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SYNTHESIS OF NOVEL BENZAZEPINE DERIVETIVE AND THERE BIOLOGICAL SCREENING

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Abstract:
This study focuses on the synthesis and comprehensive characterization of novel benzazepine derivatives, a class of compounds with potential pharmaceutical applications. The synthesis was carried out using [2-amino-3-(4-bromobenzoyl) phenyl]acetic acid (1) and a diverse range of ketones as starting materials. The reaction was performed under optimized conditions in aqueous sodium hydroxide solution to obtain the desired benzazepine derivatives with high yield and purity. A series of new compounds (5a to 5i) were successfully synthesized through this method, showcasing the versatility and efficiency of the synthetic approach. The newly synthesized benzazepine derivatives were thoroughly characterized using a combination of advanced spectroscopic techniques. Infrared (IR) spectroscopy was employed to identify key functional groups and structural features. Mass spectrometry provided accurate molecular mass information and fragmentation patterns, confirming the molecular composition of the synthesized compounds. Proton nuclear magnetic resonance (1H-NMR) spectroscopy was utilized to elucidate the detailed structural arrangements and confirm the presence of specific proton environments within the molecules. The structural elucidation of these newly synthesized derivatives provides valuable insights into their chemical properties, including electronic distribution, bond characteristics, and potential reactivity. This comprehensive characterization not only confirms the success of the synthetic methodology but also lays the foundation for understanding the structure-activity relationships of these compounds. Furthermore, this research significantly contributes to the expansion of the benzazepine scaffold, a structural motif of considerable interest in medicinal chemistry. The diversity of the synthesized compounds (5a to 5i) demonstrates the potential for generating a library of structurally related benzazepine derivatives with varied substituents. This structural diversity may have important implications for pharmaceutical development, as it allows for the exploration of a wider chemical space in the search for compounds with desirable biological activities. The successful synthesis and characterization of these novel benzazepine derivatives open up new avenues for further research in medicinal chemistry. These compounds may serve as potential lead structures for the development of new therapeutic agents, particularly in areas where benzazepine-based drugs have shown promise, such as in the treatment of neurological disorders or as anti-inflammatory agents. In conclusion, this study presents a robust synthetic methodology for novel benzazepine derivatives, coupled with comprehensive spectroscopic characterization. The findings contribute significantly to the field of heterocyclic chemistry and provide a solid foundation for future investigations into the biological activities and potential pharmaceutical applications of these compounds. ***Keywords****: Benzazepine derivatives, ketone, [2-amino-3-(4-bromobenzoyl)phenyl]acetic acid, spectroscopic characterization, medicinal chemistry, heterocyclic compounds, structure-activity relationships, pharmaceutical development.ization and Model Tuning*
Newly synthesized derivatives (5a to 5i) have been evaluated on the basis of spectral and analytical data like IR, mass and ^{1}H-NMR spectroscopy.
Keywords: *Benzazepine,ketone,[2-amino-3-(4-bromobenzoyl)phenyl]acetic acid*

INTRODUCTION:

Heterocyclic compounds are biologically active;therefore they play an important role in the fordevelopment of newdrugswhich having newactivity. Some heterocyclic compounds have shown antimycobacterialactivity eg. derivatives of pyrrole,[1] indole,[2]furan,[3] and benzazepine.[4]Among of theseheterocycliccompounds, benzazepine derivatives are significant of biological activities. Benzazepine and its derivatives are reported to be physiologically and pharmacologicallyactive and find applications in the treatment of severaldiseases such as epilepsy, diabetes and antifertility.[5] It is an importantpharmacophore and privileged structure in medicinalchemistry[6]encompassing a diverse range of biological activitiesincludingproton pump inhibitors,[7] anti-psychotics,[8] antihelmentics,[9] antibacterial,[10] antifungal,[11] anti-inflammatory,analgesic,[12]anti-acne,[13]antihistamine,[14]antitubercular,[15]antiulcerative,[16] antiallergic,[17] cytotoxicity,[18] and anti-HIV-1.[19]Owing to the importance of benzazepine, we now wish to describeour efforts towards the synthesis of new **N- substituted 2-(4-((4, 4-dimethylthiochroman-6-yl)ethynyl)phenyl)-1H-benzo[d]imidazole derivatives** and their biological activity screeningstudies.

