

Asian Journal of Research in Chemistry and Pharmaceutical Sciences

Journal home page: www.ajrcps.com

<https://doi.org/10.36673/AJRCPS.2024.v12.i01.A01>



AN EFFICIENT ONE POT SYNTHESIS OF ACRIDINEDIONES EMPLOYED BY AMMONIUM MOLIBDATE AND STUDY OF ANTIMICROBIAL ACTIVITY

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ABSTRACT

A simple approach to the synthesis of acridinediones via one-pot three-component condensation of an aromatic aldehyde, 5, 5-dimethyl-1, 3-cyclohexanedione (dimedone) and p-Toluidine in isopropanol promoted by the use of as an efficient Ammonium molibdate catalyst is described. Excellent yields, catalyst recovery and reusability and easy work-up are attractive features of this green protocol. All the synthesized acridinediones were characterized on the basis of their melting-points, elemental analysis and spectral data such as IR, ¹HNMR, ¹³CNMR and LCMS and the evaluation of antimicrobial activity.

KEYWORDS

Acridinediones, Aromatic aldehyde, Ammonium molibdate, p-Toluidine and Antimicrobial activity.

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INTRODUCTON

Multicomponent reactions (MCRs) are a promising, vital field of chemistry because the synthesis of complicated molecules can be achieved rapidly and efficiently without the isolation of intermediates¹. In MCR condensations, three or more compounds reacting a single event, but consecutively, to form a new product, which contains the essential parts of all the starting materials? MCRs meet the requirements of an environmentally friendly process, with fewer synthetic steps and less energy consumption and waste production. Moreover, MCRs offer the advantage of simplicity and synthetic efficiency over conventional chemical reactions. Therefore, the search for new MCRs and full exploitation of known MCRs is of considerable interest.

Acridinediones dyes are a new class of laser dyes with lasing efficiency comparable to that of coumarin-102^{2,3}. These dyes have been shown to mimic NADH analogues to a greater extent because of their tricyclic structures, which protect the enamine moieties⁴. The design and synthesis of 1, 3-dithiol-linked acridinediones functionalized gold nanoparticles was described recently⁵, as was the design and synthesis of an acridinediones functionalized gold nanoparticle-based PET anion sensor⁶. 1, 8-(2H, 5H)-Acridinediones were synthesized with the Hantzsch procedure, which involves thermal reaction of 5, 5-dimethyl-1, 3-cyclohexanedione (dimedone) with an aldehyde and ammonia. Various methods have been used to synthesize acridinediones, including the microwave^{7,8}, ionic liquid^{9,10}, LiBr¹¹, proline¹², silica-bonded S-sulfonic acid¹³, ceric Ammonium nitrate¹⁴ and Methanesulphonic acid²⁸ catalysts. Acridinediones are also synthesized in aqueous media¹⁵⁻¹⁸; however, many of the methods described have drawbacks, such as use of hazardous organic solvents, long reaction times, low yields, formation of side products and multistep synthesis. Subsequently, there is a demand and scope for developing an efficient, easy, eco-safe approach to obtain acridinediones.

We found that efficient Ammonium molybdate as catalyzed the synthesis of 9-Phenyl-3, 3, 6, 6-tetramethyl-10-p-tolyl-hexahydro acridine-1, 8-dione analogous in the reaction of substituted aromatic aldehydes, dimedone and p-Toluidine in isopropanol at 70°C. The aim of the study reported here was to synthesize acridinediones with Ammonium molybdate as the catalyst.

MATERIAL AND METHODS

EXPERIMENTAL

Chemicals were purchased from Merck, Fluka and Aldrich Chemical companies. All yields refer to isolated products unless otherwise stated. ¹H Nuclear magnetic resonance (NMR) (400 MHz) and ¹³C NMR (100 MHz) spectra were obtained on a Bruker DRX-400 Avanceat ambient temperature, with tetramethylsilanes as the internal standard and Duetrochloroform (CDCl₃) as the solvent. Fourier

transforms infrared (IR) spectra were obtained as KBr discs on a Shimadzu spectrometer. Mass spectra were determined on a Varion Saturn 2000 gas chromatograph–mass spectrometer. Elemental analyses were conducted with a Perkin Elmer 2400 CHN elemental analyzer flowchart.

