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BIOACTIVE SYNTHESIS OF N-ALKYL DERIVATIVES OF ACRIDONES ANALOGOUS

C. Satya, B. Priyanka and Dr. N. Krishna Rao*

PRISM PG & DG College, (Affiliated to Andhra university), Visakhapatnam, India.

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***Corresponding Author**
Dr. N. Krishna Rao
PRISM PG & DG College, (Affiliated to Andhra university), Visakhapatnam, India.

ABSTRACT

Five series of N-alkyl substituted acridones such as10-benzyl-9-(3,4-dimethoxyphenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione derivatives have been designed and synthesized using an efficient methodology from suitable acridones intermediates such as. 9-(3,4-dimethoxyphenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione which is obtained from3,4-dimethoxy benzaldehyde, dimedone and Ammonium chloride. In addition to the study of antimicrobial activity.

KEYWORDS: Dimedone, aromatic aldehyde, 9-(3,4-dimethoxyphenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1, 8(2H,5H)-dione,10-benzyl-9-(3,4-dimethoxyphenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione,Antimicrobial activity.

1. INTRODUCTION

Acridine and its derivatives are important structural motifs possessing antimalarial, antiviral, and antiallergic properties.[1–3] acridine act as potent drugs for antitumor activity both in vitro and in vivo against a range of murine and human tumors.[4] They are also found to act as fluorescent molecular probes for monitoring polymerization processes.[5] and are used as -type semiconductors and in the electroluminescent devices. Recently fluorinated acridones are reported to possess anticancer activity.[6–9] There are a few reports in the literature on the three-component Hantzsch-type condensation of aromatic aldehydes, anilines, and dimedone via traditional heating in organic solvents,[10,11] under microwave irradiation,[12] and in ionic liquids.[13] The main drawbacks of these methods are the inability to synthesize profuse quantity of acridine using substituted anilines containing electron withdrawing groups.[14] Further, the reactions are carried out in refluxing organic solvents, which require higher temperature and longer hours for completion.[10,15] and unusual breaking of C–N bond takes place under certain reaction conditions as noticed in a few cases.[16] Hence, the exploration of a simple, efficient, and green method for the synthesis of acridones using electron-deficient amines and electron-deficient aldehydes is of current interest. In continuation with our work on one-pot multicomponent reactions under sonic condition,[17–19] we, herein, report the synthesis of a series of acridness by a one-pot four-component reaction as shown in Schemes 1. To the best of our knowledge, the synthesis of acridones from fluorinated aromatic amines and heterocyclic amines using an inexpensive catalyst under sonic condition is not reported yet.

2. METHODS AND MATERIAL

2.1. Experimental section

All the synthetic grade reagents and chemicals were procured from Fine Chemicals PVL and they supplied the purity of the solvent without furtherpurification. The melting points of newly synthesized compounds were determined by Agrwal thermometer in open capillaries and are uncorrected. 1H NMR and 13CNMR spectra were recorded on a Brucker 400MHz spectrometer using CDCl3 as solutions. Chemical shifts are given in ppm relative to TMS, multiplicative are reported as singlet(s),doublet (d), triplet(t), quartet (q), some consideration of these, multiplicative (m).Molecular weight of the newly synthesized compounds were recorded by LCMS spectrometer.Reaction mixture was checked monitored on Fine Chemicals PVL .Visualization was accomplished either an UV chamber (or) in Iodine Vapour. Coloumnchromotogrphy was carried out on silica gel (100mesh) Merck chemicals.

2.2.1. General Procedure 9-(3,4-dimethoxyphenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahy droacridine-1,8 (2H,5H) -dione

A mixture of 3,4- dimethoxybenzaldehydes (1mmole), dimedone (2mmole) and Ammonium chloride (1.5mmole) and freshly prepared catalytic amount of I_2 in ethanol was taken in four neck 50mL RB flask. When the solution becomes clear, was added and the reaction mixture was refluxed for 3-4 hours. The reaction was checked by TLC (5:5, ethyl acetate: n-hexane) after the completion of reaction. The reaction mixture was poured into crushed ice. The solution was neutralized with aqueous solution of sodium bi carbonate and product was extracted using ethyl acetate, the combined organic layer

was washed using water and organic layer seperated. The organic layer was dried on anhydrous sodium sulphate and the solvent was removed under reduced pressure with using vacuum pump to get the solid product. All the titled compounds were recrystallized from ethanol.

