

SYNTHESIS, CHARACTERIZATION, AND ANTIBACTERIAL ACTIVITIES OF NAPHTHO[2,1-*b*]FURAN DERIVATIVES

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ABSTRACT

The naphthofuran and its derivatives are important biological compounds so we have focused on the synthesis of naphthofuran derivatives. The synthesized compounds of ethyl 1-(acetylamino)-5-nitronaphtho[2,1-*b*]furan-2-carboxylate (3), and *N*-[2-(hydrazinylcarbonyl)-5-nitronaphtho[2,1-*b*]furan-1-yl]acetamide (4) used for the synthesis of *N*-(2-[(2*Z*)-2-benzylidenehydrazinyl]carbonyl)-5-nitronaphtho[2,1-*b*]furan-1-yl]acetamide compounds of four derivatives 5 (a-d) and 1-acetamido-5-nitro-*N*-(5-oxo-2-phenylthiazolidin-3-yl)naphtho[2,1-*b*]furan-2-carboxamide compounds of four derivatives 6 (a-d). The prepared compounds were confirmed by FTIR NMR, and mass methods. The prepared naphthofuran derivatives used for the *antibacterial* activity versus both Gram(+ve) and Gram(-ve) bacteria show excellent results. Therefore the synthesized compounds are used for further *antibacterial* studies in the medical field.

Keywords: Naphthofuran, Antibacterial, Heterocyclic, Biological Activity, Naphthol.

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INTRODUCTION

In medicinal chemist, nitrogen, oxygen, and sulfur-containing heterocycles are important f compounds. The naphthofuran is an organic derivative formed from the naphthalene ring into a heterocyclic furan ring.¹ Naphthofuran molecule has good structural moieties so it is used in important natural biological products and shows good biological and pharmacological activities.²⁻⁴ Therefore, an attempt has been made to synthesize various novel *N*-substituted naphthofuran carboxamides carrying naphthofuryl rings and to study their antibacterial activities. So that we selected the naphtho[2,1-*b*]furan derivatives are chosen in the research work. Many synthetic compound with furan ring skeleton correlated with novel biological activities like *antibacterial*, *antifungal*⁵⁻⁷, *antitumor*⁸⁻⁹, *antiviral*¹⁰, *anti-trypanosomal*, *cytotoxicity*¹¹, and *anthelmintic*.¹² Thiazole is yet another major class of five-membered heterocyclic ring systems. Various naphtho [2,1- *b*]furan derivatives are fused with thiazole moiety and have been prepared and studied for many biological and pharmacological activities such as *antibacterial*, *antifungal*, *diuretic*, *anthelmintic antimicrobial*, *analgesic*, and *anti-inflammatory*.¹³

In this present work in order to study the various antibacterial activities we have described the synthesis of new naphthofuran derivatives of compound (3), and compound (4) used for the synthesis of *N*-(2-[(2*Z*)-2-benzylidenehydrazinyl]carbonyl)-5-nitronaphtho[2,1-*b*]furan-1-yl]acetamide compounds of four derivatives 5 (a-d) and 1-acetamido-5-nitro-*N*-(5-oxo-2-phenylthiazolidin-3-yl)naphtho[2,1-*b*]furan-2-carboxamide compounds of four derivatives 6 (a-d). The prepared compounds were confirmed by NMR, FTIR, and mass, methods. The prepared naphthofuran derivatives were used for the antibacterial activity versus both Gram(+ve) and Gram(-ve) bacteria shows excellent results.

EXPERIMENTAL

Materials and Methods

The top-quality chemicals and solvents were used without purification. The FTIR spectra were recorded by Perkin-Elmer 1600 FT-IR spectrophotometer. $^1\text{H-NMR}$ spectra were measured by Varian Mercury 200 MHz spectrometer. Water's SYNAPT G2 QTOF LCMS instrument was used to record the mass spectra.

