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Research Article

Green and Efficient One-pot Synthesis of 2-Substituted Quinazolin-4(*3h*)-Ones by Using Scandium Triflate Catalyst

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Abstract

Quinazolin-4(*3H*)-ones derivatives represent an important class of nitrogen-containing heterocycles with diverse biological and pharmacological activities, and their significance makes their efficient synthesis highly desirable. In this context, we report here a simple, efficient and green one-pot synthetic protocol for the preparation of 2-substituted quinazolin-4(*3H*)-ones via cyclocondensation of 2-aminobenzamide with variety aldehydes, employing scandium triflate [$Sc(OTf)_3$] as a water-tolerant and recyclable Lewis acid catalyst. The reactions were carried out at room temperature in dichloromethane, proceeding smoothly without the need for any external oxidant. The transformation involves in situ imine formation followed by intramolecular cyclization and aerobic oxidation. A broad range of aldehydes, including aromatic and heteroaromatic substrates bearing both electron-donating and electron-withdrawing groups, were well tolerated affording the desired products in good to excellent yields (70–90%) within short reaction times (3–5 hours). This green methodology offers several significant advantages, such as mild reaction condition, operational simplicity, high selectivity and reduced environmental impact. A key feature of this methodology lies in the facile recovery of $Sc(OTf)_3$ catalyst through simple aqueous extraction and its recyclability over multiple cycles without significant defeat of efficiency, underscoring its potential in sustainable organic synthesis.

Keywords

One-pot Synthesis, Scandium Triflate, Water-tolerant Lewis Acid, 2- Substituted Quinazolin-4(*3H*)-One Scaffolds, Green Approach

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1. Introduction

Heterocyclic compounds are pivotal organic substances extensively found in medicinal chemistry, owing to their abundant presence in nature and their incorporation into several natural compounds, including vitamins, hormones, and antibiotics. Notably, nitrogen-containing heterocycles like imidazole, indole, pyridine, pyrimidine, triazole, and others hold particular significance in the field of medical sciences. [1] Their biological properties and their capacity to interact with different biological targets are enriched by the occurrence of multiple physiologically active pharmacophores within a single molecular entity [2] quinazolinone derivatives are a preferred class of N-containing heterocyclic compounds because they exhibit a spacious range of therapeutic characteristics, including anticancer, [3-6] antibacterial, [7] antifungal, [8, 9] antimalarial, [10] antiviral, [11] and anti-microbial cholinesterase inhibitors. [12, 13] Quinazolin-4(*3H*)-ones are essential components of many naturally occurring alkaloids as well as a no. of biologically active substances.[14-17] Some of them are summarized in Figure 1.

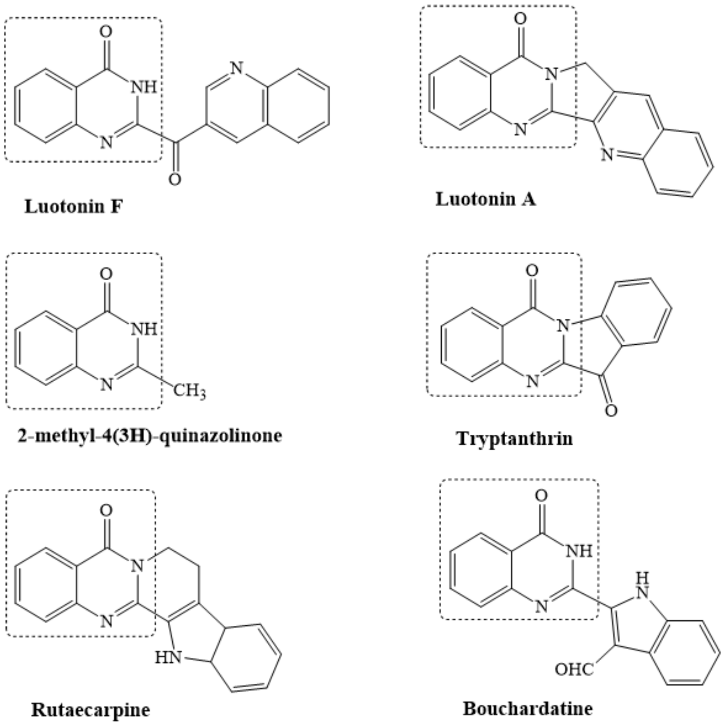


Figure 1. *Bioactive natural products bearing quinazolinone moiety.*

In recent years, focusing on several environmental issues and considering the pharmacological importance of quinazolinone scaffolds there are numerous synthetic methods have been reported. Mostly appealing ways to produce 2-substituted quinazolin-4(*3H*)-one derivatives is through the direct coupling of C-N bonds in metal-catalyzed (like Pd, MnO_2, Ru, Ni, Cu) cascade reactions that involve o-aminobenzamides reacting with benzylic and other alcohols. [18-24] Recently, *Ke Wang et. al.* also reported new methodology for the preparation of 2-substituted quinazolin-4(*3H*)-one derivatives from 2-nitrobenzamide and alcohols in presence of Pd(dppf)Cl_2. [25] Furthermore, the most popular methods start with aldehydes, acyl chlorides, or their analogues and 2-aminobenzoic acid in presence of different metal catalyst like $SbCl_3$, ionic liquid at 120°C, $K_2S_2O_8$ in MeCN to produce the matching structural motifs. [26-32] Among the reported synthetic protocols, several synthetic approach have some limitations, such as use of catalyst which is hazardous in nature, higher temperature with low yield, longer time, harsh reaction conditions, arduous setup, the potential for side reactions, and the production of acidic/metallic wastes. Considering these limitations of reported synthetic methods, there is a requirement for designing appropriate synthetic methods by using new catalyst with higher efficiency and recyclability to be environmentally benign. Finding efficient methodology for the preparation of fused heterocyclic compounds has been the focus of our research work. [33]

