cm^{-1} and v (O-H) acid at (3400-2500) $cm^{-1}$with appearance of v (C=0) ester at v (1726) and v (C-O) ester at (1281) cm^{-1}

FTIR Spectrum of compound [4] showed disappearance of absorption due to v (C=0) and v (C-O-C) ester at (1726) cm^{-1} and (1281)cm^{-1}respectively with the appearance of v(NH-NH_2)absorption band at (3429-3167)cm^{-1}proving success of hydrazide formation .The FTIR showed other bands (1732)cm^{-1},(1660)

cm^{-1} due to vsy and vasy(C=0)imide and (1602) cm^{-1},(1558) cm^{-1} ,(1377) cm^{-1}due to v(C=0)amide, v(C=C)aromatic, v(C-N)imide respectively.

The next step in this work involved introducing of compound (4) in different synthetic two paths the first path introduced various new schiff bases [5-8](scheme1) and the second path produced new heterocyclic (1, 3, 4- triazole) [10](scheme2) all of them contain two phthalimides moiety.The first synthetic path involved introducing of compound [4] in reaction with different aromatic aldehydes in ethanol in the presence of few drops of glacial acetic acid under reflux producing the new Schiff base (5-8).

FTIR Spectra of compound [5-8] respectively showed disappearance of absorption bands due to v (NH_2) absorption at (3429,3313)cm^{-1}. The IR Spectra showed absorption at v (NH) absorption bands at (3173-3120) cm-^{1}. Also the IR Spectra showed absorption at (1674-1643) cm^{-1},(1745-1735) cm^{-1},(1616-1597) cm^{-1},(1552-1512) cm^{-1} ,(1375-1348) cm^{-1} due to v (C=0)amide, v (C=0)imide v (C=N)imine , v (C=C)aromatic, v (C-N)imide, respectively.

^{1}HNMR spectrum of compound [7] figure(2) is shown δ 3.4 ppm(1,S, CHCO) proton, δ 2.3ppm (1,S,CH_3) protons, δ 8.67ppm (1,S, NH), δ (7.3-7.78)ppm(13,m, aromatic and imine proton)

The second path involved introducing of compound [4] in reaction with carbon disulfide in presence of potassium hydroxide to producing salt [9] F.T.IR Spectrum of compound [9] showed the disappearance absorption band at the (3429, 3167) cm-^{1}due to asymmetric and symmetric stretching vibration of the (NH-NH2) group. Appearance band at (1205) cm^{-1}due to (C=S) also appearance band at (1442) cm^{-1}due to (-N-C=S). These bands are good evidence for the presence of this compound. Treatment of compound [9] with access amount hydrazine hydrate produced compound [10]. F.T.IR spectrum compound [10] showed absorption band due to v (NH) indicates (the presence of tautomerism).The weak band at (2582) cm-^{1}due to v (S-H), (3307-3163) cm^{-1}due to v (NH,NH_2).Other bands (1730, 1705)cm^{-1} due to vsy and vasy

(C=O)imide. Reaction of 5-(N, N –diphthalimidyl) methyl -3-thion-1, 2, 4triazole with different substituted aldehyde produced compounds [11,12] . F.TIR Spectrum of compound [11] showed the disappearance of v (NH) group due to the possibility of hydrogen bonding ,(1614)cm-^{1}for v (C=N)imine,(1267)cm^{-1}for v (C=S) and (1450) cm^{-1}for v (-N-C=S). F.TIR Spectrum of derivative [12] showed single stretching band due to (NH) group appeared at (3140) cm^{-1}, (1608) cm^{-1}due to v (C=N) imine, (1269) cm^{-1}due to v (C=S), and(1431) cm^{-1}due to v (-N-C=S).cm^{-1}, (1552) cm^{-1} (1375) cm^{-1}, due to v (C=0) imide, v (C=C) aromatic, v (C-N) imide. ^{1}HNMR for compound [11],figure(3) shown δ 3.4 ppm(1,S,CHCO) proton , δ 11ppm (1,S,OH)protons, δ 9 ppm (1,S,NH)proton , δ (6.9-7.7ppm aromatic protons and imine proton.

