

SYNTHESIS OF 1, 3-OXAZINE DERIVATIVES CATALYZED UNDER CERIC AMMONIUM NITRATE IN AN AQUEOUS MEDIUM

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Abstract

The synthesis of heterocyclic compounds has gained significant importance due to their wide range of biological and pharmaceutical applications. Among these, 1,3-oxazine derivatives represent an important class of compounds exhibiting diverse biological activities such as antimicrobial, anti-inflammatory, and anticancer properties. In the present study, an efficient and environmentally benign method has been developed for the synthesis of 1,3-oxazine derivatives using ceric ammonium nitrate (CAN) as a catalyst in an aqueous medium. The reaction involves the condensation of suitable aldehydes, amines, and phenolic compounds under mild conditions, leading to the formation of 1,3-oxazine derivatives with good to excellent yields. The use of water as a green solvent and CAN as a readily available, cost-effective catalyst offers significant advantages such as reduced reaction time, operational simplicity, and minimized environmental impact. The methodology avoids the use of hazardous organic solvents and harsh reaction conditions, making it a sustainable approach in organic synthesis. The synthesized compounds were characterized using standard spectroscopic techniques such as IR, NMR, and mass spectrometry. The results demonstrate that this protocol provides an efficient, eco-friendly, and scalable route for the preparation of structurally diverse 1,3-oxazine derivatives. We report that ceric ammonium nitrate mediated the synthesis of 1, 3-oxazine derivatives under the green method. In the reaction, 20 mol% catalysts are sufficient for the complete conversion of the reaction. The condensation reaction of b-naphthol with formaldehyde and primary amines those containing electron-donating as well as electron-withdrawing substituents under the aqueous reaction condition at ambient temperature in the presence of ceric ammonium nitrate has been developed as an efficient and eco-friendly precursor for the determination of 1, 3-oxazine derivatives. This approach has numerous benefits beyond conventional systems, notably ease of use, expense, reusability of the catalyst, and faster reaction times.

Introduction

The one-pot multicomponent reaction has been designed and acknowledged as a useful method for creativity synthesized in drug development processes by companies and academic investigators throughout the last 10 years.¹⁻³ (MCRs) are a clean and effective technique having significant characteristics such as conversion efficiency, minimal by-products, rapid creation of compounds containing complicated structures, and providing a varied set of compounds for biopharmaceutical design variables.⁴ There is a need for more effective and less hazardous ways to complete molecular transformations in today's chemical technology. The majority of catalytic reaction solvents are harmful to both the environment and humans. As an excellent green solvent, water is frequently employed as a basic mixture and is acquiring greater relevance in conventional biological applications as a result of the aforementioned insurmountable problems.⁵⁻⁷

Multicomponent reactions (MCRs) have recently risen to prominence as a synthetic technique for generating fundamentally complicated chemical structures with appealing bioactivities by forming and breaking numerous carbon-carbon and carbon-heteroatom bonds in a single-step synthesis.⁸ Owing to its unique bioactive components along with anti-bacterial anti-tumor, anti-malarial, and anti-HIV activity, modified 1, 3-oxazine compounds have received a lot of interest.⁹ (Figure 1) Burke et al. reported the first 1, 3-oxazine analogs in 1953 utilizing the Mannich reaction of primary aryl amine with formaldehyde and *b*-naphthol.¹⁰ Chemical catalysts, ionic liquid, zirconia (IV) chloride, alum, thiamin hydrochloride (VB₁), Brønsted acid, and others have been explored in recent decades.¹¹ 1, 3-oxazine analogs have sparked a lot of attention due to their varied genetic characteristics, which include anticonvulsant¹² analgesic,¹³ antibacterial,¹⁴ anti-cancer,¹⁵ anti-tubular¹⁶ actions. The three-component cyclic condensation of cyclic and primary

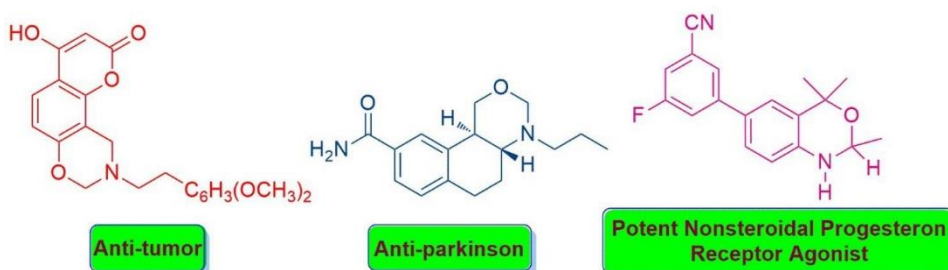


Figure 1. Structure of some bioactive 1, 3-Oxazine derivatives.