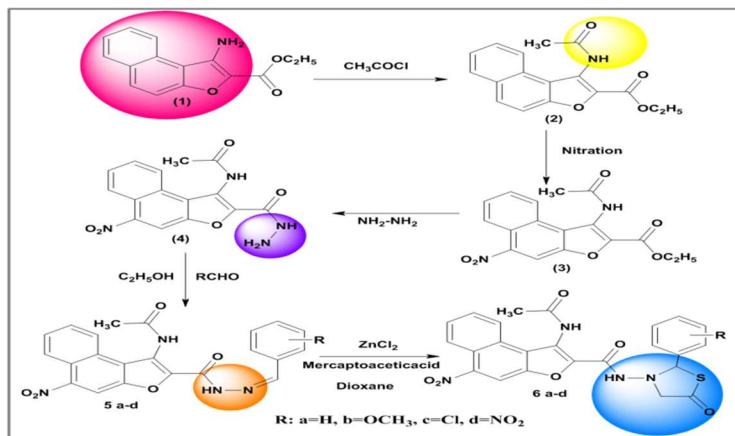
Synthetic route of naphthofuran derivatives.

Synthesis of ethyl 1-aminonaphtho[2,1-b]furan-2-carboxylate (1)

The 2-hydroxy-1-naphthaldoxime (0.80 g, 0.04 M), ethylchloro acetate (6.10 g, 0.04 M) and potassium carbonate (4.2 g, 0.04 M) were refluxed in anhydrous dimethyl formamide (60 ml) at 80-90 $^{\circ}\text{C}$ for 15 h. The obtained product was cooled, the remained potassium salt was filtered and the obtained filtrate was transferred into the ice-cold water to get the light brown colored product. The obtained product was filtered and it was re-crystallized using aqueous ethanol (Yield: 60%).¹⁴

Synthesis of ethyl 1-acetamidonaphtho[2,1-b]furan-2-carboxylate (2)

Compound 1 (2.50 g, 0.01 M) was dissolved in an aq sodium hydroxide (3N, 30 ml) then it was treated with acetyl chloride (6.0 ml) with vigorous stirring. After 30 min, the product was obtained and transferred to ice water. Thus the obtained product was filtered and re-crystallized by ethyl alcohol.¹⁴



Scheme-1: Synthesis of Naphthofuran Derivatives [compound 1, 2, 3, 4, 5(a-d), and 6(a-d)]

Synthesis of ethyl 1-(acetylamido)-5-nitronaphtho [2,1-b]furan-2-carboxylate (3)

Compound 2 (1.75 g, 0.02 M) is mixed with acetic acid (20 ml) at 0-4 $^{\circ}\text{C}$, a cooled mixture of conc. HNO_3 and conc. H_2SO_4 (1:2, 35 mL) was added drop by drop for upto 30 min with constant stirring for about 4.5 h. The product was transferred into ice-cold water, the yellow-colored compound was obtained, and it was filtered and re-crystallized using ethanol to obtain the pure compound (3). 65% yield was recorded. IR (KBr) cm^{-1} : 3610-3256 (amide NH-str), 3148-2945 (C-H, str-aromatic), 1646-1569 (C=O), 1804-1635 (C=C), 2001-1886 (C=N), 1580-1494 (C- NO_2), 664-599 (CH, aliphatic). MS (m/z): $\text{M}+1$ is 342.30.

Synthesis of N-[2-(hydrazinylcarbonyl)-5-nitronaphtho [2,1-b]furan-1-yl]acetamide(4)

The compound (3) (0.02 M), hydrazine hydrate (0.02 M), and ethanol (50 ml) were under reflux at 70-80 $^{\circ}\text{C}$ for 18-20 h. Thus solid obtained was separated and collected. The dry compound was recrystallized by ethanol to get the compound (4). % of yield is 60%, IR (KBr) cm^{-1} : 3644-3273 (amide NH-str), 3141-2901 (C-H, str-aromatic), 1702-1646 (C=O), 1804-1701 (C=C), 1990-1886 (C=N), 1624-1580 (C- NO_2), 669-604 (CH, aliphatic). MS (m/z): M^+ is 328.08.

