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ORIGINAL RESEARCH ARTICLE

Synthesis and biological studies of some novel furo naphthyridine compounds contain chalcone

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ABSTRACT

Through a Vilsmeier–Haack condensation 2-chloro-3-formyl-1.8-nathyridine (I) was synthesized. When treated with aqueous hydrochloric acid converted to compound (II) was successfully obtained in high yield and then converted to Novel 1-(8-methyl) furo[2,3-b] -(1,8-naphthyridine-2-yl) ethenone (III) through Claisen –Schmidt condensation. The condensation between compound III (ketone) and benzaldehyde yield Novel (furo[2,3-b] (1,8-naphthyridine-2-yl)-2-acetyl called chalcone (4). The reaction between chalcone and bromine water yields dibromide (6), Iodo chalcone (5) produced by treatment of chalcone with one to two pieces of crystal iodine in dimethyl sulfoxide. Spectral analysis techniques such as 1H-NMR, FT-IR were employed to identify and confirm the structural formula of all products. The synthesized compounds (3-6) were evaluation for their anti- bacterial activity against both gram negative and gram-positive bacteria, demonstrating significant compared to standard drug. All these compounds showed moderate activity.

Keywords: furo2,3-b; chalcone derivatives; Vilsmeier–Haack reaction; Claisen–Schmidt condensation; antibacterial activity; dibromide; iodo chalcone

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Highlight

- Novel furo2,3-b chalcone derivatives were synthesized via multi-step reactions.
- Structures were confirmed by IR and 1H NMR spectroscopy.
- Halogenated derivatives (dibromo and iodo chalcones) were successfully prepared.
- Compounds 3–6 exhibited moderate antibacterial activity against Gram-positive and Gram-negative bacteria.
- The results suggest these scaffolds as promising leads for antimicrobial drug development.

1. Introduction

Naphthyridine compounds have played a significant role in heterocyclic chemistry, where their mixed acid-base properties have altered the expected products as they react with electrophilic and cyclophilic agents, and through the carbon and nitrogen atoms. The chemistry of naphthyridine compounds have been of increasing interest snice these compounds have been found to be useful as

chemotherapeutic agent against malaria[1], bacteria[2] and widely distributed in nature as essential to life fund as potential[3]. The hetero cyclic compounds that contain nitrogen and oxygen are one of the most extensive compounds that show diverse pharmacological activities. The chalcones derivatives of these groups show activities as anti-tuberculosis agent[4,5] as important as natural products belonging to flavonoid family[6]. Chalcones are precursors in the preparation of many important hetero cyclic such as flavones[7], benzo thiazepine[8] pyrazolines[9]. Diels-Alder cyclization was the most important method used to synthesis naphthyridine compounds[10] 1,8-naphthyridine and tri ethylene glycol ether-Linked are used as important binding unit in molecular design of synthetic receptors[11] and the compound fluro benzyl)-3-(2-tolyl)-1,8-naphthyridine (1H)-2-one are used in memory treatment in Al-Zheimer's disease[12]. Now a day, the quinoline compounds are used as starting materials for synthesis of many fused naphthyridine compounds[13,14] new dibromide and iodo naphthyridine compounds have been prepared through selective iodination and bromination of chalcones[15]. Bacteria and fungi that are resistant to antibiotics require ongoing research and development of new antibacterial agents through the synthesis of novel chemicals[16]. It also possesses many therapeutic properties and a wide spectrum of antibacterial, antifungal and antiviral properties, and has played an important role in many cancer treatments and chronic diseases. In our work, a mouse ring was synthesized on synthetic naphthyridine via the Vilsmeier–Haack reaction, which contained a terminal acetyl group at position 2 of the furan ring, which was converted to a chalcone, and the products had good biological effects. This study aimed to synthesize novel furonaphthyridine derivatives and characterized and identify by using IR, ^{1}H-NMR and then studies their antibacterial activity against Gram positive and negative bacteria.