Biological Activity

The effect of compounds [5, 6, 7, 10, 11,and 12] prepared in (10% DMF solution) were tested against two types of bacteria Escherichia coli and staphylococcus aureus the experiment was conducted by using nutrient agar plates[26]. The plates were incubated at 37 C^{0} for 24 hrs. The study showed all compounds have a varying biological activity toward mentioned bacteria accept compound [12] has no biological activity toward the E.coli

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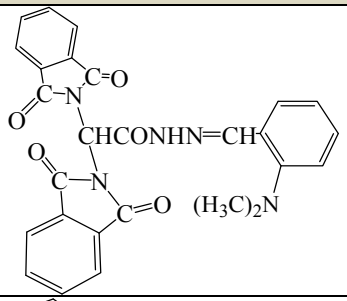
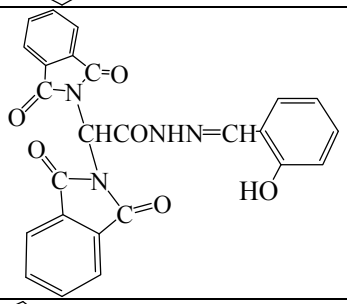
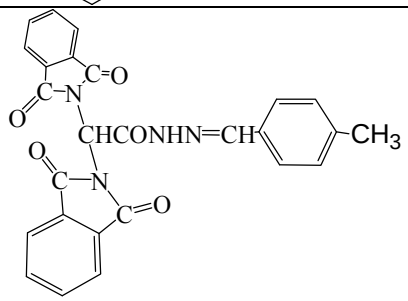
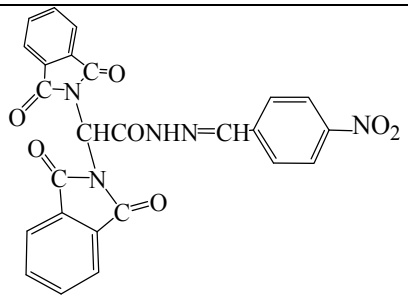
Table NO. (1) Physical properties of compounds (2-12)

Com No.	Molecular Formula	M.W G/Mole	M.P. C	Yield %	color	Solvent recryst
2	$C_{18}H_{10}N_2O_6$	350	195-200	80	White	Ethanol
3	$C_{20}H_{14}N_2O_6$	364	-	99	Oil	-
4	$C_{18}H_{12}N_4O_5$	364	300 dec-	75	White Greenish	Methanol-
5	$C_{27}H_{21}N_5O_5$	495	250-253	80	Yellow	Aceton
6	$C_{25}H_{16}N_4O_6$	468	210-215	76	Yellow	Aceton
7	$C_{26}H_{18}N_4O_5$	466	130-132	75	Yellow	Methanol
8	$C_{25}H_{15}N_5O_7$	497	300 dec	60	Reddish Yellow	Methanol
9	$C_{19}H_{11}N_4O_5S_2K$	407	110-112	90	Greenish-yellow	-
10	$C_{19}H_{12}N_6O_4S$	420	300dec	80	White	Methanol
11	$C_{26}H_{16}N_6O_5S$	524	190-193	75	Yellow	Methanol
12	$C_{26}H_{15}N_6O_4SBr$	583	170 - 175	75	Yellow	Methanol

Table N0. (2): the compound structure and nomenclature of (5-8)

Com No.	Compound Structure	compound name
5		diphthalimidyl ethanoic acid (2-dimethyl amino-benzlidene) hydrazine
6		diphthalimidyl ethanoic acid (2-hydroxy-benzlidene)hydrazine
7		diphthalimidyl ethanoic acid (4-methyl-benzlidene)hydrazine
8		diphthalimidyl ethanoic acid (4-nitro-benzlidene) hydrazine