Characterizationof9-(3,4-dimethoxyphenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahy droacridine-1,8(2H,5H)-dione:

Palebrown,yields-91%.1HNMR(400Mz,$CDCl_3$)ppm:9.176(s,1H,NH),7.102-6.876(m,3H,Ar-H), 4.234(s,1H,-CH),3.774 (s,1H,-CH-), 3.610(s,3H,-OCH3),2.142(s,2H,-CH2),1.724(s,2H,-CH2), 1.126(s,3H,CH3),0.942(s,3H,-CH_3);^{13}CNMR (100 MHz, $CDCl_3$) ppm: 195.72, 145.56, 141.36, 133.72, 126.44, 123.96, 118.82, 115.35, 110.62, 55.27, 51.09, 41.32, 84. 28.09, 27.44 .LCMS (m/z):410.36(M+H). Molecularformule: $C_{25}H_{31}NO_4$, Elemental Analysis: Calculated C-73.32.,H-7.63,N-3.42. Obtained: C-73.25, H-7.61, N-3.49.

2.2.2. Geneneralprocedurerof10-benzyl-9-(3,4-dimethoxyphenyl)-3,3,6,6-tetramethyl-3,4,6, 7,9,10-hexahydroacridine -1,8(2H,5H)-dione derivatives

A mixture 9-(3,4-dimethoxyphenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dioneand substituted (bromomethyl)benzene is dissolved in triethylamine and methylene dichloride which is taken in four neck 50mL RB flask. When the solution becomes clear, was added and the reaction mixture was refluxed for 3-4 hours. The reaction was monitored by TLC (4:6, ethyl acetate: n-hexane) after the completion of reaction. The reaction mixture was poured into crushed ice. The solution was neutralized with aqueous solution of sodium bi carbonate and product was extracted using ethyl acetate, the combined organic layer was washed using water and organic layer separated. The organic layer was dried on anhydrous sodium sulphate and the solvent was removed under reduced pressure with using vacuum pump to get the solid product. All the titled compounds were recrystallized from ethanol.

2.2.2.1.10-benzyl-9-(3,4-dimethoxyphenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione (5a)

Paleredsolid,yields-85%.1HNMR(400Mz,$CDCl_3$)ppm:7.346-7.278 (m,5H, Ar-H),7.084-6.945 (m,3H,Ar-H),4.245(s,1H,-CH),3.964 (s,2H,-CH2),3.584(s,6H,(OCH3)2),2.342-1.845(m,2H,-CH2-),1.624-1.074(m,2H,-CH-),0.942 (s,3H,CH3), 0.879 (s,3H,-CH3).13CNMR(100MHz, CDCl3) ppm:194.23, 155.56, 142.54, 138.72, 134.64, 130.25, 128.94, 128.35, 128.22, 127.44, 123.96, 118.82, 113.35, 110.62, 55.27, 53.45, 50.32, 40.15, 30.75, 28.12, 27.65 .LCMS(m/z): 500.14(M+H). Molecular formulae: C32H37NO4, Elemental Analysis: Calculated C-76.92.,H-7.46,N-2.80. Obtained: C-76.85, H-7.44, N-2.89.

2.2.2.2.9-(3,4-dimethoxyphenyl)-3,3,6,6-tetramethyl-10-(4-methoxylbenzyl)-3,4,6,7,9,10-hexahydroacridine-1,8 (2H,5H) -dione (5b):

Paleyellowsolid,yields-85%.^{1}HNMR(400Mz,$CDCl_3$)ppm:7.252-7.089(m,4H,Ar-H),6.989-6.745 (m,3H,Ar-H),4.246 (s,1H,-CH), 4.025 (s,2H,-CH_2),3.794 (s,6H,OCH3), 3.675(s,6H, (OCH3)2) ;2.312-2.056(m,2H,-CH2-),1.970-1.246(m, 2H,-CH-), 0.967(s,3H,CH3),0.845(s,3H,-CH3). 13CN MR (100 MHz, CDCl3)ppm:192.46,155.32,150.75, 148.09, 140.25, 134.75,129.66,128.77, 127.45,119.08, 115.51, 113.66,55.65,54.72,52.62,50.07,48.28,31.36,28.16, 27.58.LCMS (m/z):530.33(M+H).Molecular formulae: C33H39NO5, Elemental Analysis: Calculated C-74.83., H-7.42, N-2.64. Obtained: C-74.75, H-7.41, N-2.71.

