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Aluminized polyborate catalyzed efficient solvent-free synthesis of 1,8-dioxo-decahydroacridines via hantzsch condensation**Dilip Aute^a, Amol Parhad^b, Vitthal Vikhe^b, Bhagwat Uphade^a and Anil Gadhave^{a*}**^aDepartment of Chemistry and Research Centre, Padmashri Vikhe Patil College, Pravaranagar, Ahmednagar, Maharashtra, India^bDepartment of Chemistry, Pravara Rural Engineering College, Loni, 413736, India**CHRONICLE***Article history:*

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*Keywords:**Hantzsch synthesis**Aluminized polyborate**1,8-dioxo-decahydroacridine**Multicomponent reaction***ABSTRACT**

In this work we report, aluminized polyborate catalyzed facile and efficient one-pot four component Hantzsch synthesis of 1,8-dioxo-decahydroacridines using substituted aromatic aldehydes (1 mmol), dimedone (2 mmol) and ammonium acetate (1.5 mmol) at 95-100°C under solvent-free condition. The prominent advantages of this methodology are good product yields (85-95%), eco-friendliness, mild reaction conditions and use of inexpensive catalyst. The structures of the targeted molecules were examined by FT-IR, ¹H-NMR and mass spectral techniques.

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1. Introduction

Multicomponent reactions (MCRs) are one-pot operations utilizing more than two starting materials for the synthesis of structurally diversified compounds of biological significance.¹ The MCRs offers easy construction of biologically important molecules by using green chemistry features like good atom economy, minimization of waste, environmental benign operations, simple purification process and use of less hazardous chemicals.² The 1,8-dioxo-decahydroacridine derivatives exhibit good biological activities such as anti-cancer, anti-inflammatory, antimicrobial, antiviral, anti-tubercular, anti-parasitic and fungicidal. They also find applications as an inhibitors of acetyl cholinesterase, fluorescent materials for visualization of biomolecules, dyes and in laser technologies.^{3,4} They are also used as photo initiators, photo sensitizers and selective fluoride ion chemosensor.⁵ They also finds application in treatment of Alzheimer's disease due to their platelet antiaggregatory activity.⁶

The literature survey reveals wide applications of boric acid as a catalyst in organic transformations. Its catalytic importance lead to the development of more reactive aluminized polyborate catalyst.⁷ The aluminized polyborate catalyst was used in our previous work for the solvent-free green synthesis of various heterocyclic molecules.⁸⁻¹⁰ In literature several methodologies are been reported for the synthesis of acridinediones among them some are β-CD-mono-SO₃H,¹¹ CdS thin film,¹² MNPs-N-propyl-benzoguanamine-SO₃H,¹³ AFGONs,¹⁴ GO/CR-Fe₃O₄,¹⁵ Iron-4 A molecular sieves,¹⁶ MCM-41-Pr-THEIC,¹⁷ Pd/AlO(OH),¹⁸ urease,¹⁹ Fe₃O₄@SiO₂@Ni-Zn-Fe LDH,²⁰ nano-Fe₃O₄@SiO₂-HBP-FeCl₃,²¹ citric acid,²² TEMPO/CuCl₂,²³ N-butyl pyridinium heptachlorodialuminate,²⁴ L-proline,²⁵ microwave irradiation,²⁶ Na⁺-MMT-[bip]-NH₂⁺ HSO₄,²⁷ MSrGONCs,²⁸ phosphate fertilizers,²⁹ ChCl:urea deep eutectic solvent,³⁰ Fe₃O₄@SiO₂-Pr-Sulfurol-SO₃H MNPs³¹ and ultrasound-assisted³² reaction.

The acridine derivatives find tremendous application in the industrial field as a therapeutic agent.³³ Though these reported protocols offer several significant features but still possess some margins like use of non-green solvent, expensive

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catalyst, complicated catalyst preparation, low product yield, use of excessive catalyst and formation of side product. Therefore there is still a need to develop a more efficient environmentally benign methods for the synthesis of 1,8-dioxo-decahydroacridine derivatives. With this view and in continuation to our work in the development of new methods for synthesis of important heterocyclic compounds,³⁴⁻³⁶ herein we report aluminized polyborate as mild solid acid catalyst for the green and efficient synthesis of 1,8-dioxo-decahydroacridines with good product yield under solvent free conditions.