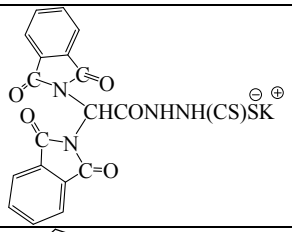
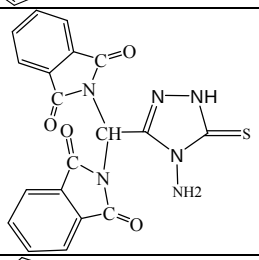
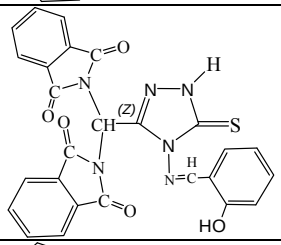
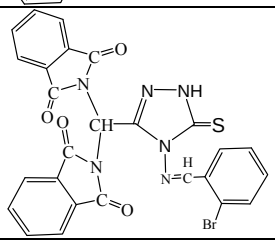
Comp. No.	Compound Structure	Compound name
9		2,2 diphalimido methyl xanthate
10		4-amino-5-(2,2diphthalimidyl-methyl)2,4-dihydro-1,2,4-triazole-3-thione
11		5-(2,2diphthalimidyl-methyl)-4-(2-hydroxy benzlidene -amino)2,4-dihydro-1,2,4-triazole-3-thione
12		5-(2,2diphthalimidyl-methyl)-4-(2-bromo benzlidene amino)2,4-dihydro-1,2,4-triazole-3-thione

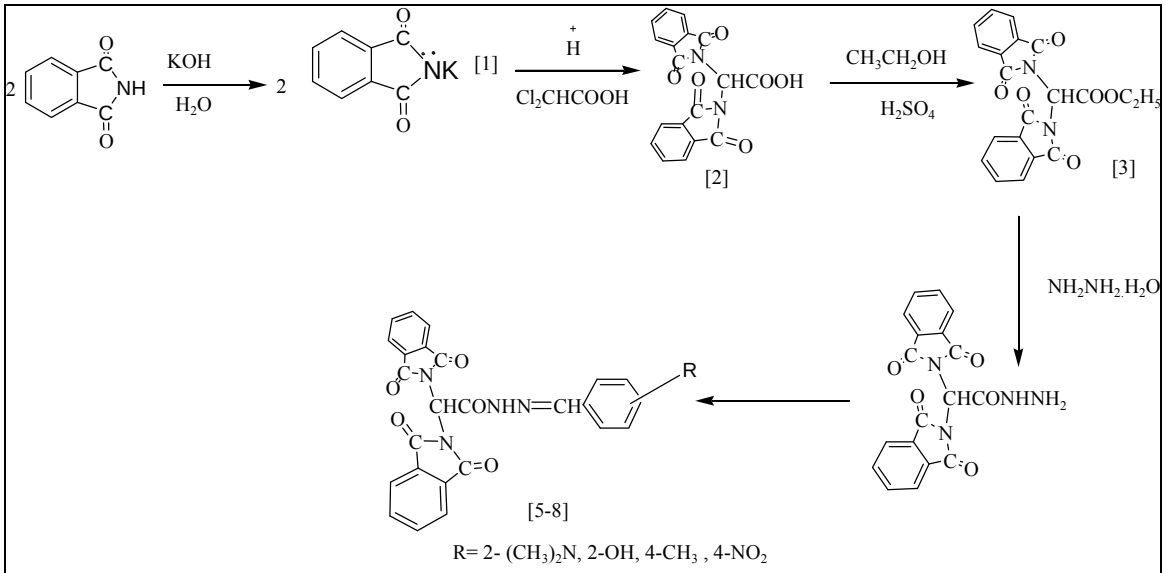
Table No. (3): The compound structure and nomenclature of (9-12)

Table No. (4) : result of biological activity for compounds [5-7] and [10-12]