General procedure for synthesis of 9-Phenyl-3, 3, 6, 6-tetramethyl-10-p-tolyl-hexahydro acridine-1, 8-dione (4a-4g)

A mixture of substituted aldehyde 1(1mmol), dimedone 2(2mmol), p-Toluidine 3(1.5mmol), Ammonium molybdate (1mmol%) and isopropanol (25mL) was placed in a 50mL flask, heated at 70°C and stirred for the appropriate time as monitored by thin-layer chromatography (hexane: ethyl acetate; 6:4). After completion of the reaction, the mixture was cooled and the resulting product was filtered, dried and recrystallized from methanol to afford the pure product (Scheme No.1). All the products were crystalline and fully characterized on the basis of their melting-points, elemental analyses and spectral data (IR, ¹H NMR, ¹³C NMR and mass spectra (LCMS)).

Spectral data for compounds (4a-4g)

9-Phenyl-3, 3, 6, 6-tetramethyl-10-p-tolyl-hexahydroacridine-1, 8-dione (4a)

Red solid: Yield-86%; M.P-214-216°C IR (KBr, cm⁻¹): 3048, 2968, 2874, 1665, 1612, 1587, 1371, 1346; ¹HNMR (400 MHz, CDCl₃) δppm: 0.947 (s, 6H, 2×CH₃), 1.090 (s, 6H, 2×CH₃), 2.115–2.346 (m, 8H, 4×CH₂), 2.387 (s, 3H, CH₃), 5.278 (s, 1H, CH), 7.078-7.346 (m, 5H, Ar-H), 7.482 (d, J = 8.8 Hz, 2H, Ar-H), 7.554 (d, J = 9.2 Hz, 2H, Ar-H) ppm; ¹³CNMR (100MHz, CDCl₃) δppm: 20.94, 27.08, 29.57, 31.55, 33.58, 41.59, 50.42, 110.57, 114.6, 128.28, 128.79, 129.25, 130.19, 132.08, 133.51, 137.51, 139.09, 150.27, 151.67, 195.08; LCMS (m/z): 440.49 (M+H)⁺; Molecular formulae: C₃₀ H₃₃ NO₂; Analysis of Elements: Calculated: C- 82.00; H- 7.52; N- 3.19; Obtained: C- 81.92; H- 7.50; N- 3.29.

9-(4-Hydroxyphenyl)-3, 3, 6, 6-tetramethyl-10-p-tolyl-hexahydroacridine-1, 8-dione (4b)

Pale Red solid: Yield-92%; M.P-224-226°C; IR (KBr, cm⁻¹): 3372, 3058, 2987, 2884, 1643, 1619, 1568, 1360, 1349; ¹HNMR (400 MHz, CDCl₃)

δ ppm: 0.957 (s, 6H, 2 \times CH₃), 0.997 (s, 6H, 2 \times CH₃), 2.021–2.186 (m, 8H, 4 \times CH₂), 2.354 (s, 3H, CH₃), 5.225 (s, 1H, CH), 7.115–7.2474 (m, 4H, Ar-H), 7.564 (d, J = 7.2 Hz, 2H, Ar-H), 7.843(d, J = 8.8 Hz, 2H, Ar-H), 9.063 (s, 1H, OH); ¹³CNMR (100MHz,CDCl₃) δ ppm: 20.77, 27.17, 29.06, 31.98, 34.84, 41.45, 50.74, 109.04, 113.59, 128.25, 128.88, 129.36, 130.58, 131.89, 133.66, 137.04, 139.54, 151.56, 152.09, 194.79; LCMS (m/z): 456.15 (M+H)⁺; Molecular formulae: C₃₀ H₃₃ NO₃; Analysis of Elements: Calculated: C- 79.12; H- 7.25; N- 3.08; Obtained: C-79.05; H-7.22; N- 3.03.