Synthesis of N-(2-[(2Z)-2-benzylidenehydrazinyl]carbonyl)-5-nitronaphtho [2,1-b]furan-1-yl]acetamide and its derivatives(5a-d)

A solution of substituted aldehydes (a-d) (0.002 M) in ethanol (25 ml) was transferred into the solution of compound (4) in DMF (20 ml) separately. The reaction mixture was refluxed at 90-95 $^{\circ}\text{C}$ for 8-9 h. The product was transferred into ice-cold water to get a good yield. This crude product was filtered and re-

Synthesis of 1-acetamido-5-nitro-N-(5-oxo-2-phenylthiazolidin-3-yl) naphtha [2,1-b]furan-2-carboxamide and its derivatives(6a-d)

The compounds (**5a-d**) (0.045 M), mercapto acetic acid (0.045 M) and anhydrous zinc chloride (catalytic amount), and 1-4 dioxane (45 ml), were refluxed at 80-90 °C for 16-18 h, the obtained product was cooled and poured into sodium bicarbonate solution for removal of excess mercapto acetic acid. Filtered the obtained product and it was recrystallized with ethanol to get the compound (**6a-d**). % of yield is 65%, IR (KBr) cm^{-1} : 3687-3109 (amide NH-str), 2994-2781 (C-H, str-aromatic), 1575-1494 (C=O), 1701-1592 (C=C), 2159-2061 (C=N), 1504-1346 (C-NO₂), 915-806 (CH, aliphatic). MS (m/z): M+1 (Table-1).

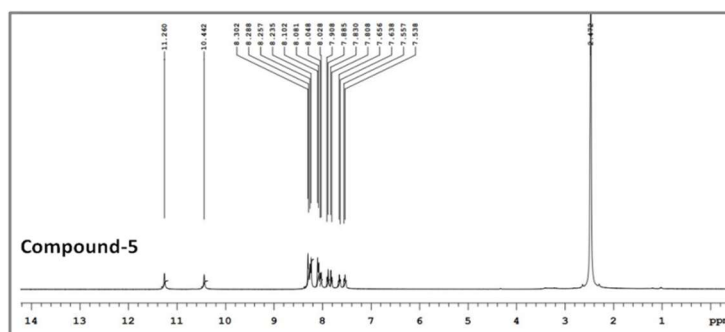


Fig.-3: NMR spectra of *N*-(2-[[*(Z)*-(2-benzylidenehydrazinyl)carbonyl]-5-nitronaphtho[2,1-*b*]furan-1-yl)acetamide and its derivatives(**5a**)

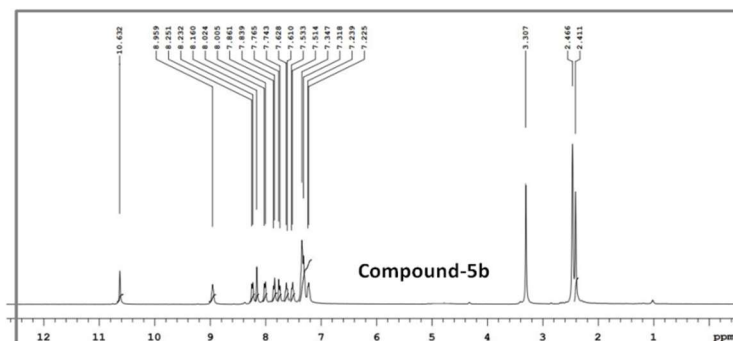


Fig.-4: NMR spectra of *N*-(2-[[*(Z)*-(2-benzylidenehydrazinyl)carbonyl]-5-nitronaphtho[2,1-*b*]furan-1-yl)acetamide(**5b**)