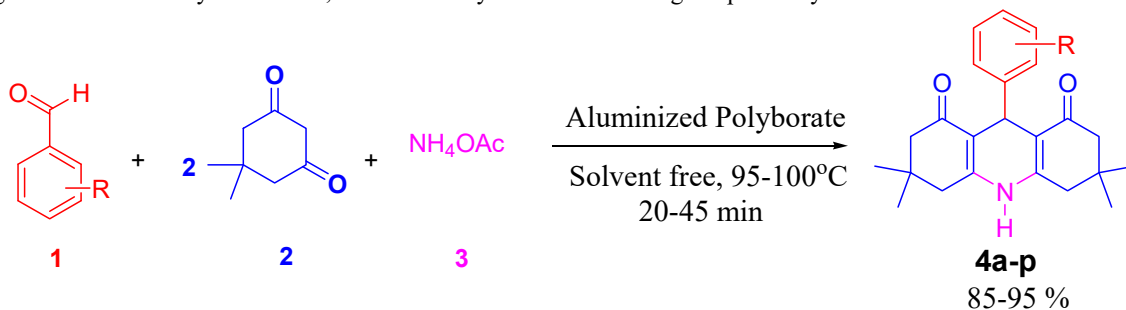


Fig. 1. Synthesis of 1,8-dioxo-decahydroacridines

2. Results and Discussion

In the present work, previously reported aluminized polyborate⁸⁻¹⁰ was successfully employed for the one-pot synthesis of 1,8-dioxo-decahydroacridines by using substituted aromatic aldehydes (1 mmol), dimedone (2 mmol) and ammonium acetate (1.5 mmol) (**Fig. 1**).

The designed study was started with the selection of model reaction between 4-chloro benzaldehyde (1 mmol), dimedone (2 mmol) and ammonium acetate (1.5 mmol) for the synthesis of 1,8-dioxo-decahydroacridine derivative (4a). In the first step, we checked the suitable solvent and temperature essential for the synthesis of 1,8-dioxo-decahydroacridines. The reaction was carried out in solvents like H₂O, EtOH and H₂O: EtOH (1:1) under various conditions. The outcomes are given in Table 1 which indicates that reaction carried out in the above listed solvent systems at room temperature, reflux conditions and under ultrasonication does not give satisfactory yields of product. Then the model reaction was performed at solvent-free condition at room temperature, 75-80°C and 95-100°C. Among them the most appropriate condition for successful synthesis of 1,8-dioxo-decahydroacridines is solvent-free reaction at 95-100°C.

Table 1. Effect of solvent and temperature on the synthesis of 1,8-Dioxo-decahydroacridines

Entry	Solvent	Condition (°C)	Time (min)	Yield (%)
1	H ₂ O	rt	60	NR
2	H ₂ O	reflux	60	15
3	H ₂ O	ultrasonication	20	19
4	EtOH	rt	60	NR
5	EtOH	reflux	60	40
6	EtOH	ultrasonication	20	27
7	EtOH:H ₂ O	reflux	60	42
8	Solvent-free	rt	60	NR
9	Solvent-free	75-80	30	60
10	Solvent-free	95-100	30	92
11	Solvent-free	95-100	25	92

Reaction conditions: 4-chloro benzaldehyde (1 mmol), dimedone (2 mmol) and ammonium acetate (1.5 mmol) and aluminized polyborate (80 mg) catalyst, NR: no reaction.

Then the model reaction was carried out under the above optimized condition to confirm the appropriate amount of catalyst required to complete the reaction. The results obtained (Table 2) indicate that an 80 mg aluminized polyborate catalyst is enough to carry the reaction with good product yield.

Table 2. The effect of amount of catalyst on synthesis of 1, 8 Dioxo-decahydroacridines