9-(4-Methoxyphenyl)-3, 3, 6, 6-tetramethyl-10-p-tolyl-hexahydroacridine-1, 8-dione (4c)

Pale Red solid: Yield-92%; M.P-204-206°C; IR (KBr, cm⁻¹): 3078, 2972, 2869, 1662, 1605, 1574,1357, 1350; ¹HNMR (400 MHz, CDCl₃) δ ppm: 0.915 (s, 6H, 2 \times CH₃), 1.124 (s, 6H, 2 \times CH₃), 2.134–2.276 (m, 8H, 4 \times CH₂), 2.288 (s, 3H, CH₃), 3.658 (s, 3H, OCH₃), 5.185 (s, 1H, CH), 7.115–7.384 (m, 4H, Ar-H), 7.447 (d, J = 8.8 Hz, 2H, Ar-H), 7.971 (d, J = 8.0 Hz, 2H, Ar-H); ¹³CNMR (100MHz, CDCl₃) δ ppm: 21.54, 26.82, 28.54, 31.08, 33.95, 41.55, 50.86, 109.07, 114.56, 127.97, 128.57, 129.56, 130.48, 131.38, 133.55, 136.82, 139.04, 150.87, 152.51, 195.59; LCMS (m/z): 470.47 (M+H)⁺; Molecular formulae: C₃₁H₃₅NO₃; Analysis of Elements: Calculated: C- 79.32; H- 7.46; N- 2.98; Obtained: C- 79.25; H- 7.45; N-3.09.

9-(4-Methylphenyl)-3, 3, 6, 6-tetramethyl-10-p-tolyl-hexahydroacridine-1, 8-dione (4d)

Pale Red solid: Yield-90%; M.P-218-220°C: IR (KBr, cm⁻¹): 3089, 2965, 2879, 1650, 1610, 1572, 1375, 1344; ¹HNMR (400 MHz, CDCl₃) δ ppm: 0.917 (s, 6H, 2 \times CH₃), 1.024 (s, 6H, 2 \times CH₃), 2.114–2.241 (m, 8H, 4 \times CH₂), 2.450 (s, 3H, CH₃), 5.257 (s, 1H, CH), 7.105–7.258 (m, 4H, Ar-H), 7.369 (d, J = 7.6 Hz, 2H, Ar-H), 7.487 (d, J = 8.0 Hz, 2H, Ar-H); ¹³CNMR (100MHz, CDCl₃) δ ppm: 20.89, 27.53,29.82, 31.74, 33.5, 39.77, 51.76, 108.58, 114.08, 126.89, 127.45, 129.07, 130.45, 132.14, 132.86, 136.50, 140.47, 150.04, 151.73, 195.44; LCMS (m/z): 454.87 (M+H)⁺; Molecular formulae: C₃₁H₃₅NO₂; Analysis of Elements: Calculated: C- 82.12; H- 7.72; N- 3.09; Obtained: C-82.05; H-7.70; N- 3.17.

9-(4-Chlorophenyl)-3, 3, 6, 6-tetramethyl-10-p-tolyl-hexahydroacridine-1, 8-dione (4e)