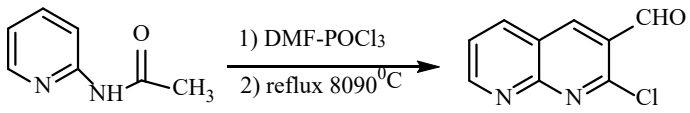
2. Experimental

The melting point was measured on electro-thermal CA9300 apparatus and uncorrected. The infrared were measured on IR spectrophotometer model Tensor 27, Bruker Co., Germany by using KBr aiscs. The ^{1}HNMR are used identify the structure on a JEol 400MHZ, using TMS as internal standard.

Synthesis of 2-chloro-3-formyl -1,8-naphthyridine (1)[17,18]

A solution of N-(pyridine-2-yl) acetamide

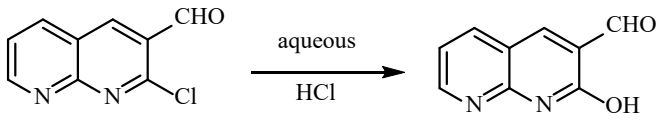
A round-bottom flask with a dropping funnel filled with (14 mol) of $POCl_3$ filled with a mixture of (2 mol) and 6 mol of dry dimethyl form amide. The circular was submerged in an ice bath between 0 and 50 degrees Celsius. After maintaining the temperature, $POCl_3$ was added drop by drop while stirring continuously. The dropping funnel was then removed, and the reaction mixture was refluxed at 80 to 900 degrees Celsius for 16 hours. The mixture was then poured into cold water and stirred for one hour, after which the precipitation was filtered, cleaned with cold water, and the crystals were recrystallized using methanol.



The yield was 60%, the m.p. was determined to be between 164 and 1660C, and the hue was light yellow. IR (KBr, cm^{-1}): 3055 (Ar-H),2720 (CHO), 1685 (C=O), 1565 (C=N), 775 (C-Cl), ^{1}HNMR (400 MHZ, DMSO-d6, ppm) 10.54 (S,1H,CHO), 8.45 (d,1H,C7), 7.86 (S,1H,C4), 7.45 (d,1H,C5), and 7.27 (t, 1H,C6).

Synthesis of 2-hydroxy-3-formyl-1,8-naphthyridine (2)

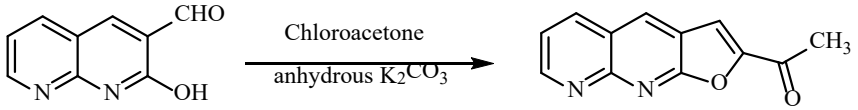
A mixture of compound (1) (0.01mol) and (35mL, 4mol) of aqueous hydrochloric acid was heated and reflux for (6 *hrs*.) then allowed to cool to room temperature. The mixture was poured onto ice cold water; the solid was separated and recrystallized from aqueous acetic acid.



The yield was 70% yellow, and the m.p. was determined to be between 151 and 153ºC. IR (KBr , V,cm^{-1}): 1690(C=O), 1665(C=N) , 3065 (Ar-H) and 3230(OH). ^{1}HNMR (400MHZ, DMSO-d6,δ,ppm). 8.77(s,1H,C=O), 8.21(d,1H,C7), 8.05(s,1H,C4), 7.18(d,1H,C5), 7.02(m,1H, C6) and 15.6(s,1H, OH).

Synthesis of (furo[2,3-b](1,8-naphthyridine-2-yl)-2- acetyl (3)

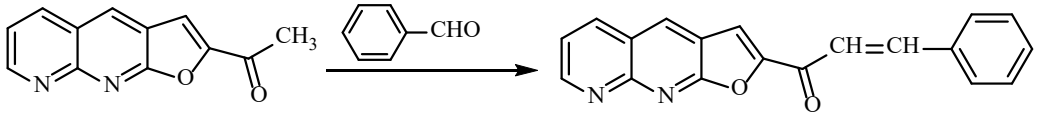
Compound (2) (0.1mol) chloroacetone (0.1mol) and anhydrous potassium carbonate (0.1mol) in (80ml) of dimethyl formamide was stirred for (6*hrs*.) at room temperature. Then mixture was reflux on water bath for 24*hrs*. to (70-90 ^{0}C) Then allowed to cool at room temperature then the mixture was poured into crush ice, then precipitate was filtered dried and recrystallized by used DMF.