In current years, scandium triflate has gained significant awareness as a versatile catalyst for an extensive range oforganic revolution due to its role as water-tolerant, reusable Lewis acid catalysts and low toxicity. [34-37] Extensive research has confirmed that, though lanthanide triflates [$Ln(OTf)_3$] and yttrium triflate are stable in H_2O and can act as Lewis acid catalyst in aqueous media, $Sc(OTf)_3$ has remarkably excellent properties even compared to $Ln(OTf)_3$ in several circumstances. Our research has similarly focused on developing Lewis acid-based, greener productive routes for the synthesizing of different types of fused heterocyclic compounds. [38-40] Taking into consideration the significant applications of scandium triflate, the present work demonstrates its application through the cyclocondensation of 2-aminobenzamide with various aldehydes under ambient, green condition.

2. Experimental Section

2.1. Reagents and Chemicals

All chemicals such as 2-aminobenzamide, aldehydes and scandium triflate were obtained from Merck (India), and all used solvents like ethanol, dioxane, DMF etc. were obtained from Loba Chemie (India) and was used without further purification. ^{1}H and ^{13}C NMR spectra were recorded on 400 MHz & 100 MHz spectrometer instruments, respectively by using TMS as an internal standard and $CDCl_3$, DMSO-d_6 as a solvent. The progress of the reactions was monitored by TLC using silica gel 60 F_{254} pre-coated plates. Melting point were determined using an open capillary tube apparatus and are uncorrected.

2.2. General Procedure for the Synthesis of 2-Substituted Quinazolin-4(*3H*)-One

A combination of the requisite 2-aminobenzamide (1 mmol) and aldehydes (1.1 mmol) in DCM (5 mL) was prepared, to which scandium triflate (5 mol%, 0.02 mmol) catalyst was added, & the resulting mixture was stirred at room temperature. No external oxidant was required, as the reaction proceeds via in situ imine formation follow by intramolecular cyclization and oxidation facilitated by atmospheric oxygen. Upon the reaction completion monitored by Thin Layer Chromatography, the reaction mixture was diluted with water (5 mL) and catalyst was improved from aqueous layer. Final product was extracted with organic layer and the combined organic layer was dried over Na_2SO_4. The crude product was purified by silica gel column chromatography (ethyl acetate/hexane, 15%) to afford the pure 2-substituted quinazolin-4(*3H*)-ones. $Sc(OTf)_3$ was almost quantitatively improved from the aqua layer and can be reused. The melting points of the prepared products were found to be in close concurrence with those found in the literature values, thereby confirming their identity and purity. [22, 27]

2.3. Spectral Data of a Representative 2-Substituted Quinazolin-4(3H)-Ones of the Series

2-Methylquinazolin-4(3H)-one (Table 2, Entry f) [41]

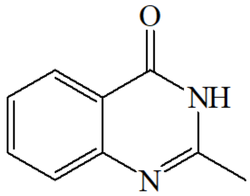
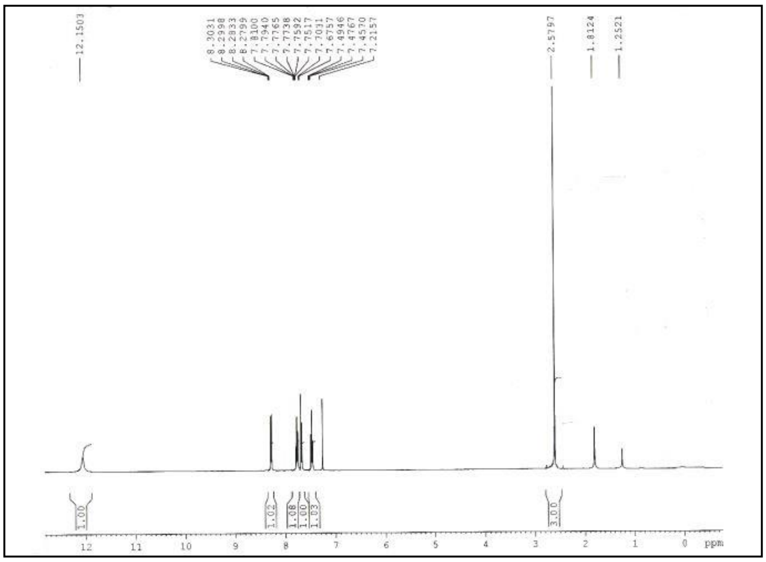
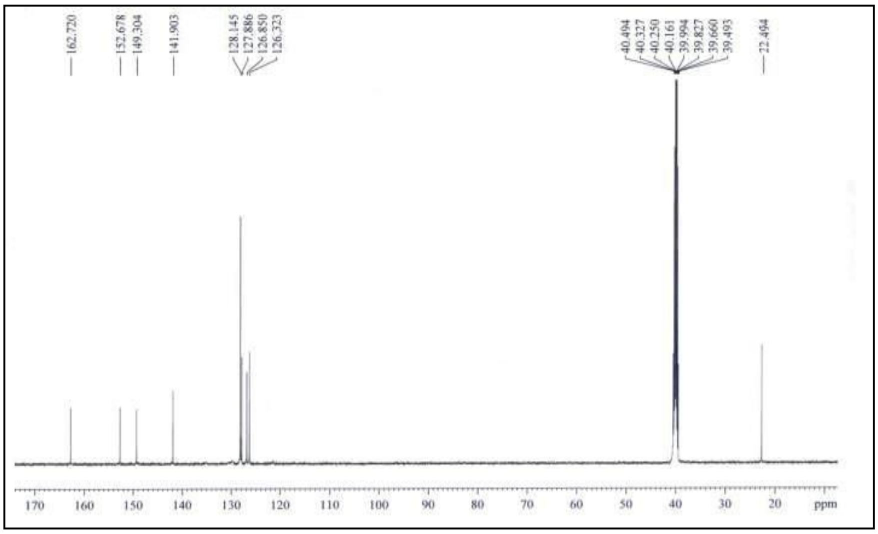