Pale Red solid: Yield-88%; M.P-232-234°C; IR (KBr, cm⁻¹): 3078, 2979, 2870, 1640, 1618, 1555,1372, 1346; ¹HNMR (400 MHz, CDCl₃) δ ppm: 0.887 (s,6H, 2 \times CH₃), 1.074 (s, 6H, 2 \times CH₃), 2.078-2.292 (m, 8H, 4 \times CH₂), 2.348 (s, 3H, CH₃), 5.224 (s, 1H, -CH-), 2.087–2.287 (m, 8H, 4 \times CH₂), 7.089–7.247 (m, 4H, Ar-H), 7.478 (d, J = 8.0 Hz, 2H, Ar-H), 7.547 (d, J = 9.2 Hz, 2H, Ar-H); ¹³CNMR (100MHz, CDCl₃) δ ppm: 21.01, 26.51, 29.42, 32.87, 33.39, 41.87, 50.64, 108.79, 113.44, 128.24, 128.98, 129.56, 130.64, 132.49, 132.88,136.56 139.07, 151.08, 151.19, 196.99; LCMS (m/z): 474.45(M+H)⁺; Molecular formulae: C₃₀ H₃₂ ClNO₂; Analysis of Elements: Calculated: C-76.04; H- 6.76; N- 2.96; Obtained: C-75.94; H- 6.78; N-2.99.

9-(4-Bromophenyl)-3, 3, 6, 6-tetramethyl-10-p-tolyl-hexahydroacridine-1, 8-dione (4f)

Dark Red solid: Yield-88%; M.P-241-243°C; IR (KBr, cm⁻¹): 3042, 2970, 2880, 1644, 1609, 1547,1378, 1355; ¹HNMR (400 MHz, CDCl₃) δ ppm: 0.874 (s,6H, 2 \times CH₃), 1.099 (s, 6H, 2 \times CH₃), 2.022–2.124 (m, 8H, 4 \times CH₂), 2.230 (s, 3H, CH₃), 5.320 (s, 1H, CH), 7.104–7.384 (m, 4H, Ar-H), 7.484 (d, J = 8.8 Hz, 2H, Ar-H), 7.778 (d, J =6.8 Hz, 2H, Ar-H); ¹³CNMR (100MHz, CDCl₃) δ ppm: 20.89, 27.20, 29.09, 32.44, 33.59, 40.79, 51.07,109.05, 113.55, 127.65, 128.45, 129.02, 130.54, 131.88, 132.12, 136.81, 141.54, 150.55, 151.55, 195.84; LCMS (m/z): 518.95 (M+H)⁺; Molecular formulae: C₃₀ H₃₂ Br NO₂; Analysis of Elements: Calculated :C-69.51; H- 6.18; N- 2.70; Obtained: C- 69.44; H- 6.16; N-2.78.

9-(4-Cyanophenyl)-3, 3, 6, 6-tetramethyl-10-p-tolyl-hexahydroacridine-1, 8-dione (4g)

Dark Red solid: Yield-85%; M.P-194-196°C; IR (KBr, cm⁻¹): 3058, 2965, 2872, 1645, 1615,2220, 1550, 1375, 1340; ¹HNMR (400 MHz, CDCl₃) δ ppm: 0.884 (s, 6H, 2 \times CH₃), 0.976 (s, 6H, 2 \times CH₃), 2.056-2.244 (m, 8H, 4 \times CH₂), 2.394 (s, 3H, CH₃), 5.254 (s, 1H, CH), 7.110–7.355 (m, 4H, Ar-H), 7.577 (d, J = 5.6 Hz, 2H, Ar-H), 7.858 (d, J = 9.4 Hz, 2H, Ar-H); ¹³CNMR (100MHz, CDCl₃) δ ppm: 21.39, 26.54, 29.92, 32.08, 33.53, 41.43, 50.56,

110.54, 113.52, 119.44, 128.63, 128.57, 129.56, 132.07, 132.01, 136.46, 140.04, 151.08, 152.53, 195.99; LCMS (m/z): 465.58 (M+H)⁺; Molecular formulae: C₃₁ H₃₂ N₂ O₂; Analysis of Elements: Calculated: C- 80.17; H- 6.89; N- 6.03; found: C- 80.11; H- 6.87; N- 6.13.

RESULTS AND DISCUSSION

The procedure afforded a versatile, environmentally benign, one-pot three-component synthesis of 9-arylacridinediones by the reaction of substituted aromatic aldehydes, dimedone and), p-Toluidine 3 (1.5mmol), Ammonium molibdate (1mmol %) and isopropanol under thermal condition (Scheme No.1).