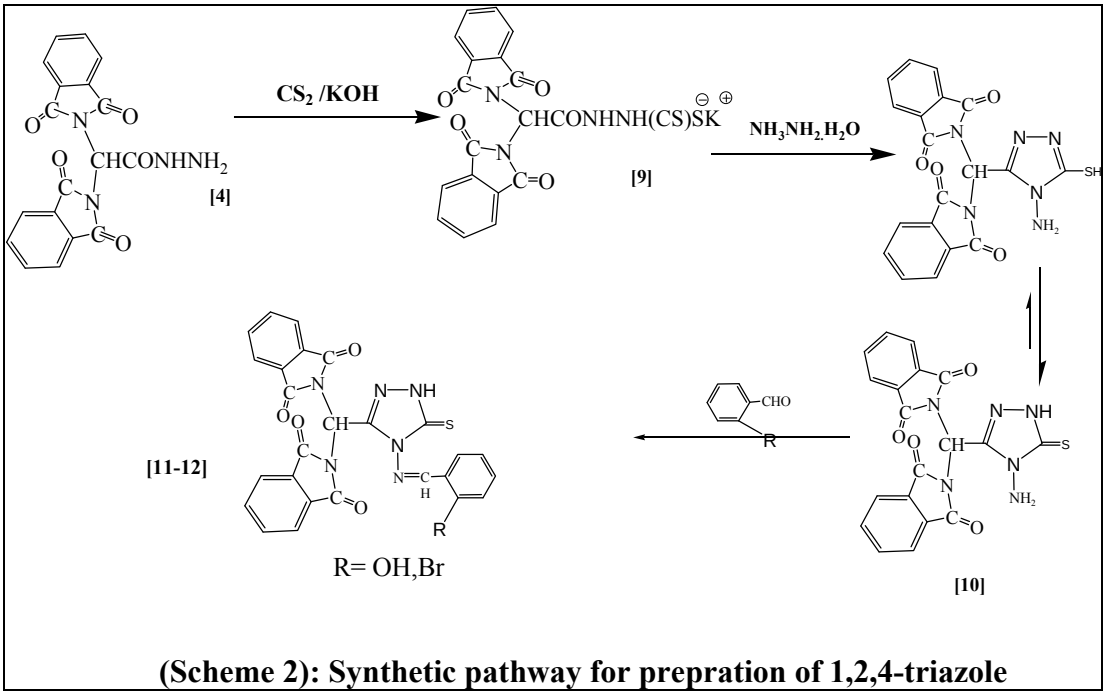
Comp.No.	E.coli	Staphylococcus aureus
Control Solvent DMF	11 mm	---
5	15 mm	20 mm
6	14 mm	16 mm
7	17 mm	18 mm
10	11 mm	15 mm
11	13 mm	18 mm
12	--	14 mm

Table No (4) result of biological activity for compounds [5-7] and [10-12]