MATERIALS AND METHODS:

[2-amino-3-(4-bromobenzoyl) phenyl]acetic acid**1, Acetone** ,ethyl methyl ketone, methyl isobutyl ketone, methyl aceto acetate and ethyl aceto acetate. Thin layer Chromatography analysis was performed using precoated silica gel plates and visualized using iodine/UV lamps. Infrared spectra were recorded on a Jasco, FT/IR-4100 type-A spectrometer using a KBr diske. The proton-NMR spectra of the compounds were recorded using JEOL 500 MHz NMR spectrometer.

RESULTS AND DISCUSSION:

[2-amino-3-(4-bromobenzoyl)phenyl]acetic acid was synthesized by a known method using (4-bromophenyl)(1*H*-indol-7-yl)methanonetreated with n- chloro succinamide in the presence of ferric chloride in methylene dichloride at room temperature to form (4-bromophenyl)(3-chloro-2,3-dihydro-1*H*-indol-7-yl)methanone, which was treated withacetic acid and ortho phosphoric acid to form 7-(4-bromobenzoyl)-1,3-dihydro-2*H*-indol-2-one followed by hydrolysis with a sodium hydroxide solution .

The ring expantion of [2-amino-3-(4-bromobenzoyl)phenyl]acetic acidwith a simple ketone afforded different benzazepine derivatives.

General Procedure for the synthesis of (4-bromophenyl)(3-chloro-2,3-dihydro-1*H*-indol-7-yl)methanone

A mixture of 75.0gm.(0.60mole) of (4-bromophenyl)(1H-indol-7-yl)methanone, 33.89gm. (0.66mole) of n chlorosuccinimide,0.075 gm ferric chloride and 1125ml methylene dichloride in a 2 lit. flask stirred for 3 hours at room temperature, Reaction progress was monitored on TLC.After reaction completion, Reaction mass was quenched with sodium metabisulphate solution and stirred for 10 min.Distilled out methylene dichloride from reaction mass ,added purified water. The product was collected in a Buchner funnel and dried 6 hrs at 55°C - 60°C. The yield is 81 g.

General Procedure for the synthesis of 7-(4-bromobenzoyl)-1,3-dihydro-2*H*-indol-2-one

A mixture of 60 gm (4-bromophenyl)(3-chloro-2,3-dihydro-1H-indol-7-yl)methanone,600 ml ortho phosphoric acid (10 volume),1800 ml of acetic acid in 3.0 lit.flaskwas heated to 120-130°C for 12 - 15 hr. Reaction progress was monitored on TLC.After reaction completion, filter reaction mass at 40°C to remove insoluble.Reaction mass was quenched with purified water and stirred for 10 min.Added methylene dichloride in reaction mass and stirred for 30 min.The methylene dichloride and aqueous layers were separated and the .methylene dichloride layer was washed with 25 ml water and distilled under reduced pressure to obtained degassed mass, which was recrystallize intoluene to obtaine the corresponding (4-bromophenyl)(3-chloro-2,3-dihydro-1H-indol-7-yl)methanone.

General Procedure for the synthesis of [2-amino-3-(4-bromobenzoyl)phenyl]acetic acid

A mixture of 35 gm (4-bromophenyl)(3-chloro-2,3-dihydro-1H-indol-7-yl)methanone,215 ml toluene,105 ml ethanol,13.28 gm sodium hydroxide in a500 ml flask was heated to 70°C -80°C for 12 - 15 hr. The reaction progress was monitoredby TLC and after completion of the reaction, the reaction mass was filtered at room temperature .charged wet solid,100 ml of dimethoxy ether was stirred for 30 min .Dimethoxy ether and water were then separated .Theorganic layer was removed under reduced pressure to obtain a degassed mass, which was recrystallize inIPA to obtain the corresponding 2-amino-3-(4-bromobenzoyl)phenyl]acetic acid.

General Procedure synthesis of Benzazepine Derivative (4-bromophenyl)(4-hydroxy-2-methyl-5H-1-benzazepin-9-yl)methanone (4).

Acetone was addedto the solution of 2-amino-3-(4-bromobenzoyl)phenyl]acetic acid in water (20ml) ; the reaction mixture was refluxed for 6 hrs.Reaction progress was monitored on TLC.After reaction completion, reaction was stoped by heating and filteration of the reaction mixture at room temperature to obtain crude (4-bromophenyl)(4-hydroxy-2-methyl-5H-1-benzazepin-9-yl)methanone.which was purified in acetone water to obtain pure (4-bromophenyl)(4-hydroxy-2-methyl-5H-1-benzazepin-9-yl)methanone (4).