Figure 2. *2-Methylquinazolin-4(3H)-one.*

White solid; yield 72%; mp 240-242; ^{1}H NMR (300 MHz, CDCl3): δ 2.57 (s, 3H), 7.21-7.67 (m, 1H), 7.71 (d, 1H, J=8), 7.75 (t, 1H, J=7.5), 8.30 (d, 1H, J=8), 12.15 (s, 1H); ^{13}C NMR (DMSO-*d6*): δ 22.4, 126.3, 126.8, 127.9, 128.1, 141.9, 149.3, 152.6, 162.7; HRMS (ESI$^+$) calcd. for $C_9H_8N_2O$ $(M+H)^+$: 160.0637; Found 161.0708.





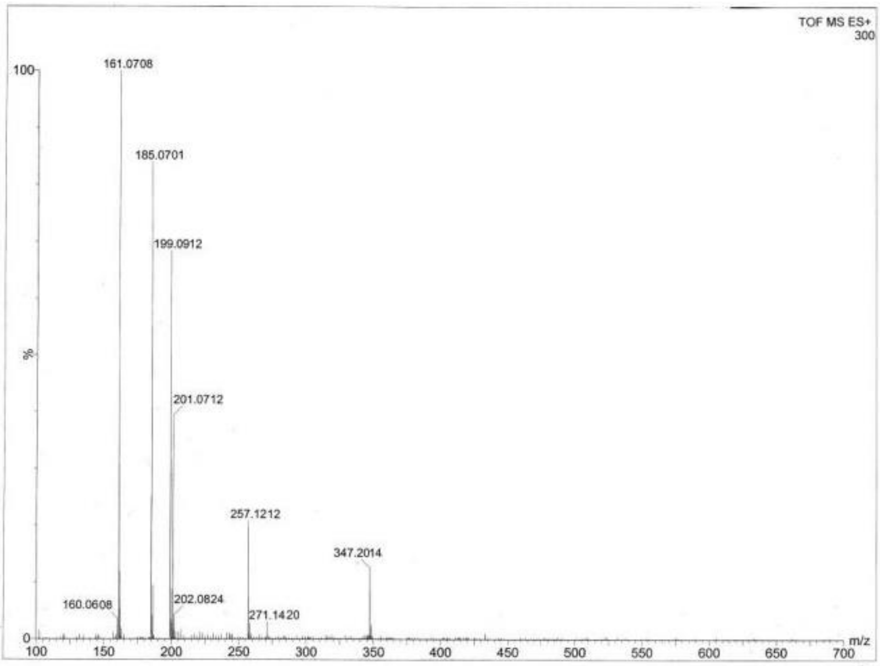


Figure 3. ^{1}H NMR, ^{13}C NMR, and HRMS spectra of 2-Methylquinazolin-4(3H)-one.

2-(4-Chlorophenyl)quinazolin-4(3H)-one (Table 2, Entry d) [22, 27]