The m.p was found (125-127^{0}C), the yield 50% yellow brown. IR(KBr,V,cm^{-1}) :1710(C=O), 2976(CH), 1655(C=N) and 1120(C-O-C), ^{1}HNMR (400MHz, DMSO-d6,δ,ppm). 2.99(s,3H,CH_3), 6.99(s,1H, furane), 7.15(t,1H,C6), 8.07(s,1H,C4), 8.21(m,1H,C5) and 8.78(d,1H,C7).

Synthesis of 1-furo[2,3-b]-(1,8-naphthyridine-2-yl)ethanone derivative (chalcone) (4)[19]

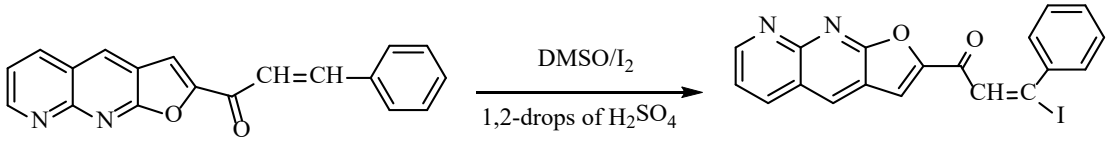
A mixture of compound (3)(0.01mol) and benzaldehyde (0.01mol) in ethanol (70mL) in the presence of aqueous potassium hydroxide (40%,15mL) is stirred at room temperature for (24*hrs*.). Then the reaction mixture was cooled for (10*hrs*.) and then poured into (300mL) ice cold water, acidified with dilute HCl, the solid was separated then air dried and recrystallized using ethanol.



The m.p found(178-180^{0}C), the yield (70%) color pale brown. IR(BKr, V, cm^{-1}): 1685(C=O), 1650(C=N). 1565(C=C),1120(C-O-C) and 1650(C=N). ^{1}HNMR (400MHZ-d6,δ, ppm) 6.53(d, CH, ethylene), 7.23-7.40(m,6H, CH furane benzene ring), 8.11(s,1H,C4), 8.67-8.68(d, 1H, C5), 7.30-7.32(d,1H,C7) and 7.41-7.43(t,1H,C6).

Synthesis of dibromide (5)[20,21]

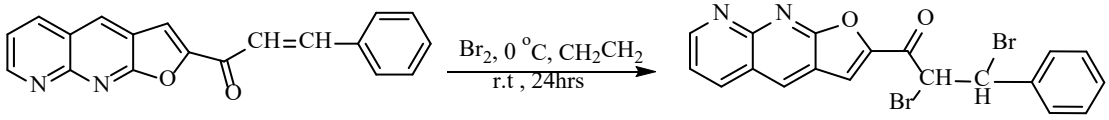
Bromine solution (0.1mol) was added dropwise over 30 minutes to cold solution of compound (4) in (15mL) of dichloromethane the mixture stirred overnight, then the solvent reduced to half by evaporator. The precipice was collected and recrystallized by using chloroform.



The m.p. is (203-205^{0}C), color brown, the yield 70%, IR (KBr,V, cm^{-1}), 1695(C=O), 1650($^{C=N}$),1125 (C-O-C), 3225(Ar-H). ^{1}HNMR(400MHZ,DMSO-d6, δ,ppm). 4.20-4.32(m,2H, 2-furane, CH-Br), 6.71(d,1H,CH), 7.59-7.92(m,6H, benzene, C6), 7.92(d, 1H, C5), 8.56(s, 1H, C4), 7.48-7.49(d, 1H,C7).

General procedure for synthesis of iodo chalcones(6)[22,23]

Two drops of sulfuric acid were added to the combination of chalcone (4) (3 mmoles) in 15 mL of DMSO after an iodine crystal (1,2) was added. After the mixture refluxed for five hours, it was dumped into cold water and agitated for two hours. The precipitate was filtered, cleaned with a five percent sodium thiosulphate solution, and then recrystallized using ethanol and twenty-five milliliters of cold water.



The m.p (227-229^{0}C), yield (50%), color yellow. IR (KBr, V, cm^{-1}): 1668(C=O), 1495(C=C), 1560(C=N) and 3150(Ar-H). ^{1}HNMR (400MHZ, DMSO-d6-δ, ppm). 7.23-7.55(m,8H, phenyl ring, 2-furan, CH=, C6), 8.15(s,1H, C4), 8.23(d, 1H, C5) and 8.52(d, 1H, C7).