Synthesis of Benzazepine Derivative ((4-bromophenyl)(4-hydroxy-2,3-dimethyl-5H-1-benzazepin-9-yl)methanone

(5) and.(4-bromophenyl)(2-ethyl-4-hydroxy-5H-1-benzazepin-9-yl)methanone(6)

To the solution of 2 gm 2-amino-3-(4-bromobenzoyl)phenyl]acetic acid in(20) ethyl methyl ketone , (20ml) was added purified water The reaction mixture was stirred at reflux for 10-12 hrs Reaction progress was monitored on TLC.TLC shows 60:40 % of two isomer.After reaction completion, stopped stirring and separated ethyl methyl ketone and water.The ethyl methyl ketone layer was then washed with purified water.Ethyl methyl ketone was distilled under reduced pressure to obtain a degas masscrude ((4-bromophenyl)(4-hydroxy-2,3-dimethyl-5H-1-benzazepin-9-yl)methanone(2) and.(4-bromophenyl)(2-ethyl-4-hydroxy-5H-1-benzazepin-9-yl)methanone(5)

which was purified using a coloumn to obtain((4-bromophenyl)(4-hydroxy-2,3-dimethyl-5H-1-benzazepin-9-yl)methanone(2) and.(4-bromophenyl)(2-ethyl-4-hydroxy-5H-1-benzazepin-9-yl)methanone(6)

Synthesis of Benzazepine Derivative (4-bromophenyl)[4-hydroxy-3-methyl-2-(propan-2-yl)-5H-1-benzazepin-9-yl]methanone

(7) and (4-bromophenyl)[4-hydroxy-2-methyl-3-(propan-2-yl)-5H-1-benzazepin-9-yl]methanone (8)

To the solution of 2 gm 2-amino-3-(4-bromobenzoyl)phenyl]acetic acid in(20)methyl isobutyl keton, (20ml) was added purified water The reaction mixture was stirred at reflux for 36-48 hrsReaction progress was monitored on TLC TLC .TLC showed 70:30 % of the two isomers,after reaction completion, stoped heating and a cooled reaction mass of 50°C.Methyl isobutyl ketone was distilled under reduced pressure to obtain degas mass to obtained 70:30 % for two isomers of (4-bromophenyl)[4-hydroxy-3-methyl-2-(propan-2-yl)-5H-1-benzazepin-9-yl]methanone

(7) and (4-bromophenyl)[4-hydroxy-2-methyl-3-(propan-2-yl)-5H-1-benzazepin-9-yl]methanone (5).which was purified using a coloumn to obtain pure (4-bromophenyl)[4-hydroxy-3-methyl-2-(propan-2-yl)-5H-1-benzazepin-9-yl]methanone

(4) and (4-bromophenyl)[4-hydroxy-2-methyl-3-(propan-2-yl)-5H-1-benzazepin-9-yl]methanone (8).

Synthesis of Benzazepine Derivative ((4-bromophenyl)(4-hydroxy-2,3-dimethyl-5H-1-benzazepin-9-yl)methanone

(9).

To the solution of 2 gm 2-amino-3-(4-bromobenzoyl)phenyl]acetic acid in(20) methyl aceto acetate , (20ml) was added purified water The reaction mixture was stirred at reflux for 16-18 hrs Reaction progress was monitored on TLC.