In an initial endeavor, substituted aromatic aldehydes (1mmol), dimedone (2mmol) and p-Toluidine (1.5mmol) were stirred at 70°C in isopropanol under reflux conditions. After 5 h, only 60% of the expected product 4c was obtained. To developed the yield and optimize the reaction conditions, the same reaction were carried out in the presence of various amounts of Ammonium molibdate acid under similar conditions. In all reactions, the conditions were optimized for 100% conversion.

The reaction condition of these derivatives was optimized at various catalyst, different amount of the catalyst and different solvent are used. The maximum yield of the compounds obtained in presence of zinc oxide (ZnO) catalyst than oxidative related catalyst such as titanium dioxide (TiO₂), copper oxide (CuO), Ammonium molibdate whereas different amount of catalyst utilized during the reaction (Table No.1).

The different solvents were used during the reaction that were evaluated (DMF, Isopropanol acetonitrile, ethanol, methanol, cyclohexane) in the model reaction. It was found to be the best medium for the reaction, with 92% product yield and was therefore used as the solvent for subsequent reactions on the merits of higher yield, green nature and easy work-up.

A significant improvement was identified, the yield of 4c being increased to 92%. Use of only 1.5m mol% was sufficient to drive the reaction forwards

within 2.0hrs. The maximum amounts of the catalyst did not improve the results. Although, use of 2.0mmol% Ammonium molibdate permitted the reaction time to be decreased to 1 h, the yield unexpectedly decreased to 77% as shown Table No.3.

Biological activities

Antibacterial and antifungal activities

The titled derivatives were evaluated for their *in-vitro* antibacterial and antifungal activities following micro broth dilution method. The *in vitro* antibacterial activity was examined against gram-positive (*Bacillus subtilis* and *Staphylococcus aureus*) and gram-negative (*Escherichia coli* and *P. aeruginosa*) microorganisms. The *in vitro* antifungal activity was evaluated against *Aspergillus Niger* and *C.albicans* microorganisms. The standard drugs used for this study were Streptomycin was used for antibacterial screening. Ketonazole was used for antifungal screening. The standard strains used for screening of antibacterial and antifungal activities were procured from the Culture collection and geneank (MTCC), Chandigarh, India. Mueller Hinton Broth was used as a nutrient medium for bacteria and Sabouraud dextrose Broth for fungal growth. Inoculums size for test strain was adjusted to 10⁸ CFU/mL by comparing the turbidity. The results were recorded in the form of primary and secondary evaluation. The stock solution (2000µg/mL) of the compounds under investigation and standard drugs were prepared by successive two fold dilution.

In the preliminary examination 500, 250 and 100µg/mL concentrations of the compounds were used. The compounds found to be active in this primary screening were further examination. In secondary screening, 200, 100, 50 and 25µg/mL concentrations were used. The inoculated wells were incubated overnight at 37°C in a humid atmosphere. The highest dilution showing complete inhibition was considered as a minimum inhibition concentration (MIC). The MIC values revealed that the synthesized compounds showed moderate to good inhibition. Compounds 4d, 4e exhibited good excellent activities against bacterial strains. The MIC values of antifungal activity shown that

compound 4c and 4c exhibited good activity against all fungal strain. Antimicrobial activity of compounds (4a-4g) is listed in Table No.1.