2.2.2.3.9-(3,4-dimethoxyphenyl)-3,3,6,6-tetramethyl-10-(4-methylbenzyl)-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione (5c)

Colourless,yields-90%.1HNMR(400Mz,CDCl3)ppm:7.256-7.112 (m, 4H,Ar-H),6.982-6.713(m, 3H,Ar-H),4.625(s,1H,-CH-), 4.226(s,2H,-CH-), 3.714(s,3H,OCH3), 3.594 (s,3H, OCH3), 2.410-2.156(m,2H,-CH-),1.845-1.234(m, 2H,-CH-), 1.845-1.234(s,3H,-CH3),1.234(s,3H,-CH2), 0.942 (s,3H,CH3),0813(s,3H,CH3); 13CNMR (100MHz, CDCl3) ppm:191.24, 154.56, 147.60, 140.75, 134.32, 130.65, 129.75, 128.78, 128.45, 126.72, 115.63, 113.55, 110.92, 55.72, 50.65, 40.58, 33.35,30.45,28.06, 27.67, 22.55.LCMS(m/z): 514.44(M+H). Molecular formulae: C33H39NO4, .Elemental Analysis: Calculated C-77.16,H-7.65,N-2.73; Obtained: C- 77.04, H-7.64, N-2.81.

2.2.2.4.10-(4-chlorobenzyl)-9-(3,4-dimethoxyphenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,1 0-hexahydroacridine-1,8(2H,5H)-dione (5d)

Colourlessslid,yields-87%.1HNMR(400Mz,CDCl3)ppm:7.412-7.112 (m,4H, Ar-H),6.984-6.714(m,3H,Ar-H),4.567 (s,1H,-CH-),4.126 (s,2H,-CH-),3.762(s,3H,OCH3),3.615(s,3H, OCH3), 2.036 (m,2H,CH2-),1.426(m,2H,-CH2),0.962 (s,3H, CH3), 0.864(s,3H,CH3);13CNMR (100MHz, CDCl3) ppm: 192..46,155.37,147.62,141.07, 135.12,131. 73, 129.68, 128.91,128.36, 126.78,116.76,113.38, 111.65, 58.34,51.46,41.66,32.03,30.78,28.76, 27.97.. LCMS (m/z): 534.09(M+H). Molecular formulae: C32H36 ClNO4, .Elemental Analysis: Calculated C-71.96,H-6.79,N-2.62; Obtained: C- 71.87, H-6.77, N-2.91.

2.2.2.5.10-(4-bromobenzyl)-9-(3,4-dimethoxyphenyl)-3,3, 6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8 (2H,5H)-dione (5e)

Colourlessslid,yields-87%.1HNMR(400Mz,CDCl3)ppm:7.578-7.298 (m,4H, Ar-H), 7.214-6.8914(m,3H,Ar-H),4.614 (s,1H,-CH-), 4.138 (s,2H,-CH-), 3.714(s,3H,OCH3), 3.605(s,3H, OCH3), 2.098(m,2H,CH_2-),1.408(m,2H,-CH_2),0.987

(s,3H,CH_3), 0.814(s,3H,CH_3);^{13}CNMR (100MHz, $CDCl_3$)ppm:193..48,156.04,148.33,141.45,136.98,130.54, 129.52, 128.96, 128.87, 126.87,116.24, 113.32, 112.85,58.12,50.84,40.47,32.12,30.32,28.08,27.96.LCMS(m/z): 579.11(M+H). Molecular formulae: C32H36BrNO4, Elemental Analysis: Calculated C-71.74,H-6.56,N-2.87; Obtained: C- 71.68, H-6.55, N-2.94.