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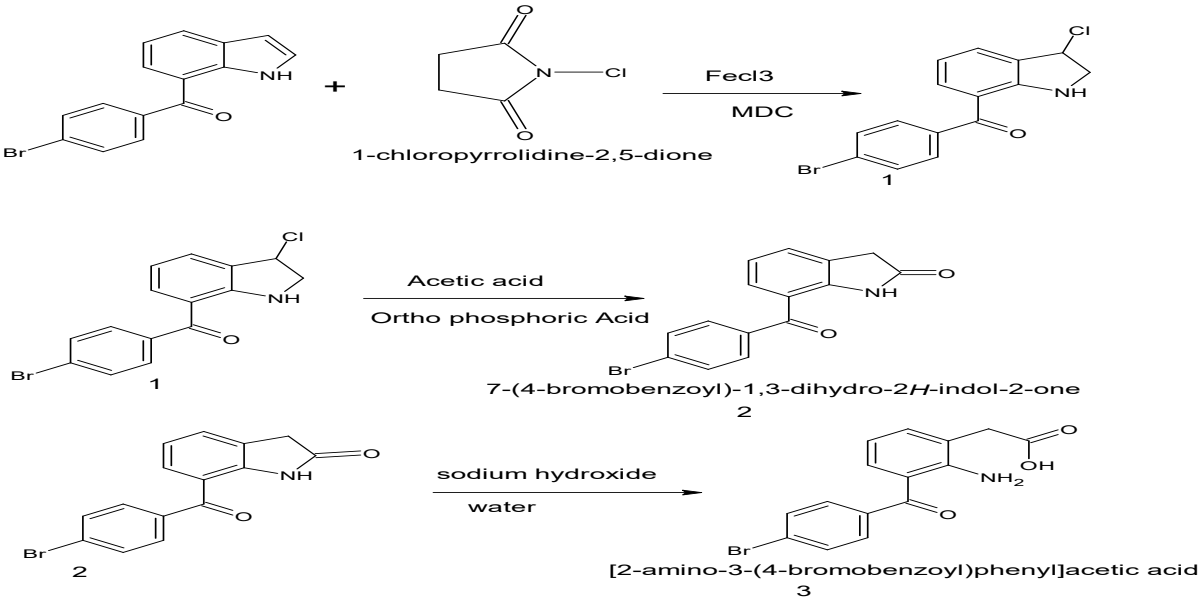
After the completion of reaction , the stopedheating of reaction and cooled reaction mass 50°C.Distilled methyl aceto acetae under reduced pressure to get degas mass to obtaine ((4-bromophenyl)(4-hydroxy-2,3-dimethyl-5H-1-benzazepin-9-yl)methanone

Synthesis of Benzazepine Derivative ethyl 9-(4-bromobenzoyl)-2-methyl-4-oxo-4,5-dihydro-3H-1-benzazepine-3-carboxylate (10).

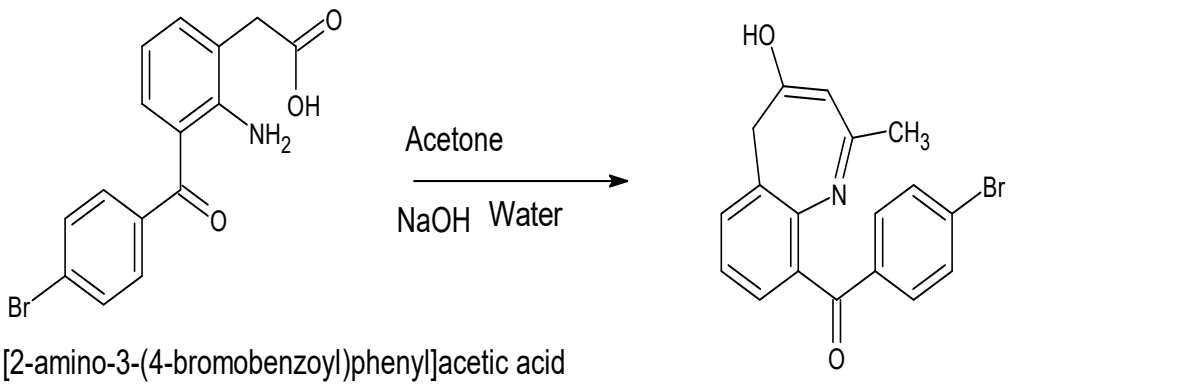
To the solution of 2 gm 2-amino-3-(4-bromobenzoyl)phenyl]acetic acid in(20) ethyl aceto acetate , (20ml) was added purified water The reaction mixture was stirred at reflux for 20-25 hrs Reaction progress was monitored on TLC.

After completion of reaction, the heating stoped and cooled reaction mass 50°C.Distilled outmethyl aceto acetae under reduced pressure to obtainedethyl 9-(4-bromobenzoyl)-2-methyl-4-oxo-4,5-dihydro-3H-1-benzazepine-3-carboxylate (10).