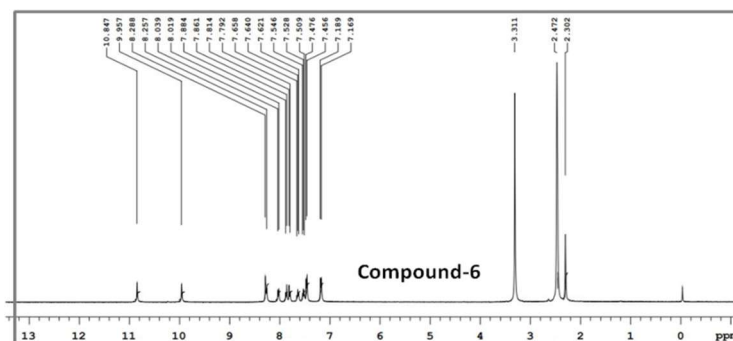


Fig.-5: NMR spectra of 1-acetamido-5-nitro-*N*-(5-oxo-2-phenylthiazolidin-3-yl)naphtha[2,1-*b*]furan-2-carboxamide(**6a**)

FTIR-Spectra

The FT-IR spectrum of compound 3, 4, 5a, and 6a (Fig.5)¹⁵⁻¹⁷

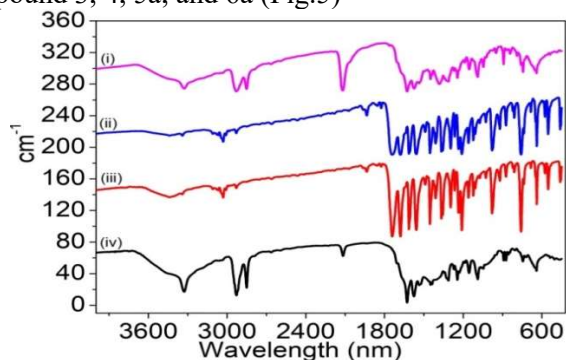


Fig.-6: The FTIR spectra of (i)Compound (5a), (ii) Compound 3, (iii) Compound 4, and (iv) Compound (6a)

IR (KBr) cm^{-1} . Compound (**3**): 3610-3256 (amide NH-str), 3148-2945 (aromatic C-H), 1646-1569 (C=O), 1804-1635 (C=C), 2001-1886 (C=N), 1580-1494 (C-NO₂), 664-599 (CH, aliphatic). Compound

(4): 3644-3273 (amide NH-str), 3141-2901 (aromatic C-H), 1702-1646 (C=O), 1804-1701 (C=C), 1990-1886 (C=N), 1624-1580 (C-NO₂), 669-604 (CH, aliphatic). Compound (5a): 3703-3110 (amide NH-str), 2995-2789 (aromatic C-H), 1498-1418 (C=O), 1749-1602 (C=C), 2173-1978 (C=N), 1591-1532 (C-NO₂), 802-669 (CH, aliphatic). Compound (6a): 3687-3109 (amide NH-str), 2994-2781 (aromatic C-H), 1575-1494 (C=O), 1701-1592 (C=C), 2159-2061 (C=N), 1504-1346 (C-NO₂), 915-806 (CH, aliphatic).

Mass Spectra

The confirmation of the prepared compound by mass spectroscopy method, which measures the mass-to-charge ratio [M+2] of ions. The actual mass of compounds 3, 4, 5a, and 6a are 342.30, 328.08, 416.11, and 490.09, and the experimental mass of compound 3, 4, 5a, and 6a exhibits a molecular ion peak at 340.66, 328.12, 414.15, and 489.10 (Figs.-7, 8, 9, 10).

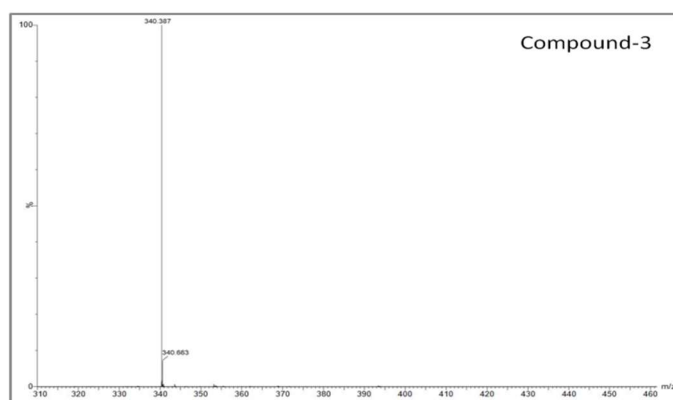


Fig.-7: Mass spectra of ethyl 1-(acetamidido)-5-nitronaphtho[2,1-*b*]furan-2-carboxylate (3)