Scheme 1. CAN catalyze the synthesis of 1, 3-oxazine derivatives in water at room temperature.

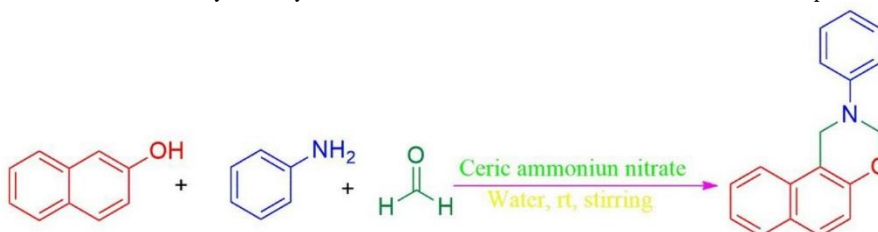


Table 1. Optimization of catalyst loading in the model reaction.

Sr. No.	Catalyst (mol%) ^a	Time (min)	Yield (%) ^b
1	0	12h	NR
2	0.5	2h	28
3	1	60	50
4	1.5	30	70
5	2.0	20	95, 82, 68 ^c
6	2.5	20	95

^aReaction condition: *b*-naphthol (10 mmol), aniline (10 mmol), formaldehyde (20 mmol) in water (5 ml).

^bIsolated yield,

^cCatalyst was reused three times.

aliphatic amines using formaldehyde and substituted phenols has been used to investigate and study 1, 3-oxazine derivatives.¹⁷ Based on the amine, temperature, and location of the compound on phenol, this method produced a variety of polymeric products. Numerous techniques for the preparation of 1, 3-oxazine analogs have been described before.¹⁸⁻²⁰ Just a handful have been developed using multi-component reactions.²¹ Currently, a lot of effort has gone into promoting sustainable and environmentally acceptable synthetic techniques for biochemical transformations that employ non-flammable and non-hazardous solvents like ionic liquid, water, or solvent-free conditions rather than volatile organic solvents.²²⁻³⁰

The ceric ammonium nitrate catalyst was utilized to synthesize 1, 3-oxazine derivatives in an

aqueous medium at room temperature in the current study. This ceric ammonium nitrate was further studied for a fast, efficient, and sustainable method for the synthesis of 1, 3-oxazine derivatives using formaldehyde, amines, and *b*-naphthol as the reactants.

1. Results and discussion

The ceric ammonium nitrate catalyst was utilized to synthesize 1, 3-oxazine derivatives in an aqueous medium at room temperature for the first. To the best of our knowledge, there are

Table 2. Optimization of solvent used in the model reaction.

Sr. No.	Solvent ^a	Time (min)	Yield (%) ^b
1	CHCl ₃	19	24
2	CCl ₄	18	29
3	CH ₃ CN	20	41
4	DMF	20	38
5	EtOH	19	72
6	Water	19	96
7	Solvent-free	180	10

^aAll the reactions were carried out at 40 °C temperature. ^bIsolated yield.

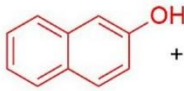
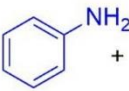
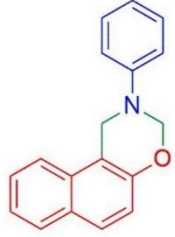
reports on the three-component one-pot synthesis of 1, 3-oxazine derivatives using ceric ammonium nitrate catalyst in an aqueous medium at room temperature. The three-component condensation of formaldehyde, amines, and *b*-naphthol results in a very effective one-pot procedure for the production of 1, 3-oxazine derivatives (Scheme 1).