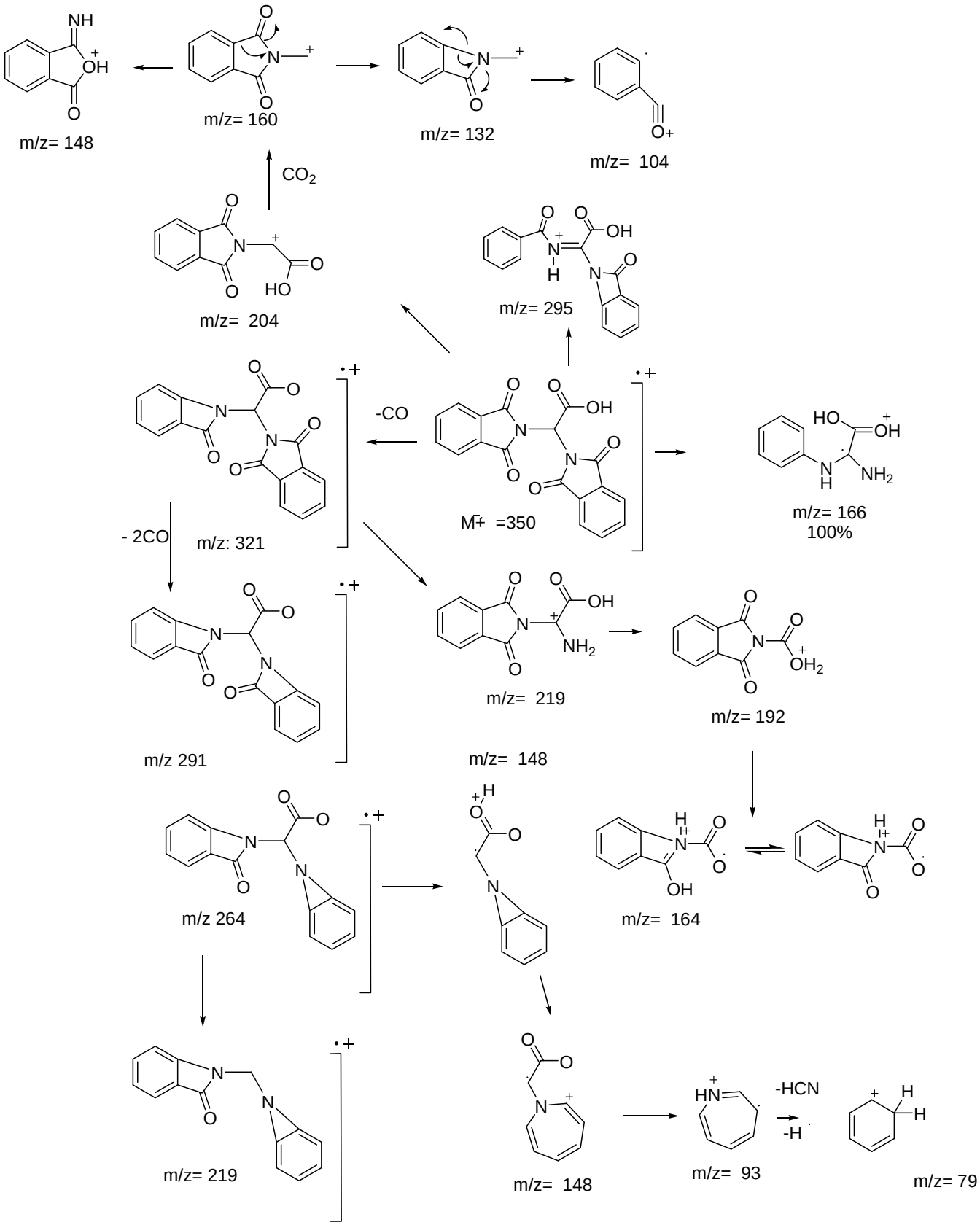
Comp.No.	E.coli	Staphylococcus aureus
Control Solvent DMF	11 mm	---
5	15 mm	20 mm
6	14 mm	16 mm
7	17 mm	18 mm
10	11 mm	15 mm
11	13 mm	18 mm
12	--	14 mm



(Scheme 1): Synthetic pathway for prepration of imine group



(Scheme 2): Synthetic pathway for prepration of 1,2,4-triazole



(Scheme 3): the most important fragments for compound [2]