3. Pharmacological activity

Preliminary investigation of the newly obtained and well characterized derivatives (4a-4l) were examined for in vitro antibacterial activity against Gram (+Ve) bacteria as well as, Gram (-Ve) bacterial strains such as P.aeruginose, E.coli, B-substills and Staphylocoecus aureus. The Ciprafloxin is used as reference drug for antibacterial growth On the other hand antifungal activity of the desired compounds against AspergillusNiger, Candida albicans and Aspergillus favus using agar well diffusion assay. The ketoconazole is used as reference drug for antifungal growth and zones of inhibition of the evaluated derivatives were expressed in mm as shown in Table-IV and Table-V.

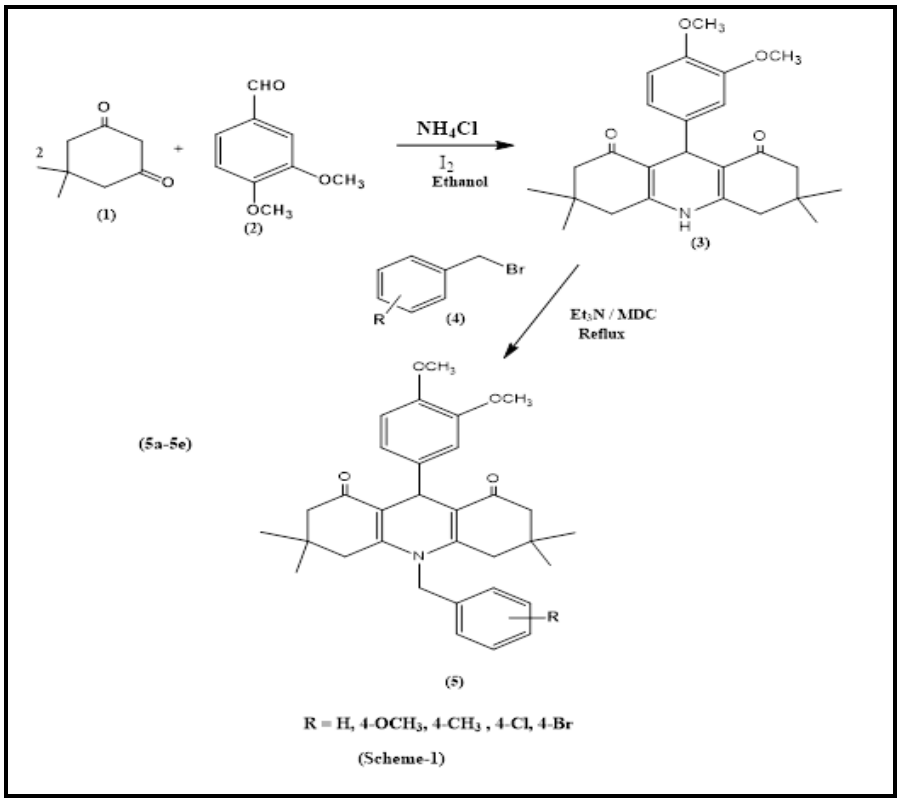
3.1. Anti-bacterial Activity

The cup plate method is used to evaluate for invitro anti-bacterial activity of titled derivatives (5a-5e).We used various pathogenic strains such as E.coli P.aeruginose, and E.coli (gram-ve) and Staphylocoecus aureus, B-substills(gram+ve) using standard drug streptomycin for antibacterial growth. The anti-bacterial activity of tested derivatives used to evaluate by Nutrient agar medium (NAM); NAM was contained a mixture of peptone (7g), NaCl (5g), beef extract (4g), agar (20g) in 100 ml of distilled water and a PH value of the mixture was maintained to neutral. NMA was sterilized in an auto clave at 37℃ 15 lbs pressure for 45 min. After sterilization, 20 ml of NAM was poured into petri dishas in a laminar air flow and allowed to solidity. After completion of the solidification, NMA was in oculated with 100 µl of the tested bacteria pathogens. The tested derivatives were dissolved in DMSO with a concentration and whatsman No.1 filter paper disks were placed in the solution and kept for one minute. After completion of the drying the disks were placed as NAM inoculated with bacteria and NAM plates were incubated at 37 ℃. After 24hrs.The zones of inhibition tested analogous were compared with Ciprafloxin as standard drug moiety. The compound ‘4a’ exhibited ‘low active potential against all bacterial strains because aromatic aldehyde possesses electron withdrawing groups on the other hand the derivatives such as ‘4b&4c’ showed moderate to good active potential having electron releasing groups. From the above table-II represented that the compound 4d exhibited excellent active potential.