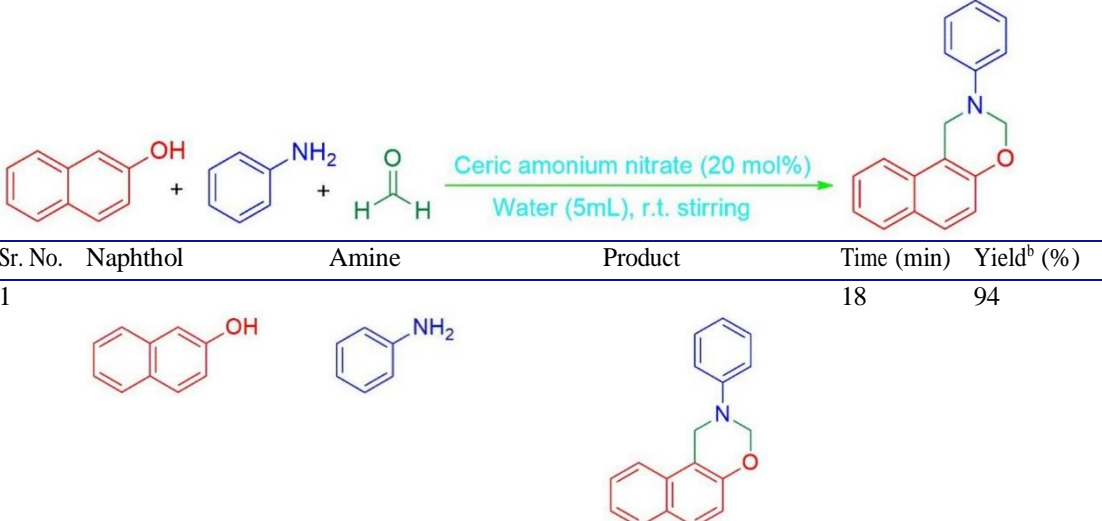
We first looked at the three-component reaction of formaldehyde, aniline, and *b*-naphthol in an aqueous medium at room temperature without a catalyst, the reaction does not give a product (Table 1, Sr. no. 1). When increasing the amount of catalyst (0.5-2 mol%) also increases the yield of the product (Table 1, Sr. no. 2-4). When 2 mol% catalyst was loaded in the reaction it gave an excellent yield (95%) of product. No change occurs when the use of catalyst after increasing the amount by about 2.5 mol% (Table 1, Sr. no. 5). The regenerated ceric ammonium nitrate catalytic performance had also been tested under standard practice models. Following the conclusion of the reaction (TLC), the target product was separated using ethyl acetate extraction. Then aqueous layer is again used for the next cycle.

The catalyst-containing aqueous phase was again reused for three additional cycles, yielding products in 95%, 82%, and 68% yields, correspondingly. After that, again increase the amount of catalyst (2.5 mol%) but does affect the reaction. As a result, ceric ammonium nitrate may be utilized as a recyclable catalyst for multi-component oxazine production (Figure 2).

The impact of different solvents (5 mL) on the aforementioned process has been investigated extensively. The data show that the solvent had an impact on the reaction's effectiveness. Aprotic solvents including chloroform, CCl₄, CH₃CN, and DMF produced low productivity (Table 2, Sr. No. 1-4). Then reactions were carried out by a protic solvent like ethanol to increase the yield of the product (Table 2, Sr. No. 5). The reaction performed in the presence of water gives excellent yield (95%) (Table 2, Sr. No. 6). Without solvents, the reaction does not give a product (Table 2, Sr. No. 7). As a result, the predicted optimal conditions were assumed when the catalyst was 2 mol% ceric ammonium nitrates in H₂O at 40 °C temperature.