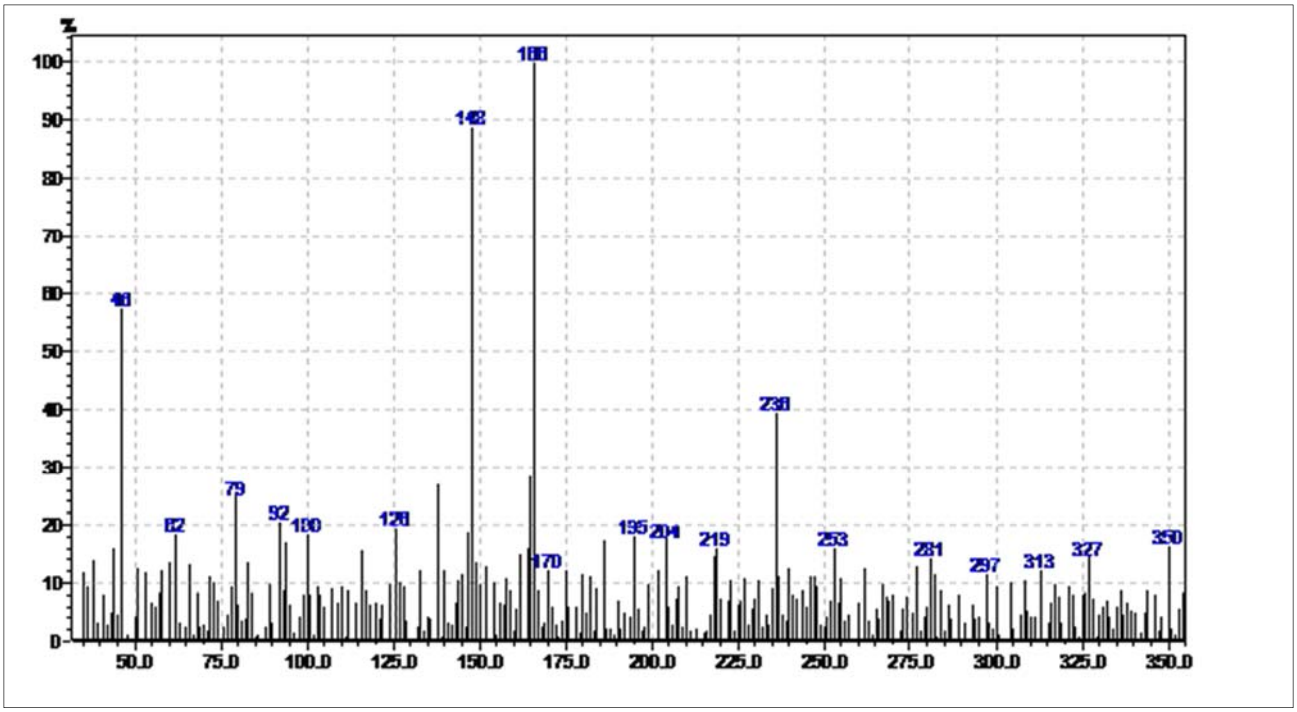


Figure (1) mass spectrum for compound [2]

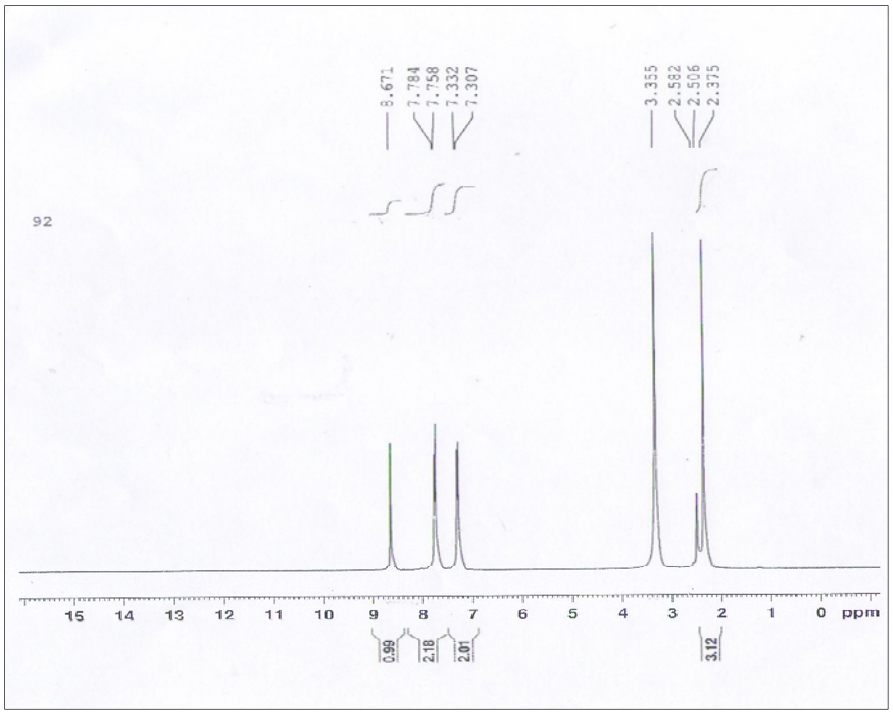


Figure (2): ^{1}HNMR spectrum for compound [7]