4. RESULTS AND DISCUSSION

4.1. Chemistry

The reaction between 9-(3,4-dimethoxyphenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione and substituted (bromomethyl)benzene was carried out by conventional methods during 30 min giving derivatives of 10-benzyl-9-(3,4-dimethoxyphenyl) -3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione 5a–5e in 85–89% yield (Scheme 1).



(Scheme-1)

The structures of compounds 5a–5e were assigned by 1Hand 13C NMR spectra and mass spectrometric data which are consistent with the proposed titled derivatives. For example, the 1H NMRand of compound 3a shows two singlets at 13.80,7.65 ppm, with integrals in the ratio 1:1, , and one multiplet integrating for 8 protons at 7.428–7.112 ppm, corresponding to aromatic protons of phenyl ring. Regarding 13C NMR spectra, DEPT experiments allowed us the assignment of the signals belonging to quaternary, tertiary, secondary, and primary carbon atoms of compounds 5a–5e.

4.2.2. Antimicrobial activity

The in vitro antibacterial activity of the derivatives of 10-benzyl-9-(3,4-dimethoxyphenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione (5a-5e) was compared with standard" Ciprafloxin'' as collected in (Table-I. As observed in Table-I the most of the synthesized analogueouswas generally evaluated active potential against all the tested bacterial strains. The tested derivatives "4d, 4e" exhibited excellent antibacterial activity against gram-(+Ve) bacterial strains viz; E.coli, P.aeruginose and gram(+Ve) bacterial strains viz; B.subtilis, and Staphylococcus aureus respectively due to such The in vitro antibacterial activity of the titled derivatives (5a5e) was compared with standard" Ciprafloxin'' as collected in (Table-I). As observed in Table-I, the most of the synthesized analgueous was generally evaluated potent activity against all the tested bacterial strains. The tested derivatives "4d and 4e" exhibited excellent antibacterial activity against gram-(+Ve) bacterial strains viz; E.coli, P.aeruginose and gram (+Ve) bacterial strains viz; B.subtilis, and Staphylococcus aureus respectively due to such compounds containing halogen atoms. The derivatives 4b, 4c, examined moderate to good active potential against bacterial strains. The compounds"4aj" showed low activity against bacterial strains.. These results represented that the derivatives having electron donating groups screened moderate to good activity than the compounds having electron withdrawing groups. The compounds containing halogen atoms exhibited excellent active potential against anti-bacterial activity.

Table I: The invitro antibacterial, activities of derivatives of 10-benzyl-9-(3,4-dimethoxyphenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione.

Compound	Anti-Bacterial Activity			
	Gram(+ve) bacteria		Gram(-ve) bacteria	
	Escherichia coli	Pseudomonas aureoginosa	Bacillus subtilis	Staphylococcus aureus
5a	10	09	08	10
5b	13	15	18	16
5c	19	18	19	20
5d	222	22	23	24
5e	23	22	25	24
Ciprafloxin	**27**	**27**	**30**	**30**
DMSO				

Table II: The invitro antifungal activities of derivatives of 10-benzyl-9-(3,4-dimethoxyphenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione.

Entry	Anti-Fungal Activity		
	Aspergillus Niger	Candida albicans	Aspergillusfavus
5a	08	07	09
5b	21	20	19
5c	18	17	18
5d	19	20	18
5e	21	20	19
Ketonozole	25	25	25
DMSO			

5. CONCLUSIONS

To conclude, we have developed a general, practical, and high yielding procedure to construct different N-H- and derivatives of 10-benzyl-9-(3,4-dimethoxyphenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8 (2H,5H)-dione from electron-deficient as well as electron-rich 9-(3,4-dimethoxyphenyl)-3,3,6,6-tetramethyl -3,4,6,7,9,10-hexahydroacridine-1,8 (2H,5H)-dione and substituted (bromomethyl)benzene which is obtained from dimedone ,3,4-dimethoxy benzaldehyde with ammonium chloride. High yields, shorter reaction durations, and mild reaction conditions are the added advantages of our energy efficient method.

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