Scheme 1



Scheme 2



[2-amino-3-(4-bromobenzoyl)phenyl]acetic acid

(4-bromophenyl)(4-hydroxy-2-methyl-5*H*-1-benzazepin-9-yl)methanone

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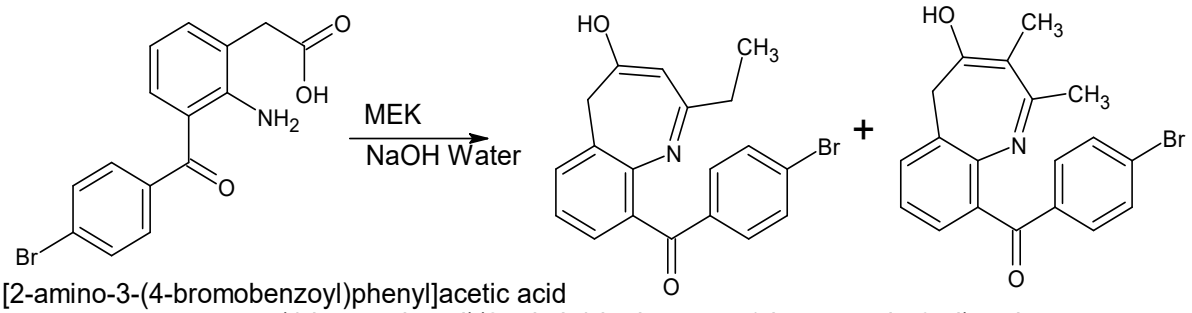
Scheme 3

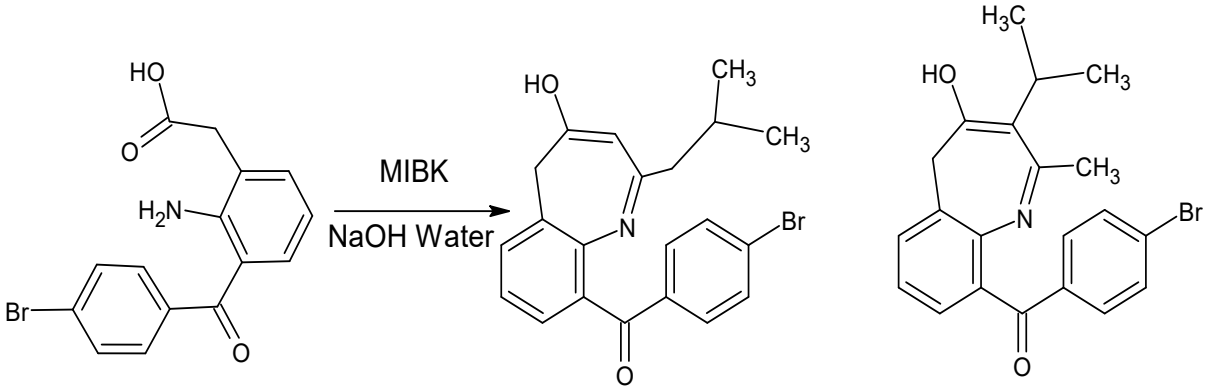


[2-amino-3-(4-bromobenzoyl)phenyl]acetic acid

(4-bromophenyl)(2-ethyl-4-hydroxy-5*H*-1-benzazepin-9-yl)methanone

(4-bromophenyl)(4-hydroxy-2,3-dimethyl-5*H*-1-benzazepin-9-yl)methanone

Scheme 4

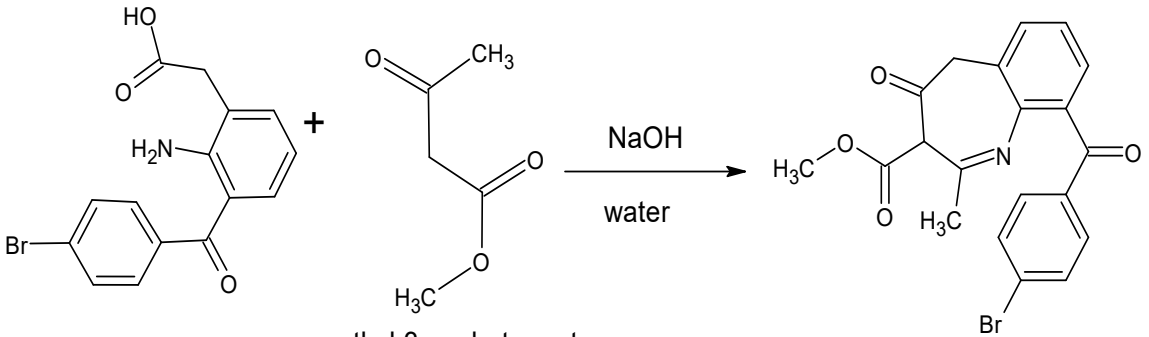


(4-bromophenyl)[4-hydroxy-3-methyl-2-(propan-2-yl)-5*H*-1-benzazepin-9-yl]methanone