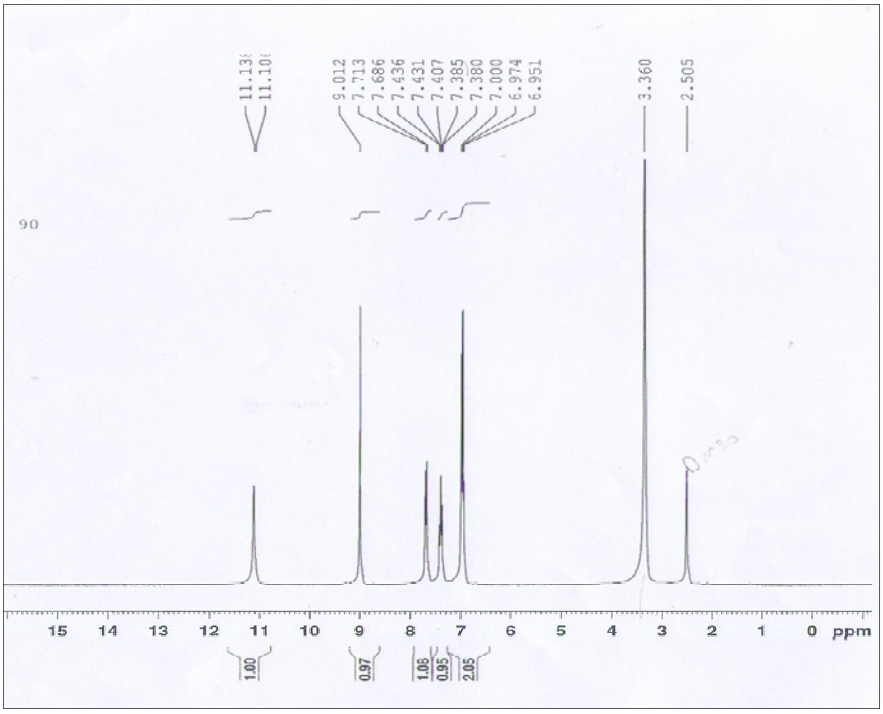


Figure (3): ^{1}HNMR spectrum for compound [11]

تحضير وتشخيص فثال ايمايدات معوضة جديدة تحتوي مشتقات ١،٢،٤ترايزول ومجموعة ايمين

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الخلاصة
تم في هذا البحث تحضير عدد من مركبات ٤،٢،١-ترايازول الجديده المشتقة من مركب فثال ايمايد .حضرت هذه المركبات باتباع عدة خطوات حيث تضمنت الخطوة الاولى تحضير المركب ٢،٢فثال ايمايديل حامض الايثانول [٢]من خلال تفاعل مولين من جزيئة الفثال ايمايد مع مول واحد من ثنائي حامض كلورواسيتك وفي خطوة ثانية المركب الناتج اجري له عملية استرة انتج مشتق استر ثنائي فثال ايمايديل استيت[٣] وهذا بدوره تم معاملته مع الهدرازين المائي ادى الى تكوين مشتق الهدرازايد [٤]في خطوة ثالثة .ومشتق الهدرازايد تم ادخاله في مسارات تحضيريه حيث عومل مع ثاني كبريتيد الكاربون قي الوسط القاعدي ثم مع الهايدرازين المائي لتكوين مشتق٤،٢،١-ترايزول [١٠] الذي اسفرت تفاعلاته مع الديهايدات مختلفه تكوين قواعد شف جديدة[١١،١٢] وعند مفاعلة المشتق [٤]مع الديهايدات مختلفة اعطى مشتقات جديدة [٨-٥] . تمت دراسة الفعالية البايلوجية للمشتقات [٥،٦،٧،١٠،١١،١٢]بكتريا ايكولاي و استافيلوكوكس واظهرت جميعها فعالية بايلوجية

كلمات مفتاحية: تحضير، ايمايد،١،٣،٤ ترايزول، قواعد شف