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Efficient aqueous Suzuki coupling at room temperature via micellar catalysis with unconventional surfactants

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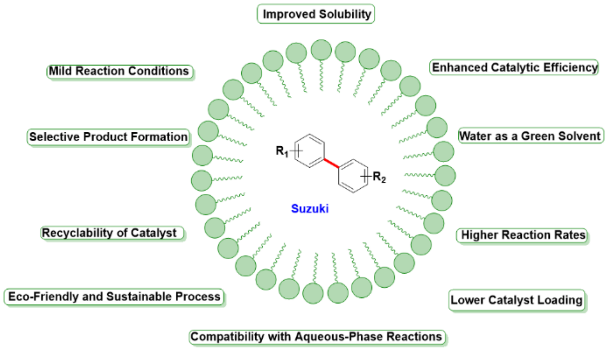
Abstract

A green and efficient Suzuki cross-coupling method using micellar catalysis in water at room temperature has been developed. Eco-friendly surfactants like sodium palmitate and sodium linoleate replace harmful organic solvents. These surfactants improve reaction efficiency, selectivity, and recyclability. Tailored surfactant systems enhance micellar catalysis for industrial use. The study reviews recent advances and mechanistic benefits of surfactant-based systems in aqueous catalytic reactions.



Keywords: Green Chemistry, Micellar catalysis, Unconventional surfactants, Sodium palmitate, Sodium linoleate, Suzuki reaction

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Introduction

The Suzuki cross-coupling reaction is crucial in synthesizing pharmaceuticals and agrochemicals such as Valsartan [1], Boscalid [2], LSZ102 [3], and various substituted biphenyls, [4] Traditional catalytic systems use expensive, air-sensitive palladium species with phosphine ligands and organic solvents, raising environmental and economic concerns [4]. Bruce H. Lipshutz pioneered the use of designer surfactants for aqueous catalysis, leading to the development of TPGS-750M from DL-α-tocopherol alkoxy polyethylene glycol [5]. This study explores micellar catalytic systems employing alternative surfactants like sodium palmitate and sodium linoleate [Figure 1] for Suzuki reactions in water. These fatty acid salts, common in soaps and detergents, [6] offer a sustainable, cost-effective solution for greener cross-coupling chemistry.

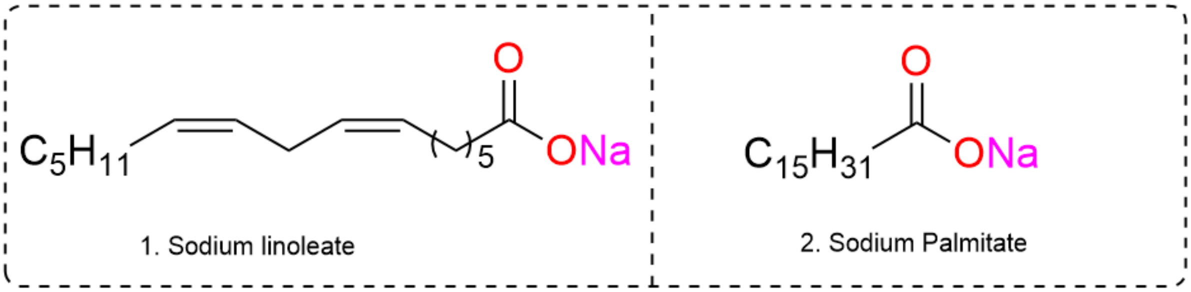


Figure 1. Chemical structure of the unconventional surfactants utilized for micellar catalysis in aqueous media at room temperature.