(4-bromophenyl)[4-hydroxy-2-methyl-3-(propan-2-yl)-5*H*-1-benzazepin-9-yl]methanone

7 8

Scheme 5



methyl 3-oxobutanoate

methyl 9-(4-bromobenzoyl)-2-methyl-4-oxo-4,5-dihydro-3*H*-1-benzazepine-3-carboxylate

9

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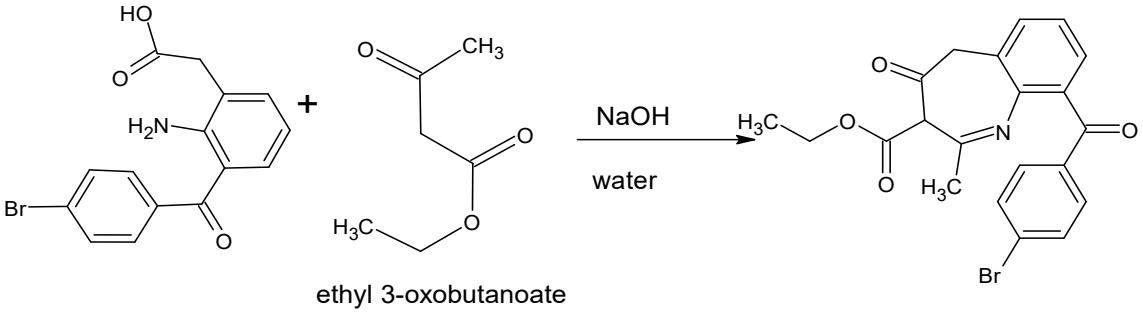
Figure 5



ethyl 3-oxobutanoate

ethyl 9-(4-bromobenzoyl)-2-methyl-4-oxo-4,5-dihydro-3*H*-1-benzazepine-3-carboxylate

10

RESULTS AND DISCUSSION:

Analytical Characterization:

(4-bromophenyl)(4-hydroxy-2-methyl-5H-1-benzazepin-9-yl)methanone (4).

Yield 85%.

IR(KBr):2960.28(Ar-H) 2852.13(C-H),2200 (C≡C), 1755.27(C=0),1451.52(Ar-C=C), 1092.90(C=N), 749.30(=C-H), 646.36(S-C)cm^{1};

^{1}HNMR(CDCl3):δ2.72(S,3H), 4.22(S,2H), 7.24(s, J1=7.6*Hz*,1H), 7.42-7.40-7.40-7.38-7.38-7.36(mJ1=7.9*Hz*,J2=1.7*Hz*,3H),7.70-7.68-7.65-7.64-7.62(m,4H),

Mass (m/z): 355.7357.65 and (M^{+}+1).

((4-bromophenyl)(4-hydroxy-2,3-dimethyl-5H-1-benzazepin-9-yl)methanone (5)

Yield 65%.

IR(KBr):2960.28(Ar-H) 2852.13(C-H), 1755.27(C=0), 1451.52(Ar-C=C), 749.30(=C-H), ^{1}HNMR(CDCl3): δ1.373-1.354-1.335-1.298(t,*J1=6.2Hz*,3H), 1.941-2.030(s,1H), 2.993-2.973-2.956-2.937(m,2H), 4.004-3.972(d,2H7.266-7.248-7.227(t, J1=7.6*Hz*,2H),7.529-7.509-7.486-7.477-7.460-7.439-7.432(m,J1=7.9*Hz*,J2=1.7*Hz*,3H),7.671-7.651(t,2H),7.736-7.719(d,*J1=2.1Hz,1*H)

Mass (m/z): 369-371 (M^{+}+1).

(4-bromophenyl)(2-ethyl-4-hydroxy-5H-1-benzazepin-9-yl)methanone(6)

Yield 45%.

IR(KBr):2960.28(Ar-H) 2852.13(C-H), 1755.27(C=0), 1451.52(Ar-C=C), 749.30(=C-H),

^{1}HNMR(CDCl3): δ1.373-1.354-1.335-1.298(t,*J1=6.2Hz*,3H), 1.941-2.030(s,1H), 2.993-2.973-2.956-2.937(m,2H), 4.004-3.972(d,2H7.266-7.248-7.227(t, J1=7.6*Hz*,2H),7.529-7.509-7.486-7.477-7.460-7.439-7.432(m,J1=7.9*Hz*,J2=1.7*Hz*,3H),7.671-7.651(t,2H),7.736-7.719(d,*J1=2.1Hz,1*H)

Mass (m/z): 369-371 (M^{+}+1).