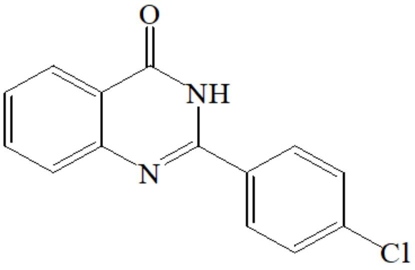
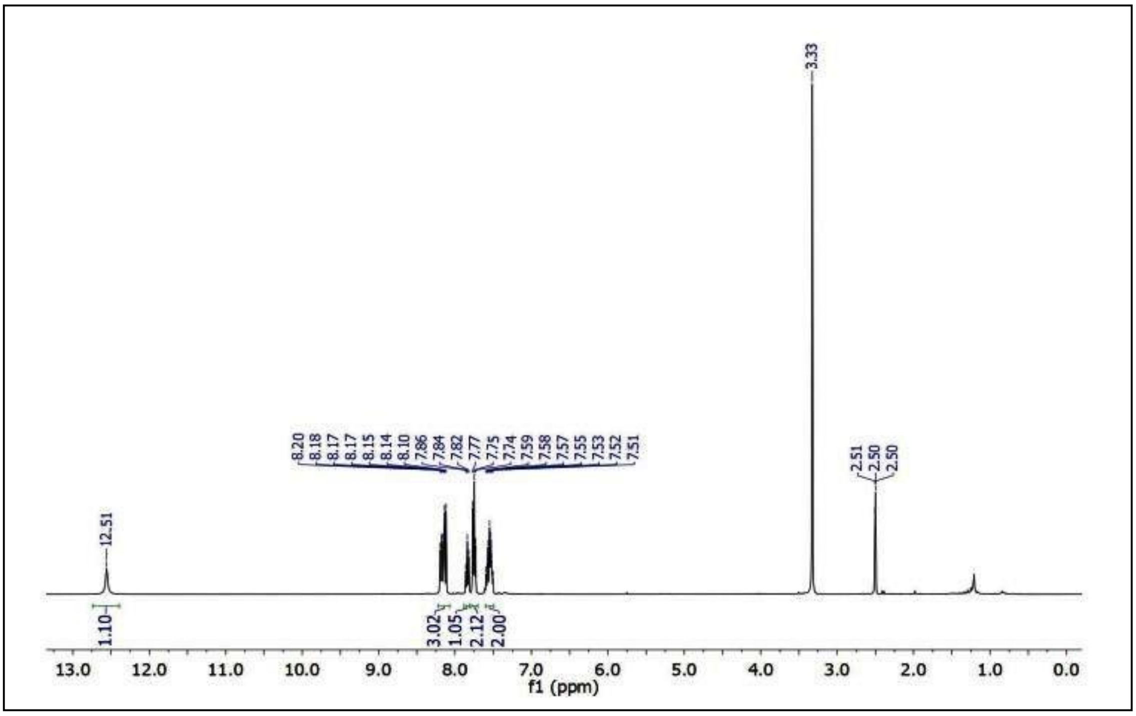
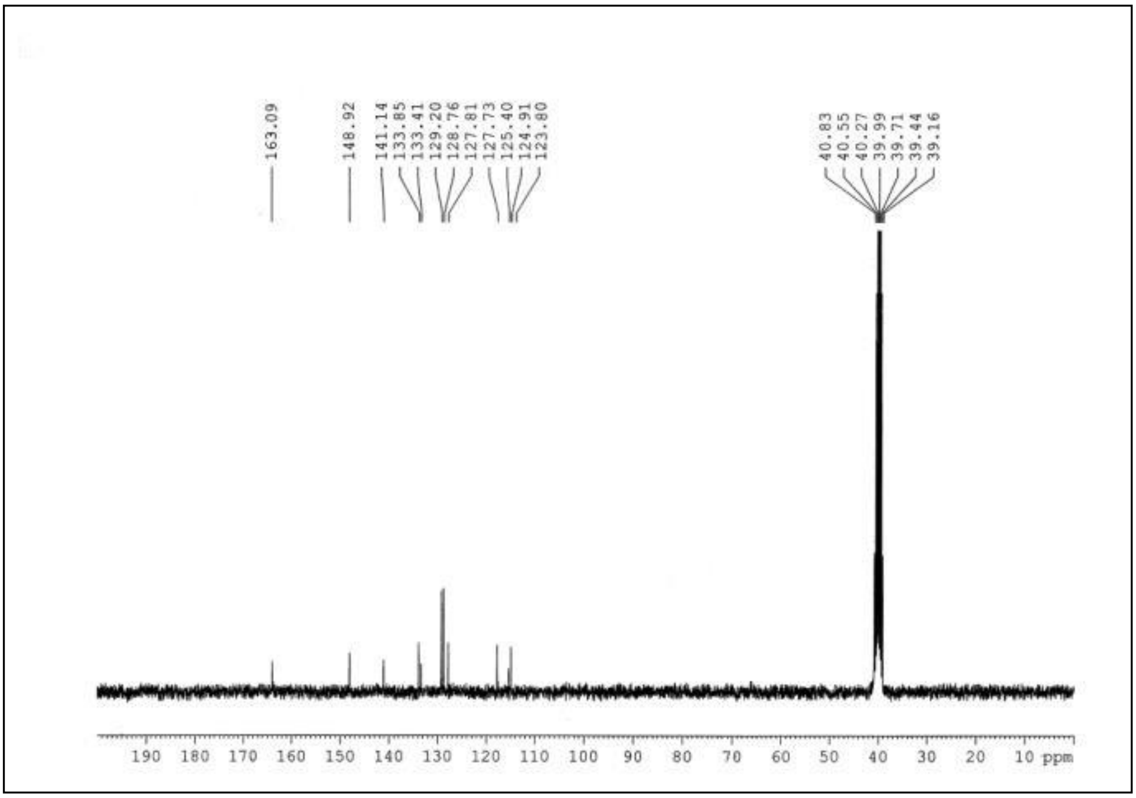


Figure 4. 2-(4-Chlorophenyl)quinazolin-4(3H)-one

White solid; yield 86%; mp 298-300; ^{1}H NMR (400 MHz, DMSO-d6): δ 12.51 (s, 1H), 8.10−8.20 (m, 3H), 7.86 (t, J =8 Hz, 1H), 7.74− 7.77 (m, 2H), 7.51−7.60 (m, 2H); ^{13}C NMR (100 MHz, DMSO-d6): δ 123.80,124.91,125.40,127.73, 127.81, 128.76, 129.20, 133.41, 133.85, 141.14, 148.92, 163.09; HRMS (ESI$^+$) calcd. for $C_{14}H_{10}ClN_2O$ (M+H)$^+$: 257.0437; Found 254.0578.