All synthesized compounds are shown in **Figure (1)** & The physical and spectral characteristics are shown in **Tables (1-2)**.

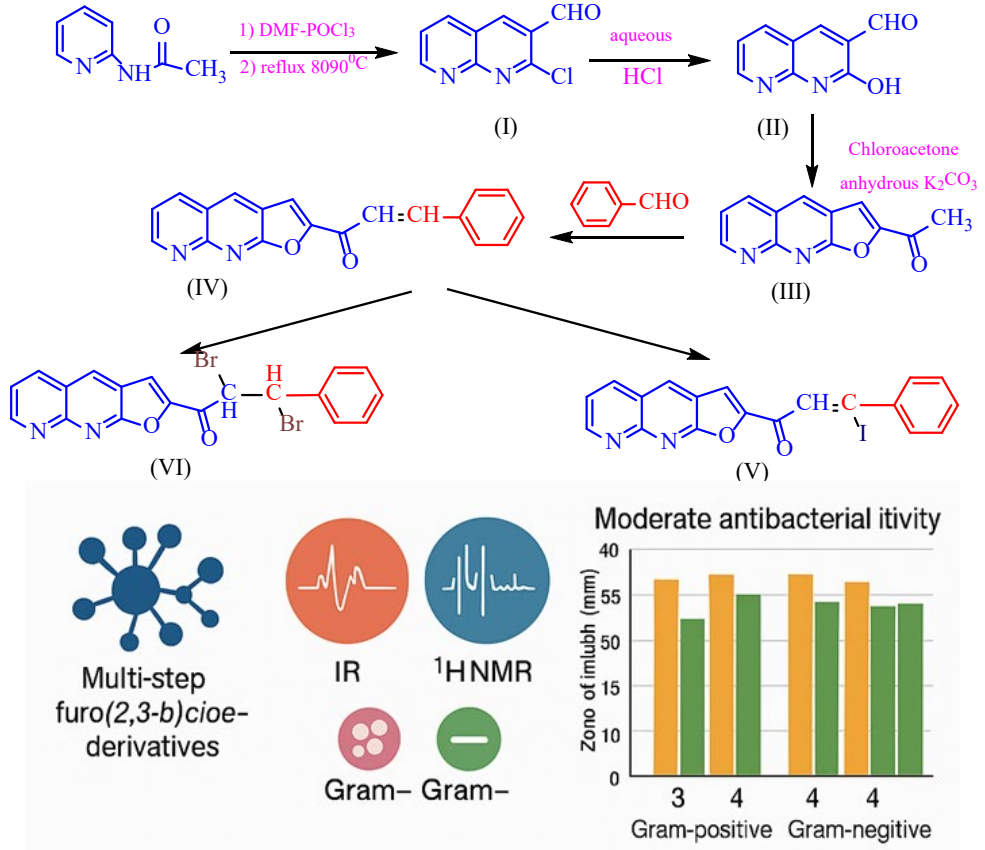


Figure 1. Synthesis of furonaphthyridine derivatives (I-VI).

3. Result and discussion

Naphthyridine may be made by using a variety of techniques, although the Vilsmeier approach is the most crucial for creating substituted naphthyridine[24,25]. In this study, 2-chloro-3-formyl-1,8-naphthyridine (1) was made by reacting to N-(pyridine-2-y)-acetamide with Vilsmeier reagent. POCl3 was added to the substrate in DMF at a temperature between 0 and 50 degrees Celsius and refluxing it for one hour. The **Figure (2)** describes how the response mechanism operates[26]:

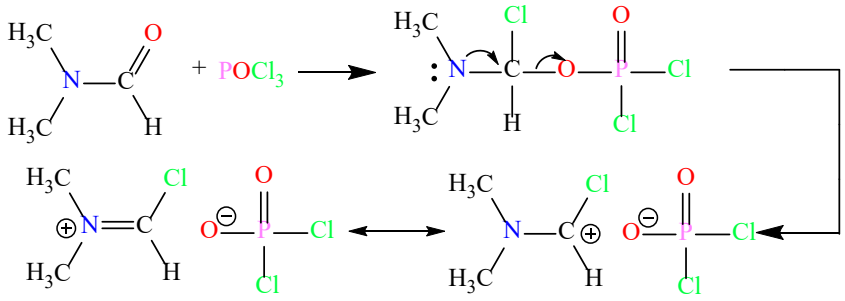


Figure 2. The mechanism of formation vilsmeier – Haack reagent.