Table 3. Scope of the substrate.^a

Sr. No.	Naphthol	Amine	Product	Time (min)	Yield ^b (%)
1				18	94



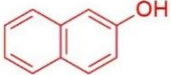
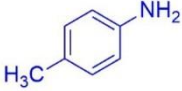
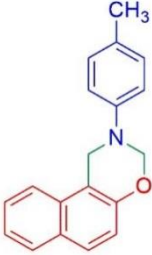

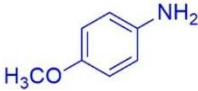
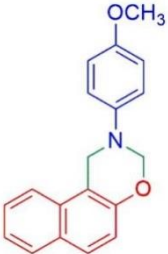

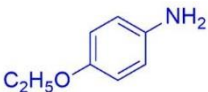
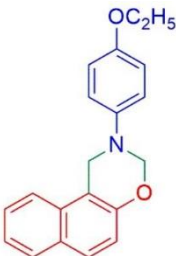
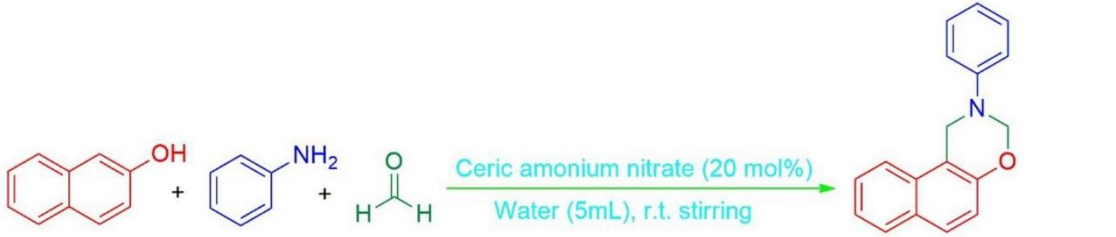

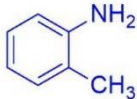
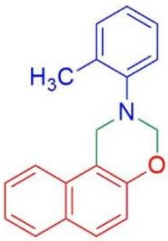

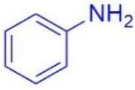
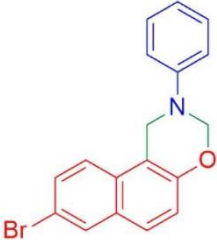
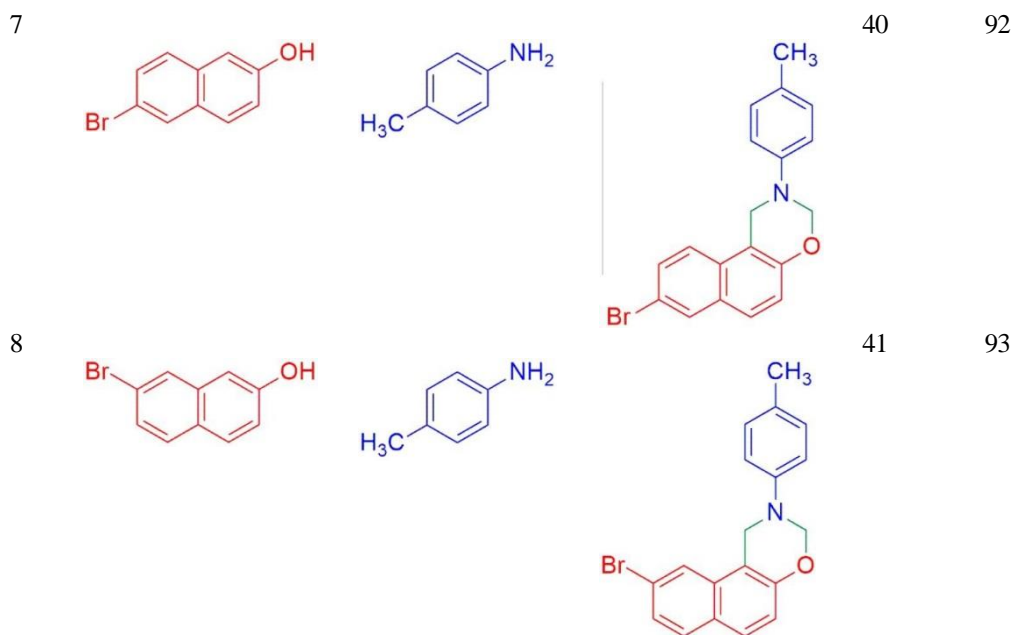
2				40	94
3				40	96
4				50	93

Table 3. Continued.