(4-bromophenyl)[4-hydroxy-3-methyl-2-(propan-2-yl)-5H-1-benzazepin-9-yl]methanone (7)

Yield 45%.

IR(KBr):2960.28(Ar-H) 2852.13(C-H), 1755.27(C=0), 1451.52(Ar-C=C), 749.30(=C-H), ^{1}HNMR(CDCl3): δ1.285-1.262-1.256-1.183-1.163-1.153(d,*J1=6.2Hz*,6H),1.140(s,3H), 2.909-2.892-2.839(d,2H),4.263-4.229(s,2H), 7.07-7.08(s, J1=7.6*Hz*,2H), 7.20-7.26(dd,J1=7.9*Hz*,J2=1.7*Hz*,2H),7.38-7.43

(m,1H),7.55(d,*J1=2.1Hz,3*H),7.60-7.62
(d,*J1=2.8Hz,*2H),
Mass (m/z): 397.95-400 (M^++1).

(4-bromophenyl)[4-hydroxy-2-methyl-3-(propan-2-yl)-5H-1-benzazepin-9-yl]methanone (8)

IR(KBr):2960.28(Ar-H) 2852.13(C-H), 1755.27(C=0), 1451.52(Ar-C=C), 749.30(=C-H), ^{1}HNMR(CDCl3): δ1.26-128(d,*J1=6.2Hz,*6H), 1.36(s,6H), 1.95-1.98(m,2H), 3.048-3.073(m,2H), 5.14-5.19(m,1H), 7.07-7.08(d, J1=7.6*Hz*,1H), 7.20-7.26(dd,J1=7.9*Hz*,J2=1.7*Hz*,1H),7.38-7.43 (m,2H),7.55(d,*J1=2.1Hz,1*H),7.60-7.62
(q,*J1=2.8Hz,*2H), 7.64-7.65(q,J1=2.8*Hz*,2H), 7.78-7.82(t,d, J1=6.9*Hz*,J2=4.4*Hz*,1H), 8.04-8.08(m, 1H).
Mass (m/z): 397.95-400 (M^++1).

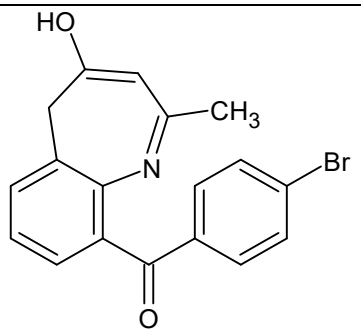
((4-bromophenyl)(4-hydroxy-2,3-dimethyl-5H-1-benzazepin-9-yl)methanone (9).

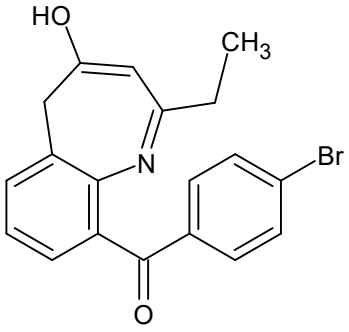
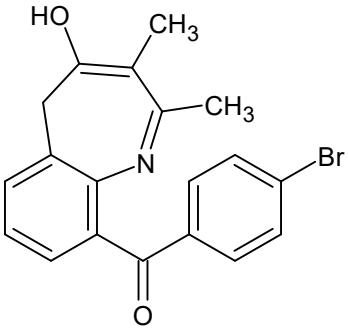
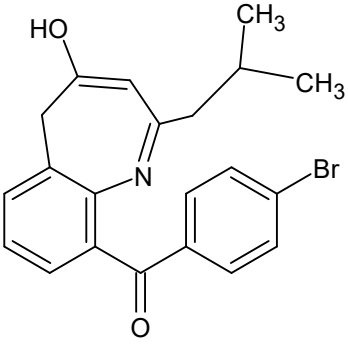
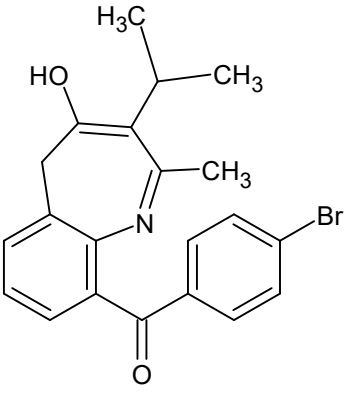
IR(KBr):2960.28(Ar-H) 2852.13(C-H), 2200 (C≡C), 1755.27(C=0), 1451.52(Ar-C=C), 749.30(=C-H), ^{1}HNMR(CDCl3): δ2.409-2.463(S,3H), δ3.372-3.312(S,3H),3.931(s,2H), 6.616(d,1H), 6.810-6.791(d,2H), 7.266-7.248-7.227(t, J1=7.6*Hz*,2H),7.529-7.509-7.486-7.477-7.460-7.439-7.432(m,J1=7.9*Hz*,J2=1.7*Hz*,3H),7.671-7.651(t,2H),7.736-7.719(d,*J1=2.1Hz,1*H),Mass (m/z): 413.90 and 415.90(M^++1).