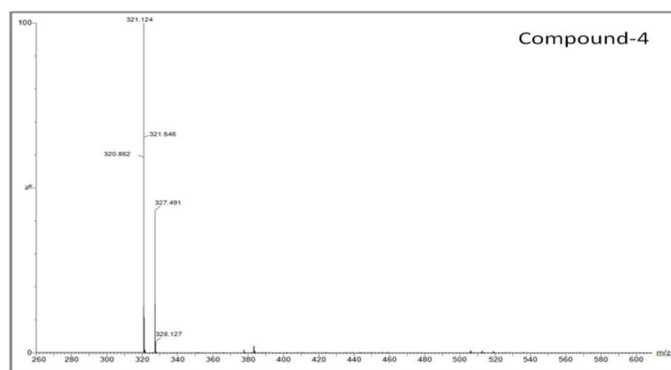


Fig.-8: Mass spectra of *N*-[2-(hydrazinylcarbonyl)-5-nitronaphtho[2,1-*b*]furan-1-yl]acetamide (4)

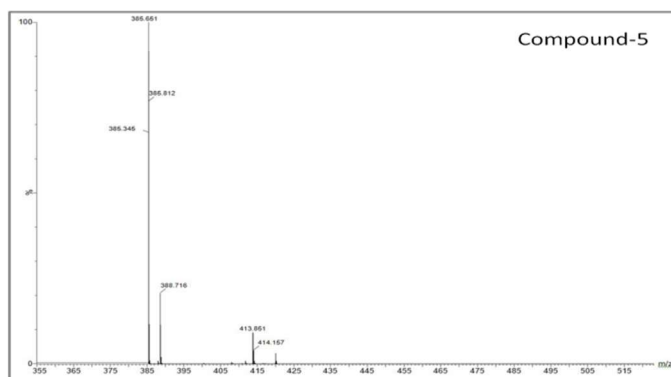


Fig.-9: Mass spectra of *N*-(2-{[(2*Z*)-2-benzylidenehydrazinyl] carbonyl}-5-nitronaphtho [2,1-*b*]furan-1-yl)acetamide and its derivatives (5a)

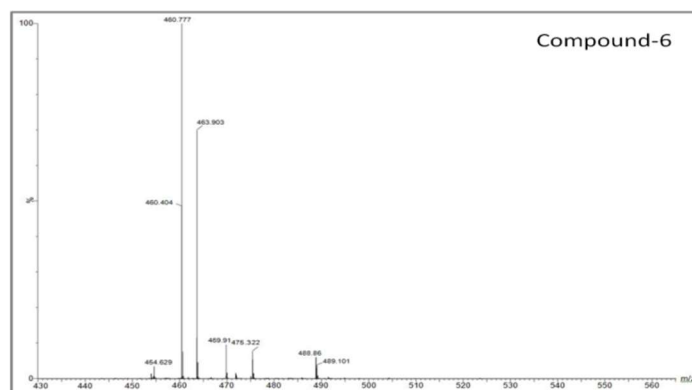


Fig.-10: Mass spectra of 1-acetamido-5-nitro-N-(5-oxo-2-phenylthiazolidin-3-yl)naphtha[2,1-b]furan-2-carboxamide(**6a**)

Elemental Analysis

Vario EL (III) C.H.N.S analyzer was used to study of elements in the prepared compounds and their composition percentage. The Compounds 3, 4, 5a-d, and 6a-d, were examined by decomposing a known quantity of the compounds with H_2SO_4 and HNO_3 mixture, followed by careful evaporation and calcinations Table-1.