Conventional surfactants like TPGS-750M, PTS, Tween 80,[7] and PS-750M [8] are widely used in micellar catalysis, but their high-cost limits large-scale application. To overcome this, we developed a cost-effective, efficient, and recyclable system using unconventional surfactants under mild aqueous conditions—advancing green chemistry.[9] Sodium palmitate and sodium linoleate, anionic surfactants with hydrophilic carboxylate heads and hydrophobic tails, enhance Suzuki cross-coupling reactions with various palladium catalysts.[10] Traditionally performed in organic solvents at elevated temperatures, Suzuki reactions benefit from aqueous media, which improve yields and reduce environmental impact [11]

Despite limited exploration, sodium palmitate and sodium linoleate show promise in micellar catalysis. This work introduces them to boost efficiency and selectivity under mild conditions (room temperature to 40 °C), using minimal palladium (ppm levels) and 1 :1 boronate-to-halide ratios, supporting scalable and one-pot multistep syntheses with improved yields. [12] Recent studies explored alternative surfactants like saponin for heteroaromatics, [13] Brij-30 for reactivity analysis, [14] and commercial products like Evonik Noblyst for low-catalyst aqueous reactions. [15] Palladium nanoparticles in lauryl methacrylate micelles [16] and nickel sulfate with bipyridyl [17] have also enabled green Suzuki couplings with high efficiency. [18]

Role of Surfactants in Pd-Catalysed Suzuki Cross-Coupling Reactions

Surfactants are pivotal in Pd-catalysed Suzuki cross-coupling reactions, especially in aqueous and micellar systems, by enhancing efficiency, catalyst stability, and sustainability. They solubilize poorly water-soluble reactants like boronic acids, esters, and Pd catalysts by forming micelles with hydrophobic cores, boosting reactivity. Surfactants also stabilize Pd species, preventing aggregation and deactivation, and modulate their electronic and steric properties, thereby improving activity and selectivity. Micellar systems facilitate mass transfer by increasing local reactant concentrations and enabling effective co-localization of hydrophobic substrates and hydrophilic catalysts at micelle interfaces. This accelerates reaction rates. Moreover, surfactant-mediated systems promote green chemistry by allowing water as a solvent, reducing reliance on hazardous organic solvents, and enhancing sustainability. Surfactants influence catalytic microenvironments, aiding in the control of Pd oxidation states, ligand coordination, and product stabilization, which improves regio- and chemo selectivity. Sodium linoleate and sodium palmitate provide micellar environments with added green benefits, enhanced solubility, and catalyst-stabilizing effects, positioning them as promising alternatives to traditional surfactants in sustainable synthesis. Although underexplored, their application in micellar catalysis offers a novel and effective approach to improving reaction performance. This method supports scalable transformations (from milligram to multigram) valuable in therapeutic and green chemistry contexts. The reactions proceed under mild conditions (room temperature to 40 °C), with 1:1 boronate-to-halide ratios, minimal Pd loadings (ppm range), and compatibility with one-pot multistep syntheses, yielding superior outcomes compared to conventional solvents. [19]

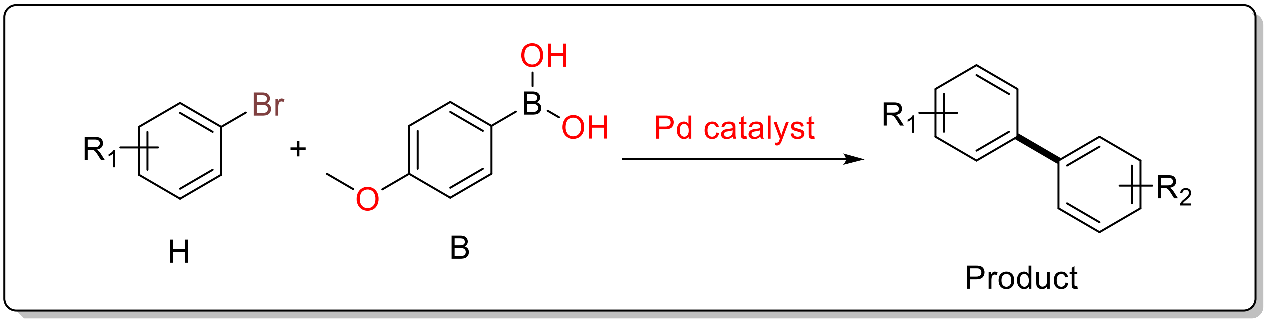
Recent studies have employed saponin as an surfactant for Suzuki cross-coupling, especially with heteroaromatic substrates in aqueous media at room temperature, offering a green, high-yielding method. [20] Substituent effects in micellar Suzuki reactions have been studied using Hammett plots and kinetics, showing enhanced reactivity with 2 wt. % Brij-30 in water. [21] Commercial surfactants like Evonik Noblyst have enabled efficient aqueous-phase couplings of aryl and heteroaryl compounds using reduced Pd catalyst loadings. [22] Additionally, Pd nanoparticles stabilized by lauryl methacrylate-based micelles have proven effective in coupling aromatic bromides with boronic acids in water [23, 24] A green Suzuki protocol using nickel sulfate hexahydrate and 2,2′-bipyridyl in aqueous media has also enabled the synthesis of diverse aryl and allyl phosphonates and sulfones with high yields [25]