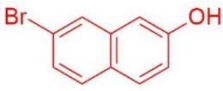
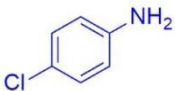
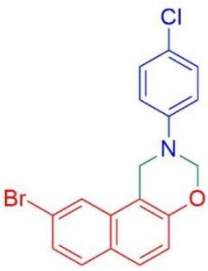
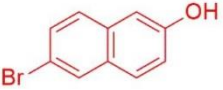
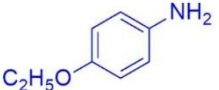
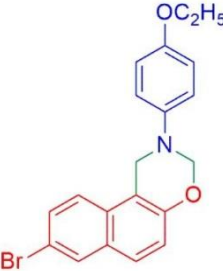
					
Sr. No.	Naphthol	Amine	Product	Time (min)	Yield ^b (%)
5				60	88
6				40	93



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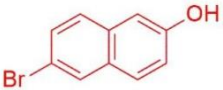
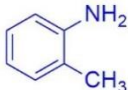
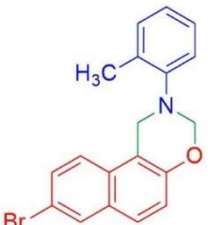
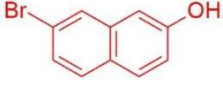
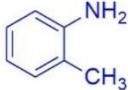

Table 3. Continued.

Sr. No.	Naphthol	Amine	Product	Time (min)	Yield ^b (%)
				30	94
9				30	94
10				30	94

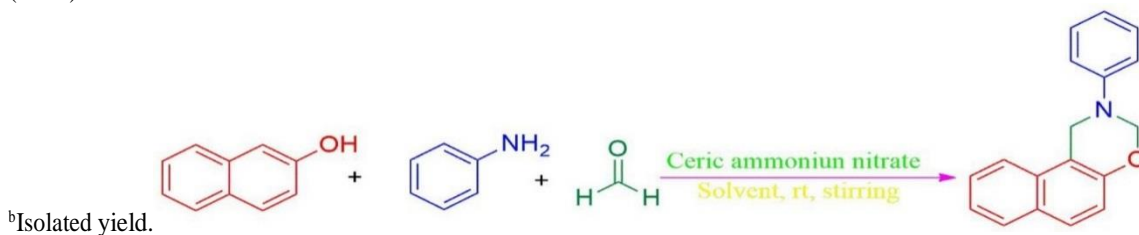
11				30	94
12				48	9

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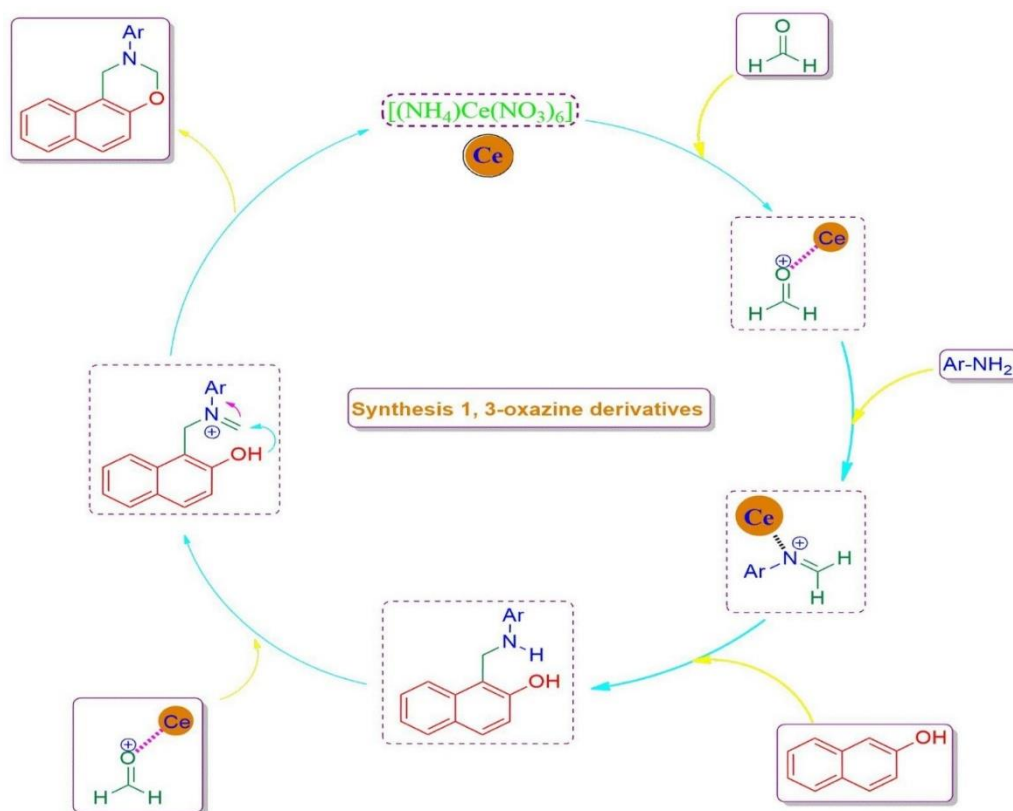
Table 3. Continued.