Table No.1: The reaction of aryl aldehyde, dimedone and Ammonium molibdate (4b)

Entry	Catalyst	Time (hrs)	Yield (%)
1	ZnO	8	59
2	TiO ₂	7	70
3	CuO	10	68
4	Ammonium molibdate	5	92

Table No.2: The reaction of aryl aldehyde, dimedone and Ammonium molibdate (4b)

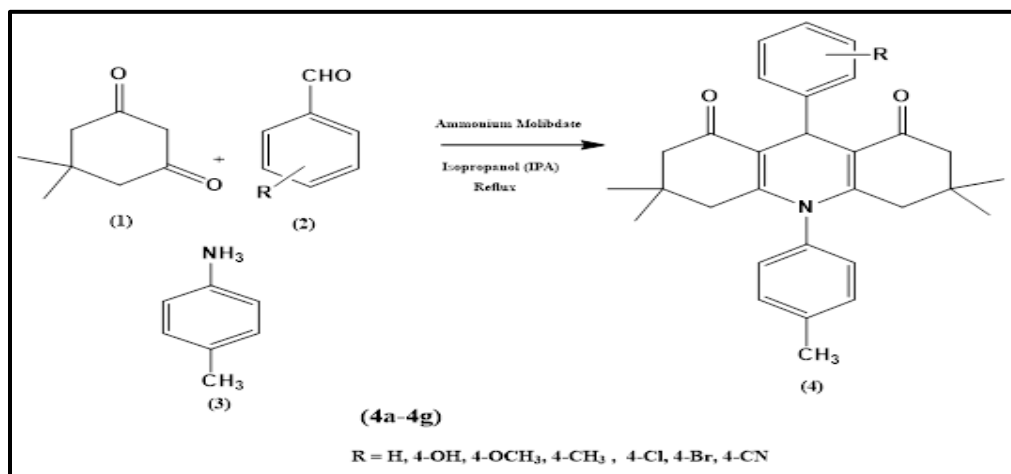
Entry	Catalyst	Time (hrs)	Yield (%)
1	DMF	5	45
2	IPA	5	92
3	CH ₃ CN	5	62
4	EtOH	5	56
5	MeOH	5	67

Table No.3: Different amounts of catalyst in Isopropanol at reflux (4b)

Entry	Amount catalyst (mmol)	Time (hrs)	Yield (%)
1	0.5	3	Traces
2	1.0	3	42
3	1.5	3	58
4	2.0	3	92

Table No.4: Antimicrobial activity of compounds (4a-4g)

S.No	Entry	Antibacterial MIC ($\mu\text{g/mL}$)				Antifungal MIC ($\mu\text{g/mL}$)	
	Strains	<i>B. subtilis</i>	<i>S. aureus</i>	<i>P. aeruginosa</i>	<i>E. coli</i>	<i>A. Niger</i>	<i>C. Albicans</i>
1	4a	06	05	08	07	05	05
2	4b	17	16	18	17	13	14
3	4c	18	16	18	14	13	14
4	4d	22	21	20	21	16	17
5	4e	21	20	19	18	16	18
6	4f	20	18	18	17	17	16
7	4g	08	07	10	09	08	06
8	Streptomycin	25	25	25	25	-	-
9	Ketozole	-	-	-	-	22	22
10	DMSO						



Scheme No.1

CONCLUSION

We have developed a new, easy, an efficient process for synthesis of substituted acridinediones derivatives via one-pot three-component condensation of substituted aromatic aldehyde, dimedone and P-toluidine in an isopropanol medium with ammonium molybdate as an efficient catalyst. The mildness of the conversion, the experimental simplicity, compatibility with various functional groups, excellent product yields and the easy work-up procedure make this approach attractive for synthesizing a variety of such derivatives. Further, the antimicrobial activity of the titled derivatives was studied. The derivatives having electron withdrawing groups exhibited excellent active potential.

ACKNOWLEDGEMENT

The authors gratefully acknowledge to the management of PRISM PG and DG College Visakhapatnam, India, for laboratory support. The authors also gratefully thank both referees for their helpful critical suggestions.

CONFLICT OF INTEREST

The author declares no conflict of interest.

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Please cite this article in press as: Srinivasarao S et al. An efficient one pot synthesis of acridinediones employed by ammonium molybdate and study of antimicrobial activity, *Asian Journal of Research in Chemistry and Pharmaceutical Sciences*, 12(1), 2024, 1-7.