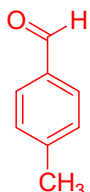
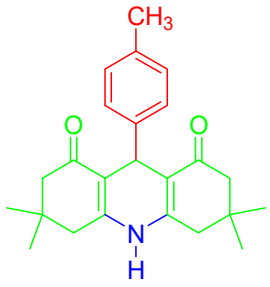
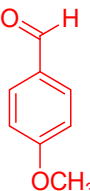
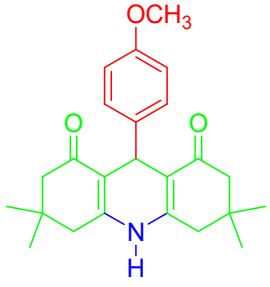
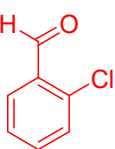
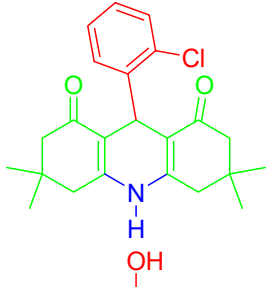
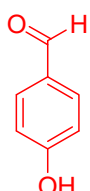
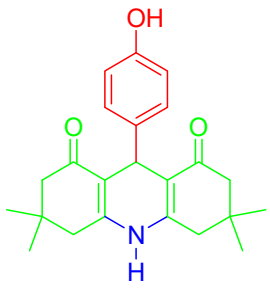
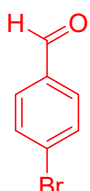
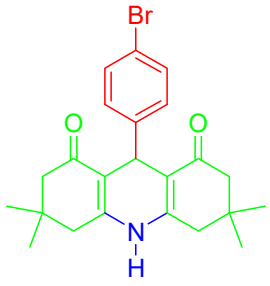
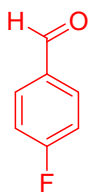
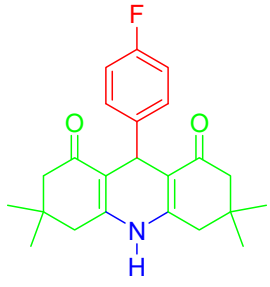
Entry	Catalyst (mg)	Time (min)	Yield (%)
1	0	25	NR
2	20	25	34
3	40	25	51
4	60	25	78
5	80	25	92
6	80	35	92
7	100	25	92

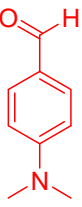
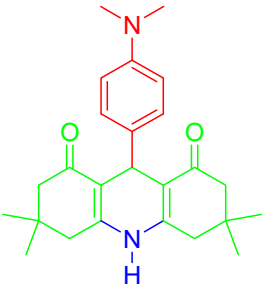
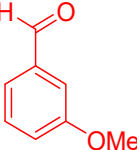
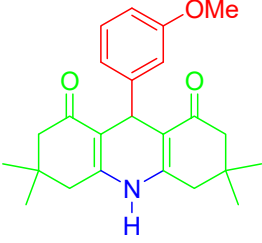
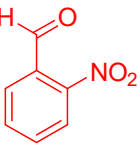
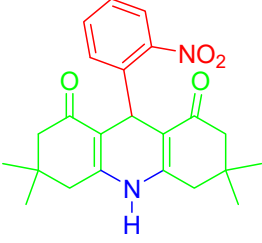
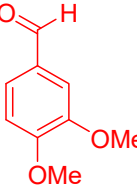
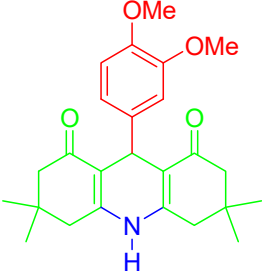
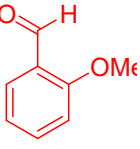
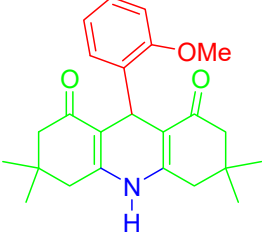
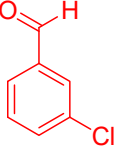
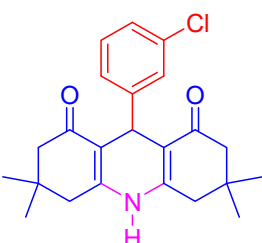
Reaction conditions: 4-chloro benzaldehyde (1mmol), dimedone (2 mmol) and ammonium acetate (1.5 mmol) under solvent-free condition at 95-100°C, NR: no reaction.

Subsequently the next factor of study was to check the applicability of the optimized condition for the synthesis of a broad range of diversified derivatives of 1,8-dioxo-decahydroacridine using the differently substituted aromatic aldehydes (1a-o). The results obtained indicate (Table 3) that all kinds of aldehydes gave satisfactory product yields (85-95%). The reaction worked smoothly with aromatic aldehydes having electron donating as well as electron withdrawing groups. The ortho substituted aldehydes gave comparatively low product yield due to steric reasons.