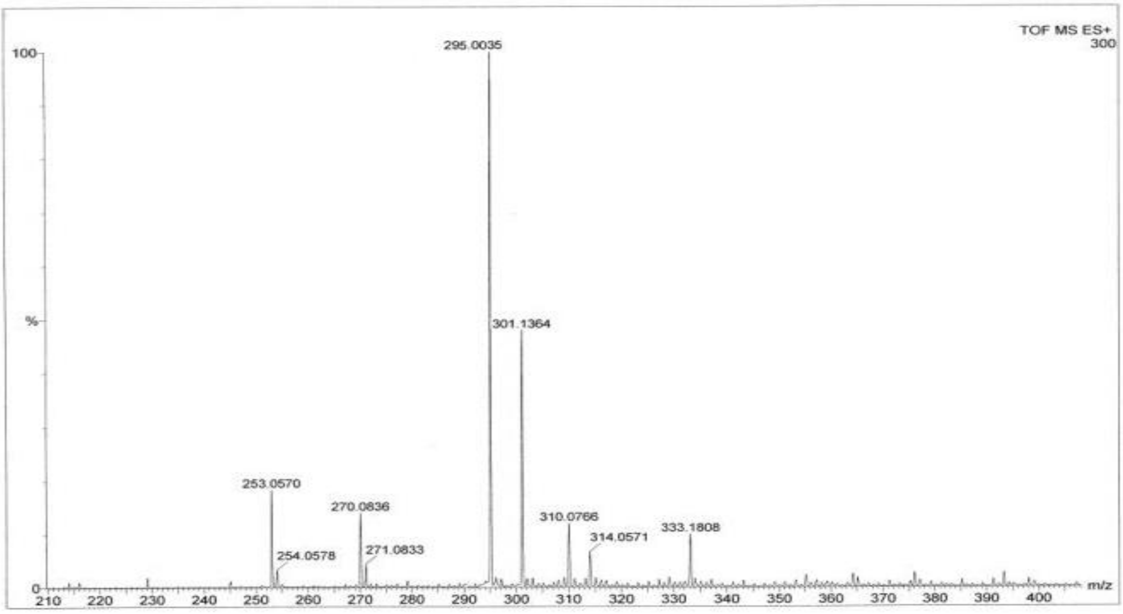


Figure 5. *^{1}H NMR, ^{13}C NMR, and HRMS spectra of 2-(4-Chlorophenyl)quinazolin-4(3H)-one*

2-(4-Nitrophenyl) quinazolin-4(3H)-one (Table 2, Entry b)[22, 27]