Sr. No.	Naphthol	Amine	Product	Time (min)	Yield ^b (%)
13				50	88
14				50	88

^aReaction condition: *b*-naphthol (10 mmol), aniline (10 mmol), formaldehyde (20 mmol), and ceric ammonium nitrate (20 mol%) in water (5 mL).



naphthol with three equivalents of formaldehyde to generate the appropriate 1,3-oxazine derivatives in good yields to test the breadth and universality of the current technique (Table 3). Aromatic amines having electron-withdrawing or electron-donating substituents might react effectively to produce the same compounds with little change. The main aromatic amines are of special interest in this study because their pharmacological behavior may be considerably altered by ring substitutions and they can perform nuclear condensation using formaldehyde. The plausible mechanism of the 1, 3-oxazine derivatives catalyzed by ceric ammonium nitrate is depicted in Scheme 2. The reaction will likely proceed *via* ceric ammonium nitrate acting as



Scheme 2. Mechanistic pathway for the synthesis of 1, 3-oxazine synthesis catalyzed by using CAN.

Table 4. Comparative study with the current work with previous literature for the synthesis of 1, 3-oxazine derivatives.

Sr. No.	Reaction condition	Time (min)	Yield (%)	Refs.
1	Montmorillonite K ₁₀ clay	30	89	32
2	Glycerol, 50 °C	5	91	33
3	Thiamin hydrochloride (VB ₁), DMSO, 100 °C	30	92	31
4	PTSA	1h	58	32
5	Acetic acid	1h	45	32
6	LiCl ₂	1h	38	32
7	Ceric Ammonium Nitrate	20	95	This work

Lewis acid, which is attracted toward the formaldehyde and formation of good electrophilic carbon. Then amine substrate gives nucleophilic addition to the obtained imine intermediate. Then, *b*-naphthol was reacted with an iminium ion and another formaldehyde derivative to obtain a member's cyclic ring. A similar type of mechanism is reported in previous work.³¹

When compared to other techniques that have been published recently, the study demonstrates that there are advantages to using a minimum reaction time, aqueous conditions, and avoiding harmful chemicals. Additionally, the catalyst that is produced can easily be recycled from the reaction medium once the desired conversion is achieved. This can be done for at least three cycles without any significant loss in catalytic performance (Table 4).

2. Conclusion

Ceric ammonium nitrate (CAN) is a green, recyclable, cheap, and easily available catalyst for the synthesis of 1, 3-oxazine derivatives from formaldehyde, amine, and *b*-naphthol with stirring at room temperature in aqueous medium. This protocol demonstrates the criteria for a green chemical practice and is extremely efficient and environmentally

friendly. It has mild conditions, high yields, good selectivity, easy isolation, and aqueous condition. We investigated aromatic naphthols and various amine substrate has been studied in the present work.

3. Experimental section

3.1. General procedure for the synthesis of 1, 3-oxazine derivatives

A mixture of *b*-naphthol (10 mmol or 1.44 g), aniline (10 mmol or 0.93 mL), and formaldehyde (20 mmol or 0.60 mL) in ceric ammonium nitrate (20 mol%) was vigorously stirred at 40 °C temperature for the appropriate time (Table 3 Sr. No. 17). The reaction progress was monitored on TLC. After the completion of the reactions, the reaction mixture was extracted with ethyl acetate (10 mL). The organic layer was dried over anhydrous sodium sulfate and concentrated under reduced pressure to give a pure product. The aqueous layer was again used in the next model reaction a minimum of 3 times.

Disclosure statement

No potential conflict of interest was reported by the authors.

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