Table 3. Synthesis of 1, 8-dioxo decahydroacridines (4a-o)

Sr. No.	Aldehyde	Product	Time (min)	Yield (%)	Melting Point (°C)		Reference
					Observed	Reported	
4a			25	92	298-300	298	12
4b			20	95	284-285	286	12
4c			30	90	293-294	293-295	17
4d			25	91	274-275	275-276	17

4e			35	87	>300	>300	20
4f			40	88	275-276	275	17
4g			45	86	219-220	219-221	15
4h			40	85	>300	>300	17
4i			30	91	>300	>300	27
4j			30	92	273-275	275-276	23

4k			40	86	280-281	280-282	13
4l			25	89	286-287	285-287	15
4m			40	86	294-295	295-297	27
4n			45	88	286-287	288-290	13
4o			45	86	297-298	>300	20
4p			30	89	290-291	289-293	15

Reaction conditions: aldehyde (1 mmol), dimedone (2 mmol), ammonium acetate (1.5 mmol) and aluminized polyborate (80 mg) under solvent-free condition at 95-100°C.

Comparison of the present work with the literature reported protocols

The present protocol was compared with that reported in literature for the synthesis of acridinedione derivatives. It was found that the present catalytic system is superior in terms of easy catalyst preparation and its non-toxic nature, good product yields, short reaction time and environmentally benign method.

Table 4. Comparison of present work with literature reported various catalytic systems for the synthesis of acridinedione derivatives

Entry	Catalyst /Solvent / Condition	Temp	Time	Yield (%)	Reference
1	CdS thin film NPs, EtOH	75-80°C	1.50-2.15 h	81-88	12
2	MNPs-N-propyl-benzoguanamine-SO ₃ H, 3EtOH: 1H ₂ O	reflux	15-25 min	80-93	13
3	AFGONs, EtOH	rt	3 h	86-92	14
4	GO/CR-Fe ₃ O ₄ nanocomposite, EtOH	70°C	10 min	90-96	15
5	Fe ³⁺ /4A, EtOH	reflux	14 h	50-99	16
6	Pd/AlO(OH), DMF, ultrasonication	40°C	60 min	>90	18
7	Fe ₃ O ₄ @SiO ₂ @Ni-Zn-Fe LDH, solvent-free	70-80°C	15-35 min	80-96	20
8	citric acid, EtOH	reflux	100-300 min	45-90	22
9	Aluminized Polyborate, solvent-free	95-100°C	20-45 min	85-95	Present work

3. Conclusions

In this article we have reported, aluminized polyborate catalyzed efficient and green one-pot multi-component solvent-free protocol for the synthesis of acridinediones by the reaction of substituted aromatic aldehydes, dimedone and ammonium acetate. The characteristic features of this methodology are operational simplicity, mild reaction conditions, good to excellent product yields, absence of toxic solvent, short reaction time and environmentally friendly approach.

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4. Experimental

4.1. Materials and Methods

The commercially available aromatic aldehydes, dimedone, ammonium acetate, boric acid, AlCl₃ and required solvents of Loba make were purchased from Atmaja Sales, Aurangabad, Maharashtra (IN) and used without purification. The melting points were measured on digital melting/boiling point EQ 730 equiptronics apparatus. ¹H and ¹³C spectra were recorded on Bruker Avance at 500 MHz NMR spectrometer at 500 and 125 MHz respectively. Chemical shifts were measured in CDCl₃ as solvent relative to TMS as the internal standard. Infrared spectra were recorded on Perkin-Elmer 400 FT-IR spectrometer KBr discs. Mass spectra of the synthesized compounds were recorded on water, Q-TOF Micromass (ESI-MS) spectrometers. The progress of reaction was monitored by thin layer chromatography technique (Silica G₆₀ F₂₅₄ plates, Merck).

4.2 Synthesis of Catalyst

The aluminized polyborate was synthesized by method reported in literature using commercially available inexpensive boric acid and aluminium trichloride.⁷ The catalyst was characterized by FT-IR, XRD, SEM and EDAX techniques which are reported in our previous work on aluminized polyborate catalyst.⁸⁻¹⁰

4.3 General procedures for the synthesis of 1,8-dioxodecahydroacridines

In 25 ml round bottom flask, the mixture of aromatic aldehyde (1 mmol), dimedone (2 mmol), ammonium acetate (1.5 mmol) and aluminized polyborate (80 mg) catalyst was heated under solvent-free condition at 90-100°C. The progress of reaction was monitored by using TLC (n-hexane 7 mL and ethyl acetate 3 mL). After completion of the reaction, the mixture was allowed to attain room temperature. This reaction mixture was washed with 10 % sodium bisulphate to remove unreacted aldehyde. Then the reaction mixture was recrystallized by using ethanol to give pure products. The formation of 1,8-dioxo-acridine derivatives were confirmed by comparing their physical data with literature reported authentic compounds. Some of them were characterized by FT-IR, NMR and Mass spectral techniques.