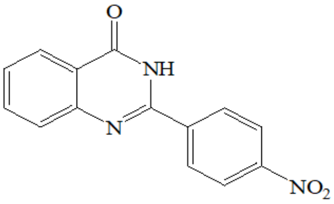
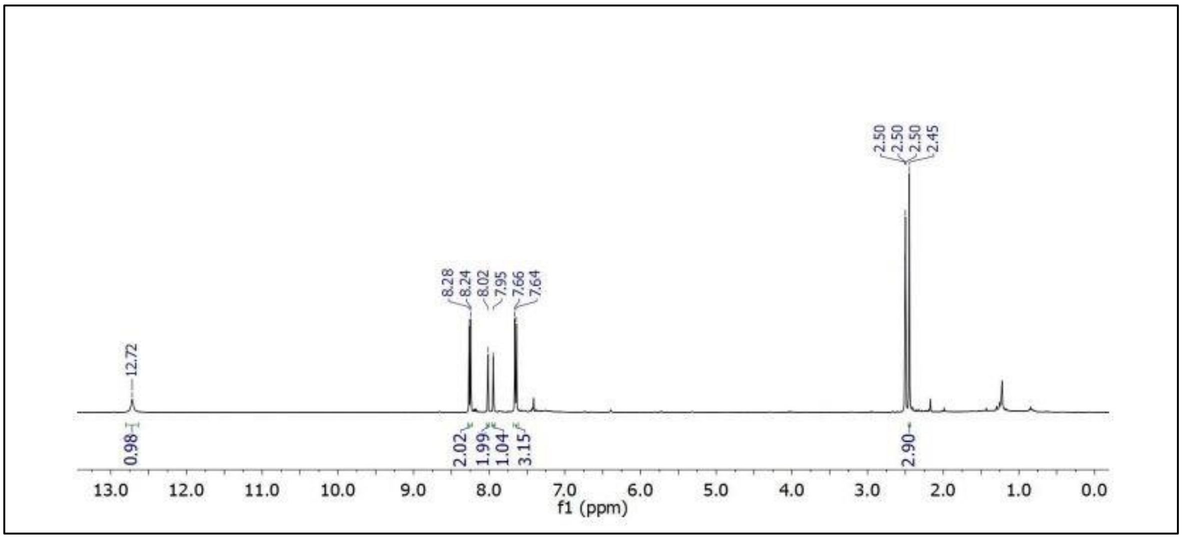
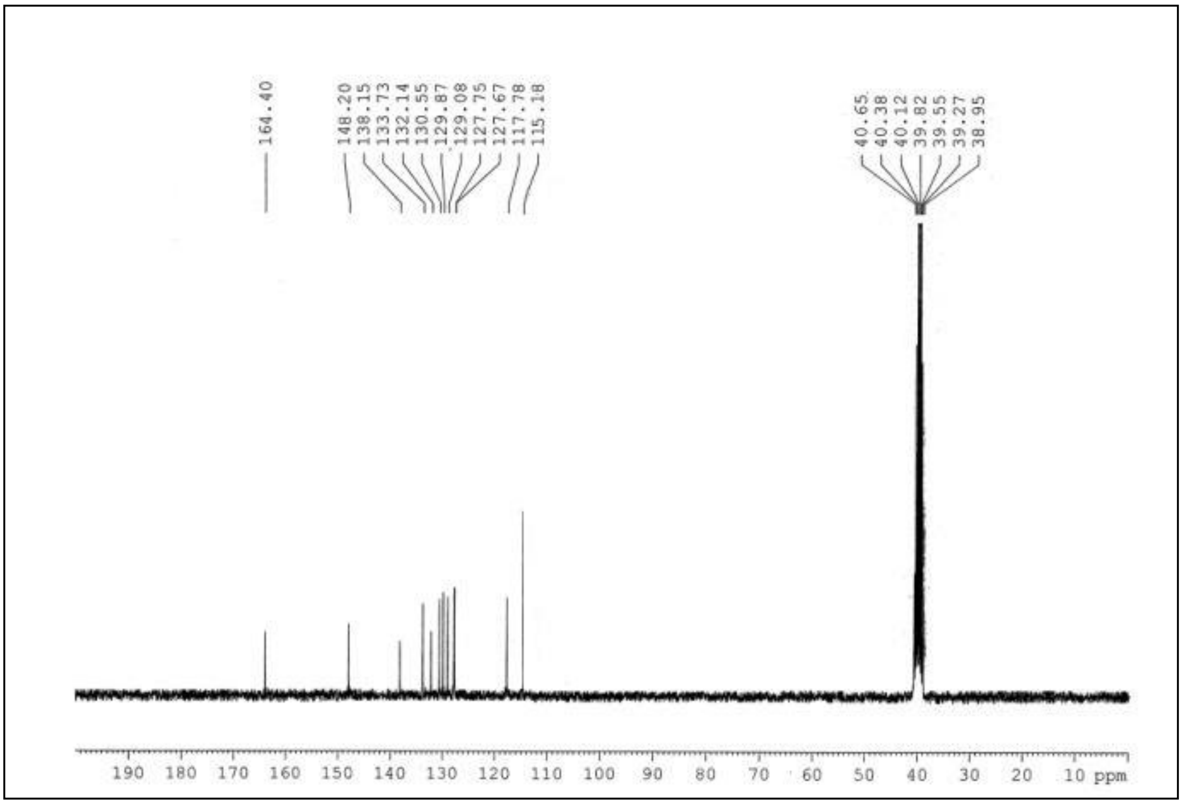


Figure 6. *2-(4-Nitrophenyl) quinazolin-4(3H)-one.*

Brown solid; yield 90%; mp 295-297; ^{1}H NMR (DMSO-d6): δ 7.64-7.66 (m, 3H), 7.95-8.02 (m, 3H), 8.24-8.28 (m, 2H), 12.7 (s,1H); ^{13}C NMR (DMSO-d6): δ 115.1, 117.7, 127.6, 127.7, 129.0, 129.8, 130.5, 132.1, 133.7, 138.1, 148.2, 164.4; HRMS (ESI$^+$) calcd. for $C_{14}H_9N_3O_3$ (M+H)$^+$: 267.0644; Found 266.9910.