Table-1: Elemental analysis of the synthesized compounds

Comp.	R_2	Molecular Formula	Molecular Weight	Yield %	Elemental Analysis					
					C	H	N	O	Cl	S
3	-	$\text{C}_{17}\text{H}_{14}\text{N}_2\text{O}_6$	342.30	65	59.63	4.10	8.17	28.01	-	-
					(59.65)	(4.12)	(8.18)	(28.04)		
4	-	$\text{C}_{15}\text{H}_{12}\text{N}_4\text{O}_5$	328.08	60	54.82	3.64	17.05	24.34	-	-
					(54.88)	(3.68)	(17.07)	(24.37)		
5a	H	$\text{C}_{22}\text{H}_{16}\text{N}_4\text{O}_5$	416.11	65	63.44	3.85	13.43	19.19	-	-
					(63.46)	(3.87)	(13.46)	(19.21)		
5b	p-OCH ₃	$\text{C}_{23}\text{H}_{18}\text{N}_4\text{O}_6$	446.12	70	61.84	4.00	12.52	21.47	-	-
					(61.88)	(4.06)	(12.55)	(21.50)		
5c	p-Cl	$\text{C}_{22}\text{H}_{15}\text{N}_4\text{O}_5\text{Cl}$	450.07	58	58.60	3.33	12.40	17.72	7.83	-
					(58.61)	(3.35)	(12.43)	(17.74)	(7.86)	
5d	p-NO ₂	$\text{C}_{23}\text{H}_{18}\text{N}_4\text{O}_6$	446.12	62	61.84	4.03	12.52	21.46	-	-
					(61.88)	(4.06)	(12.55)	(21.50)		
6a	H	$\text{C}_{24}\text{H}_{18}\text{N}_4\text{O}_6\text{S}$	490.09	71	58.76	3.68	11.40	19.53	-	6.51
					(58.77)	(3.70)	(11.42)	(19.57)	-	(6.54)
6b	p-OCH ₃	$\text{C}_{25}\text{H}_{20}\text{N}_4\text{O}_7\text{S}$	520.10	63	57.67	3.85	10.72	21.50	-	6.16
					(57.69)	(3.87)	(10.76)	(21.52)	-	(6.16)
6c	p-Cl	$\text{C}_{24}\text{H}_{17}\text{N}_4\text{O}_6\text{SCl}$	524.05	64	54.90	3.22	10.64	18.25	6.72	6.10
					(54.91)	(3.26)	(10.67)	(18.29)	(6.75)	(6.11)
6d	p-NO ₂	$\text{C}_{24}\text{H}_{17}\text{N}_5\text{O}_8\text{S}$	535.08	56	53.82	3.18	13.06	23.86	-	5.95
					(53.83)	(3.20)	(13.08)	(23.90)	-	(5.99)

Antibacterial Activity (Agar Diffusion Method)

The newly synthesized compounds were used for the study of *antibacterial activities*. The four different strains of bacteria are used to find the [Gram(+ve) and Gram(-ve)] using the agar diffusion technique. The well-known described cup-plate method was used to determine the *antibacterial activity*. It involves cups of std diameter, the nutrient agar medium, and std bacterial inoculum. The test samples were subjected to the cups and the diameters of the zones of inhibition were recorded. *Escherichia coli* [Gram(-ve)], *staphylococcus aureus* [Gram(+ve)], and *pseudomonas* [Gram(-ve), *streptococci* [Gram(+ve)] were used test for *antibacterial activity* by prepared compounds. The obtained results are shown in the Table-2.

The mean zone of inhibition is including bore diameter (8 mm).

$$\text{Activity index} = \frac{\text{Testcompound}}{\text{Standardcompound}}$$

The prepared compound has been studied for its *antibacterial activity* at concentrations of 50 µg/ml and 100 µg/ml by agar diffusion std method, verses below given bacteria's:

- (1) (a) *Streptococci*(G+ve) (b) *Staphylococcus aureus*(G+ve).
 (2) (a) *Escherichia coli*(G-ve) (b) *Pseudomonas*(G-ve).