4.4 Spectral analysis of some compounds

9-(4-chlorophenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2*H*,5*H*)-dione (4a) : IR (*v* max, cm⁻¹): 3462 (NH), 3078 (CH₃, str.), 1647 (C=O), 1608 (C=C), 1591 (C=C), 1089 (Ar-Cl); ¹H-NMR (500 MHz, CDCl₃, δ ppm): 8.63 (s, 1H, NH), 7.27 (d, 2H, J=8.4 Hz, ArH), 7.15 (d, 2H, J=8.4 Hz, ArH), 5.06 (s, 1H, CH), 2.12 (m, 8H,

4 x CH₂), 1.06 (s, 6H, 2 x CH₃), 0.94 (s, 6H, 2 x CH₃); ¹³C-NMR (125 MHz, CDCl₃, δ ppm): 196.23 (C=O), 150.00 (Ar-C₄-Cl), 145.34 (Ar-C₃), 131.53 (Ar-C₂), 129.46 (Ar-C₁), 128.09 (=C-NH), 112.63 (=C-C=O), 50.86 (CH-Ar), 40.46 (C(CH₃)₂), 33.38 (CH₂), 32.59 (CH₂), 29.59 (CH₃), 27.02 (CH₃); MS for C₂₃H₂₆ClNO₂ calculated (m/z): 384 [M+H]⁺ and observed 384.35 [M + H]⁺.

9-(4-methoxyphenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione (4f) :

IR (ν max, cm⁻¹): 3275 (NH), 3072 (CH₃, str.), 1643 (C=O), 1606 (C=C), 1508 (C=C), 1263 (C-O); ¹H-NMR (500 MHz, CDCl₃, δ ppm): 8.64 (s, 1H, NH), 7.24 (d, 2H, J=8 Hz, ArH), 6.70 (d, 2H, J=8 Hz, ArH), 5.04 (s, 1H, CH), 3.65 (s, 3H, OCH₃), 2.11 (m, 8H, 4 x CH₂), 1.05 (s, 6H, 2 x CH₃), 0.94 (s, 6H, 2 x CH₃); ¹³C-NMR (125 MHz, CDCl₃, δ ppm): 196.35 (C=O), 157.62 (Ar-C₄-OMe), 149.82 (Ar-C₃), 139.29 (Ar-C₂), 128.92 (Ar-C₁), 113.27 (=C-NH), 113.12 (=C-C=O), 54.98 (OCH₃), 50.94 (CH-Ar), 40.38 (C(CH₃)₂), 32.76 (CH₂), 32.55 (CH₂), 29.61 (CH₃), 27.06 (CH₃); MS for C₂₄H₂₉NO₃ calculated (m/z): 379 [M+H]⁺ and observed 380.43 [M+H]⁺.

9-(3-chlorophenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione (4p) :

IR (ν max, cm⁻¹): 3396 (NH), 3064 (CH₃, str.), 1718 (C=O), 1641 (C=O), 1610 (C=C), 1471 (C=C), 1070 (Ar-Cl); ¹H-NMR (500 MHz, CDCl₃, δ ppm): 8.52 (s, 1H, NH), 7.30 (s, 1H, ArH), 7.25 (d, 1H, J=7.8 Hz, ArH), 7.12 (t, 1H, ArH), 7.04 (d, 1H, J=7.8 Hz, ArH), 5.07 (s, 1H, CH), 2.14 (m, 8H, 4 x CH₂), 1.07 (s, 6H, 2 x CH₃), 0.96 (s, 6H, 2 x CH₃); ¹³C-NMR (125 MHz, CDCl₃, δ ppm): 196.17 (C=O), 150.02 (Ar-C₃-Cl), 148.70 (Ar-C₄), 133.75 (Ar-C₂), 129.24 (Ar-C₁), 128.17 (Ar-C₃), 126.44 (Ar-C₆), 119.62 (=C-NH), 112.51 (=C-C=O), 50.84 (CH-Ar), 40.52 (C(CH₃)₂), 32.61 (CH₂), 29.55 (CH₂), 28.05 (CH₃), 27.06 (CH₃); MS for C₂₃H₂₆ClNO₂ calculated (m/z): 384 [M+H]⁺ and observed 384.37 [M+H]⁺.

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