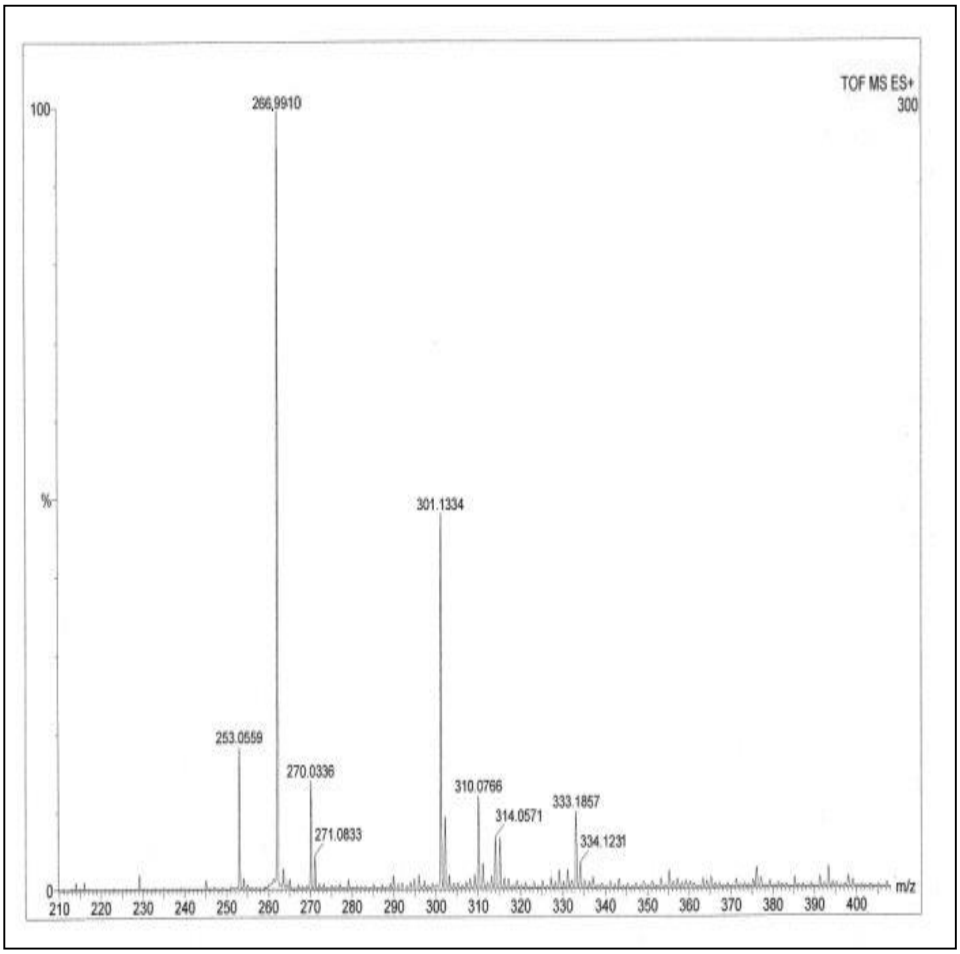


Figure 7. ^{1}H NMR, ^{13}C NMR, and HRMS spectra of 2-(4-Nitrophenyl) quinazolin-4(3H)-one.

3. Result and Discussion

The Preparation of quinazolin-4(*3H*)-ones derivatives was carried out by cyclocondensation of 2-aminobenzamide with various aldehydes in the existence of scandium triflate $Sc(OTf)_3$ in dichloromethane (DCM). The reaction mass was stirred at RT for 3-5 hrs afforded quinazolin-4(*3H*)-one derivatives (2a–k) with good to excellent yield. This reaction is outlined in Figure 8.

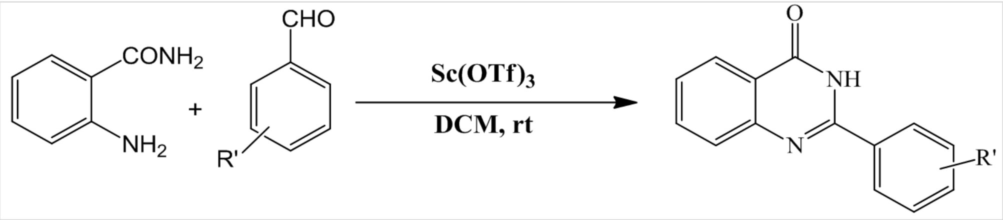


Figure 8. *Synthesis of quinazolin-4(3H)-one derivatives (2a-k) using $Sc(OTf)_3$ catalyst.*

Initially, for optimization the reaction conditions with respect to solvents and catalysts; scandium triflate catalyzed reaction between2-aminobenzamide (1mmol) and benzaldehyde (1.1mmol) was selected as a model reaction under different concentrations of catalyst and solvents, results are summarized in Table 1. It was observed that scandium triflate (5 mol%) was found to be a perfect for reaction in DCM at RT compared to other solvents such as ethanol, ethanol-water, CH3CN, dioxane or toluene (Table 1, entry e). Higher catalyst loading (10 mol%) showed no improvement and even slightly reduced yield, likely due to competitive side reactions. In this protocol, the solvent (DCM) modulates the Lewis acidity and coordination behavior of $Sc(OTf)_3$. In nonpolar media such as DCM, the catalyst remains in a highly active, weakly solvated form. This enhances its electrophilic coordination to the carbonyl oxygen of the aldehyde, thereby accelerating imine formation and the subsequent intramolecular cyclization.

Moreover, the immiscibility of DCM with water allows convenient separation and recovery of $Sc(OTf)_3$ after the reaction, thereby enhancing both catalytic efficiency and recyclability. Hence, the mild and solvent-controlled activation by $Sc(OTf)_3$ distinctly differentiate this method from last protocols that relied on harsher oxidizing or dehydrating agents.

During optimization it is further observed that solvent effects were significant: ethanol or ethanol-water is also comparatively good solvent media for said transformation but catalyst, reactant and formed product are soluble in ethanol-water and it gives homogenous reaction mixture, hence it is difficult to separate the catalyst and product. DCM, by contrast, allowed easy separation of catalyst and product post-reaction due to its immiscibility with water.