All the compounds exhibit good activity and the compounds are fully active against both (G+ve) and (G-ve) organisms. The prepared samples 4, 5a, 5b, 5c, 6a, 6b, 6d, and 6e result in maximum antibacterial activity at lower to higher concentrations (50-100 µg/ml) versus *Streptococci* (G+ve), related to std *procaine penicillin drug*. The samples 5c, 5f, 6b, 6c, and 6f exhibit *auspicious antibacterial activity* against *pseudomonas* (G-ve), bacteria when compared with relatively standard *streptomycin drugs*. The compounds 3, 5d, 5f, 6f, 6c, and 5c, exhibit adequate activity against *Staphylococcus aureus* (G+ve) bacteria compared to std *procaine penicillin drug*. *Scrutiny* of Table-2 shows that compounds show relatively high in their inhibitory action showed versus *Escherichia coli*(G-ve) bacteria when compared with relatively standard *streptomycin drugs*.

Table-2: Antibacterial Activity of Compounds 3, 4, 5a-d, and 6a-d

S. No.	Compound	Mean zone of inhibition in (mm)							
		<i>Staphylococcus aureus</i> (G+ve)		<i>Pseudomonas</i> (G-ve)		<i>Streptococci</i> (G+ve)		<i>Escherichia coli</i> (G-ve)	
		50 µg	100 µg	50 µg	100 µg	50 µg	100 µg	50 µg	100 µg
1	3	11 (0.51)	12 (0.55)	12 (0.56)	14 (0.61)	12 (0.63)	14 (0.69)	12 (0.63)	13 (0.70)
2	4	13 (0.60)	15 (0.64)	13 (0.62)	15 (0.65)	14 (0.62)	16 (0.68)	13 (0.60)	15 (0.63)
3	5a	15 (0.66)	19 (0.80)	15 (0.74)	17 (0.81)	15 (0.71)	19 (0.83)	11 (0.52)	13 (0.56)
4	5b	12 (0.55)	16 (0.67)	14 (0.70)	16 (0.72)	12 (0.56)	13 (0.61)	12 (0.61)	15 (0.68)
5	5c	12 (0.55)	15 (0.65)	12 (0.61)	14 (0.68)	13 (0.60)	15 (0.63)	13 (0.64)	15 (0.72)
6	5d	14 (0.62)	15 (0.70)	10 (0.52)	12 (0.60)	14 (0.65)	16 (0.70)	12 (0.62)	15 (0.68)
7	6a	12 (0.55)	15 (0.64)	13 (0.62)	15 (0.66)	15 (0.72)	17 (0.76)	14 (0.72)	16 (0.75)
8	6b	14 (0.62)	18 (0.66)	15 (0.74)	17 (0.78)	14 (0.63)	15 (0.69)	13 (0.62)	15 (0.65)
9	6c	14 (0.62)	15 (0.69)	13 (0.64)	15 (0.71)	12 (0.54)	15 (0.57)	12 (0.58)	13 (0.63)
10	6d	12 (0.55)	16 (0.61)	13 (0.60)	16 (0.68)	13 (0.60)	14 (0.67)	14 (0.71)	15 (0.78)

Std: *Streptomycin* and *Procaine Penicillin*.

CONCLUSION

The compounds of ethyl 1-(acetylamino)-5-nitronaphtho[2,1-*b*]furan-2-carboxylate (3), and *N*-[2-(hydrazinylcarbonyl)-5-nitronaphtho[2,1-*b*]furan-1-yl]acetamide (4) used for the synthesis of *N*-(2-[(2*Z*)-2-benzylidenehydrazinyl]carbonyl}-5-nitronaphtho[2,1-*b*]furan-1-yl)acetamide compounds of four derivatives 5(a-d) and 1-acetamido-5-nitro-*N*-(5-oxo-2-phenylthiazolidin-3-yl)naphtho[2,1-*b*]furan-2-carboxamide compounds of four derivatives 6(a-d). All these newly synthesized compounds were confirmed by NMR, Mass, and FTIR methods. The synthesized naphthofuran derivatives were used for the study of *antibacterial activity* against both Gram(+ve) and Gram(-ve) bacteria shows excellent results. Therefore the synthesized compounds are used for antibacterial studies in the medical field.

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