Compound (1) IR spectra revealed a distinctive absorption band at 1685 cm^{-1} for C=O, 1565 cm^{-1} for C=N, and 775 cm^{-1} for C-Cl. The molecule's (1) 1HNMR spectra revealed the following notable peaks: a singlet band at 10.54 ppm for aldehyde proton (CHO), at 7.86 ppm for H-4, a doublet at 8.45 ppm for H-7, at 7.45 ppm for H-5, and a triplet at 7.27 ppm for H-6 table(1,2). Compound (2) was created by refluxing these chemicals (1) in diluted aqueous hydrochloric acid. The IR spectrum for these compounds showed a characteristic band at 1690 cm^{-1} for carbonyl group. The ^{1}HNMR for compound (2) showed a singlet at 8.77ppm for (CHO) group and at 5.60 ppm for OH group and between (7.02-8.70) for proton of naphthyridine ring table(1,2). The compound (3) was prepared from reaction of compound (2) with chloro acetone and potassium carbonate. The IR spectra for theses compound showed many band at 1710cm^{-1} for (C=O) group and at 1655cm^{-1} (C=N) group and at 1120cm^{-1} for (-C-O-C-) group. The ^{1}HNMR spectra for these compounds showed three singlet signal at 2.99 ppm for CH_3 group and at 6.99 ppm for furane proton and at 8.07ppm for C4 proton for naphthyridine ring table(1,2)[27]. Clasen-Schmidt condensation[28] used to prepared compound (4) from reaction of compound (3) with benzaldehyde in ethanolic solution[19,20]. These compounds' (4) infrared spectra revealed a prominent band at 1685 cm^{-1} for the (C=O) group and 1565 cm^{-1} for the (C=C) group. The ^{1}HNMR spectra of compound (4) revealed three signals: a doublet at 6.53 ppm, 8.67-8.68 ppm, 7.30-7.32 ppm for the α-CH proton of ethylene and for (C5,C7) for the naphthyridine ring, a singlet at 8.11 ppm for the C4 proton for the naphthyridine ring, a multipleted at 7.23-7.40 ppm for the benzene proton and CH Furane, and a triplet at 7.41-7.43 for the naphthyridine ring table(1,2). Compound (4) was brominated to produce dibromide (5).

The reaction done by adding 0.1ml of bromine to α,β-unsaturated compound (5) in methane dichloride drop wise in an ice bath under string for 1/2*hr*.[29]. The IR spectra for compound (5) showed absorption at 1695cm^{-1} for (C=O) group and at 1125cm^{-1} for (C-O-C) group. The ^{1}HNMR for dibromide showed a singlet signal at 8.56 ppm for C4 and two doublet at 7.92 and 7.48-7.49 for C5, C7 and multipleted band at 4.20-4.32 ppm for furane and CHBr proton) table(1,2). Dimethyl sulfoxide in iodine is used as and iodinating agent for α,β–unsaturated ketone act as an oxidizing agent. The compound (6) is synthesized from adding one crystal of iodine to solution of compound (6) then acidified with two drops of H_2SO_4[20]. Then the solution was refluxed for (1*hr*.)[30], the IR spectra of compound (6) showed a strong absorption at 1685cm^{-1} for (C=O) group and at 1565cm^{-1} for (C=C) group and at 1120$cm^{-1}$for (C-O-C) group. The ^{1}HNMR for compound (6) showed a doublet at 6.76ppm for CH of ethylene and multipleted signal at 7.23-7.55ppm for (benzene ring and CH of ethylene and C6 and CH furane) and singlet at 8.15 for C4 and two doublet at (8.23, 8.52ppm) for (C5,C7) table(1,2).

Table 1. Physical and IR spectra data of compounds(I-VI).