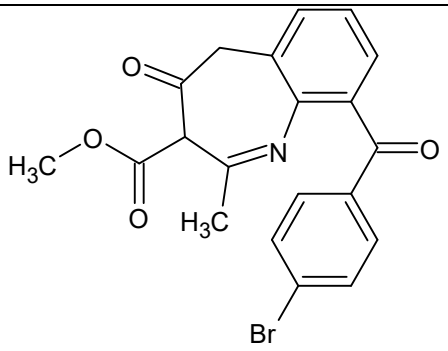
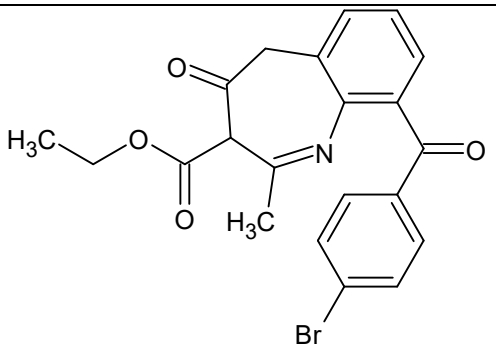
ethyl 9-(4-bromobenzoyl)-2-methyl-4-oxo-4,5-dihydro-3H-1-benzazepine-3-carboxylate (10).

IR(KBr):2960.28(Ar-H) 2852.13(C-H), 1755.27(C=0), 1451.52(Ar-C=C), 749.30(=C-H), ^{1}HNMR(CDCl3): δ1.058-1.040-1.022(t,*J1=6.2Hz,*3H), 2.854(s,3H), 4.157-4.139-4.121-4.103(m,2H), 4.257(s,2H), 7.266-7.248-7.227(t, J1=7.6*Hz*,2H),7.529-7.509-7.486-7.477-7.460-7.439-7.432(m,J1=7.9*Hz*,J2=1.7*Hz*,3H),7.671-7.651(t,2H),7.736-7.719(d,*J1=2.1Hz,1*H),
Mass (m/z): 427.8-429.9.5 (M^++1).

aTable 1: Benzazepine new derivative using different ketones.

Sr.no.	Solvent	Name of Benzazepine	Structure of Benzazepine
1	Acetone	(4-bromophenyl)(4-hydroxy-2-methyl-5*H*-1-benzazepin-9-yl)methanone	
2	Methyl ethyl ketone		

2a		(4-bromophenyl)(2-ethyl-4-hydroxy-5*H*-1-benzazepin-9-yl)methanone	
2b		(4-bromophenyl)(4-hydroxy-2,3-dimethyl-5*H*-1-benzazepin-9-yl)methanone	
3	Methyl isobutyl ketone		
3a		(4-bromophenyl)[4-hydroxy-3-methyl-2-(propan-2-yl)-5*H*-1-benzazepin-9-yl]methanone	
Sr.no.	**Solvent**	**Name of Benzazepine**	**Structure of Benzazepine**
3b		(4-bromophenyl)[4-hydroxy-2-methyl-3-(propan-2-yl)-5*H*-1-benzazepin-9-yl]methanone	

4	Methyl Aceto Acetate	methyl 9-(4-bromobenzoyl)-2-methyl-4-oxo-4,5-dihydro-3*H*-1-benzazepine-3-carboxylate	
5	Ethyl Aceto Acetate	ethyl 9-(4-bromobenzoyl)-2-methyl-4-oxo-4,5-dihydro-3*H*-1-benzazepine-3-carboxylate	

CONCLUSION: The structures of all synthesized compounds were well supported by spectroscopy analysis data..

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