Table 1. *Optimization of reaction condition for the preparation of 2-phenylquinazolin-4(3H)-one*^{a}*.*

Entry	**Catalyst**	**Solvents**	**Timeb (h)**	**Yieldc(%)**
a	No catalyst	EtOH	10	20
b	$Sm(OTf)_3$ (5 mol%)	EtOH	5	75
c	$Sc(OTf)_3$ (5 mol%)	$EtOH/H_2O$	3	82
d	$Sc(OTf)_3$ (5 mol%)	Toluene	4	60
e	$Sc(OTf)_3$ (5 mol%)	DCM	3	88
f	$Sc(OTf)_3$ (10 mol%)	DCM	3	85
g	$Sc(OTf)_3$ (5 mol%)	Dioxane	3	75
h	$Sc(OTf)_3$ (5 mol%)	CH_3CN	4	60

(a-Stirring at rt, b-Time required for completion of reaction, c-Isolated yield)

With optimized conditions, a range of aromatic, aliphatic, and heteroaromatic aldehyde were treated with 2-aminobenzamide to afford corresponding quinazolin-4(*3H*)-ones (2a–k). The method displayed broad substrate tolerance, including electron-donating and electron-withdrawing groups. Mostly the reactions proceeded very smoothly; aldehydes containing a variety of sensitive functional groups were good enough under these condition.

As a general trend, electron-deficient aldehydes such as 4-nitrobenzaldehyde gave the better yield in shorter duration as compared to electron-rich aldehydes. Products were obtained in high to excellent yields (70–90%), with excellent regioselectivity and purity. The obtained results are summarized in Table 2.

Table 2. *Sc(OTf)$_3$ catalyzed synthesis of 2-substituted quinazolin-4(3H)-one from aldehydes*^{a}.

Entry	Products	Yieldb(%)	Timec(h)
a	2-Phenyl quinazolin-4(3H)-one	88	3
b	2-(4-Nitrophenyl) quinazolin-4(3H)-one	90	3
c	2-(4-Methylphenyl) quinazolin-4(3H)-one	85	5
d	2-(4-Chlorophenyl) quinazolin-4(3H)-one	86	4
e	2-(4-Methoxyphenyl)quinazolin-4(3H)-one	78	5
f	2-Methyl quinazolin-4(3H)-one	72	5
g	2-Isopropyl quinazolin-4(3H)-one	74	4
h	2-(Thiophen-2-yl)quinazolin-4(3H)-one	82	5
i	2-(4-Fluorophenyl) quinazolin-4(3H)-one	85	4
j	2-(4-Hydroxyphenyl) quinazolin-4(3H)-one	70	5
k	2-(2-Chlorophenyl) quinazolin-4(3H)-one	80	4

(a-Reaction conditions: aldehydes (1.1mmol), 2-aminobenzamide (1 mmol), Sc(OTf)$_3$(5 mol%), DCM (5 mL), stirring at rt, b-Isolated yield, c-Time required for completion of reaction.)

After completion of the reaction, the catalyst separated by adding water (5mL) in reaction mixture. Scandium triflate is perfectly soluble in H_2O than that in organic solvent; hence product was extracted with DCM. The catalyst was improved almost quantitatively from the aqueous layer and reused. The recovered catalyst was used for second reaction, and it was experimental that the recovered catalyst retained nearly the same catalytic efficiency as that of fresh catalyst shown in Figure 9.

Sc(OTf)$_3$ was readily recovered by aqueous extraction after each post-reaction, dried and notably, reused for at least four consecutive cycle with only a minor decline in yield (Table 3). These findings highlight the superior recyclability and sustainability of Sc(OTf)$_3$ over conventional non-recyclable Lewis acids, offering a sustainable alternative to traditional acidic catalysts. Owing to its distinctive characteristics, utilization of scandium triflate in chemical processes not only mitigates environmental issue allied with conventional Lewis acids but also enhances the overall sustainability of the method and aligns well with green chemistry principle.

Figure 9. *Synthesis of 2-phenyl quinazolin-4(3H)-one using 2-aminobenzamide and benzaldehyde in presence of Sc(OTf)$_3$.*

Table 3. *Catalyst reusability study for the reaction of benzaldehyde with 2-aminobenzamide*[a].

Entry	Yieldb (%)	Catalyst recovery (%)
a	88	95
b	86	92
c	83	90
d	80	88

(a- Reaction condition: benzaldehyde (1.1mmol), 2-aminobenzamide (1 mmol), $Sc(OTf)_3$ (5 mol%), DCM (5 mL), stirring at rt, b-Isolated yield.)

4. Conclusion

We have developed a green route of one-pot the synthesis of 2-substituted quinazolin-4(*3H*)-ones derivatives by the cyclocondensation reactions of 2-aminobenzamide with various aldehydes by using scandium triflate as a water-tolerant Lewis acid catalyst. The present methodology offers substantial benefit over predictable approach in terms of mild condition, broad functional group tolerance, high yields in short duration and excellent catalyst recyclability. Overall, ambient-temperature, additive oxidant-free operation combined with environmental compatibility and excellent reusability of the catalyst establishes present approach as a distinct sustainable and efficient alternative over traditional metal- or Lewis acid-catalyzed systems for the green synthesis of biologically relevant quinazolinone derivatives.

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Author Contributions

Amarsinha Babasaheb Gorepatil: Conceptualization, Data curation, Formal Analysis, Methodology, Investigation, Writing – original draft

Prashant Ganpatrao Kumdale: Conceptualization, Methodology, Investigation, Writing – review & editing

Akshay Balasaheb Ghumare: Investigation, Formal Analysis

Krishna Vitthal Kele: Investigation, Formal Analysis

Mahadev Vilas Gaikwad: Investigation, Formal Analysis

Pratapsinha Babasaheb Gorepatil: Supervision, Formal Analysis, Project administration

Conflicts of Interest

The authors declare no conflicts of interest.

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