Com. No.	Color	M.P.	Yield	IR data of compounds (cm^{-1})								
				C=O	CHO	C=N	C-Cl	Ar-H	OH	C-O-C	C-H	C=C
1	Pale Yellow	164	60%	1685	2720	1565	755	3035	--	--	--	--
2	Yellow	151	70%	1690	2720	1560	--	3065	3230	--	--	--
3	Yellow brawn	125-127	50%	1710	--	1555	--	3055	--	1120	2976	--
4	Pale Yellow	178-180	70%	1685	--	1550	--	3055	--	1120	--	1565
5	Brawn	203-205	70%	1695	--	1550	--	3060	--	1125	--	1580
6	Yellow	227-229	50%	1668	--	1560	--	3150	--	1125	--	--

Table 2. ^{1}H-NMR spectra data of compounds(I-VI).

Comp. No.	^{1}H-NMR Chemical shift (DMSO-d6) δ , ppm
1	10.54(s,1H,CHO), 8.45(d,1H,C_7), 7.86(s,1H,C_4), 7.45(d,1H,C_5), 7.27(t,1H,C_6)
2	8.77(s,1H,CHO), 8.21(d,1H,C_7), 8.05(s,1H,C_4), 7.18(d,1H,C_5), 7.02(t,1H,C_6), 5.60(s,1H,OH)
3	2.99(s,3H,CH_3), 6.99(s,1H,Furan), 7.15(t,1H,C_7), 8.07(s,1H,C_4), 8.21(m,1H,C_5), 8.78(d,1H,C_6)
4	6.53(d,CH, ethylene), 7.23-7.40(m,H-Furan,benzene ring), 7.30-7.32(d,1H,C_7), 8.11(s,1H,C_4), 8.67-8.68(m,1H,C_5), 7.41-7.43(d,1H,C_6)
5	7.20-7.32(m,2H,Furan,CH-Br), 7.56-7.92(m,6H, benzine ring &C_6), 7.92(d,1H,C_5), 8.56(s,1H,C_4), 7.48-7.49(d,1H,C_7)
6	7.23-7.55(m,8H, benzene ring, Furan, CH= & C_6), 8.15(s,1H,C_4), 8.23(d,1H,C_5), 8.52(d,1H,C_7)

Antibacterial activity

Anti-bacterial activity of new compounds was evaluated via paper disc-agar diffusion technique[30] Ampicillin was used as standard for comparison. Anti-bacterial test was carried out by agar diffusion technique[31,32] by measuring the diameter of zone of inhibition against test organisms at the end of (24h) at 37^{o}C.

The compounds 3 and 6 showed inhibitory value against *Staphylococcus aureus* ranging at (18, 16) mm respectively and these value were less than the value of control factor antibiotics while compounds (4, 5) showed high inhibitory activity against the same bacteria the value of which was (20) mm which was very similar to an antibacterial effect of antibiotic ampicillin. The compounds (3, 4, 5) showed inhibitory value against the Grame negative bacteria *E. Coli* which ranged in value (16, 14) mm respectively , these values less than the control factor (18) mm while the compound (6) showed high inhibitory activity against *E. ccoli.*The compounds (3, 4 , 5 , 6) showed inhibitory value against *staphylococcus epidermis* (18, 18, 16, 12) respectively and these values were less than the value of control while all compound showed inhibitory vale against *proteus vagaries* less than the value of control. The results of biological tests are shown in **Table (3)**.

Table 3. Anti-bacterial activity data for compounds.

Compounds NO	Staphylococcus aurous	Staphylococcus epidermise	E.coli	Proteus vagaries
Ampicillin	20	20	18	18
Compound 3	18	18	16	12
Compound 4	20	18	16	14
Compound 5	20	16	14	12
Compound 6	16	12	18	14

4. Conclusion

The present study successfully synthesized a series of novel furor 2,3-b derivatives, including chalcone, dibromide, and iodo chalcone compounds, through multi-step reactions involving Vilsmeier–Haack condensation and Claisen–Schmidt condensation. The synthesized compounds were structurally confirmed by IR and ^{1}H NMR spectroscopy. Biological screening revealed that compounds (3–6) exhibited moderate antibacterial activity against both Gram-positive and Gram-negative bacteria, indicating their potential as promising scaffolds for future antimicrobial drug development.

Acknowledgment

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Conflict of interest

The authors declare no conflict of interest.

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