Key advantages of our method include catalyst and surfactant recyclability, high surfactant stability, reduced organic waste, and simplified procedures—offering a cost-effective, environmentally friendly solution. The use of unconventional surfactants exemplifies the progress of green chemistry in pharmaceutical synthesis by combining energy efficiency, cost savings, and eco-friendly, non-toxic, non-flammable solvents. Sodium linoleate and sodium palmitate form effective micellar environments, improve solubility, stabilize catalysts, and support green chemistry goals. Their application as unconventional surfactants offer a sustainable alternative to traditional solvents and surfactants. In conclusion, integrating surfactants into Pd-catalysed Suzuki reactions enhances catalyst stability, solubility, kinetics, and selectivity, supporting scalable and environmentally benign synthetic methodologies. The ongoing development of surfactant-assisted catalysis holds strong potential for advancing sustainable organic synthesis.

Results and Discussion

To evaluate the efficiency of the Suzuki cross-coupling reaction, we explored a diverse range of boronated compounds, including boronic acids, boronated esters, and potassium boronate salts, each featuring various aromatic and aliphatic groups. These substrates encompass electron-donating, electron-withdrawing, and neutral substituted aromatic groups, as well as aliphatic chains and cyclic aliphatic boron compounds. Additionally, we tested halogenated compounds with similar structural diversity, along with heterocyclic and multi-substituted substrates bearing functional groups such as p-OMe, m-OMe, m-Cl, m-NO_2, p-CN, p-COOEt, -CO-Me, lactone, N-Boc, and m-CF_3

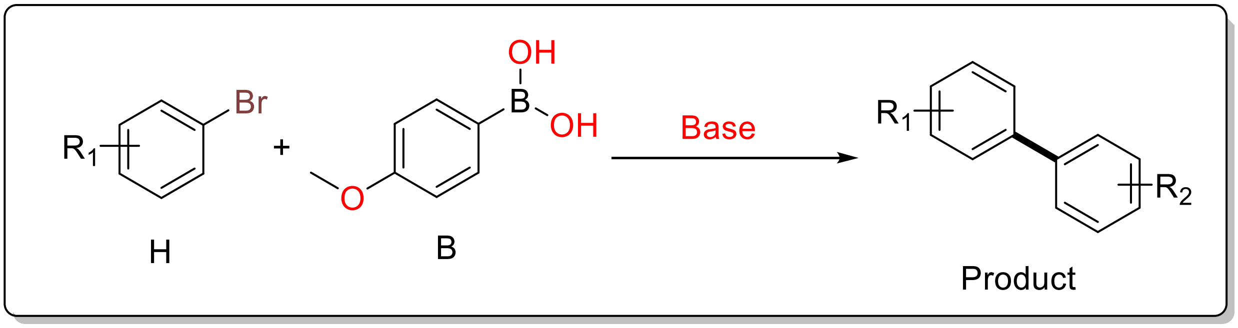
Table 1. Optimization of reaction condition with different palladium catalyst



Entry	Catalyst	Halogen (H)	*Conversion (%)
		Catalyst optimization	
1	Pd $(OAc)_2$	2-bromonaphthalene	91.32
2	$PdCl_2$(dppf)	2-bromonaphthalene	97.27
3	$PdCl_2$(dppf). DCM complex	2-bromonaphthalene	97.18
4	Pd2$(dba)_3$	2-bromonaphthalene	97.01
5	Pd(btbpf)	2-bromonaphthalene	96.90
6	Pd $(OAc)_2$	1-bromo-4-methoxybenzene	97.54
7	$PdCl_2$(dppf)	1-bromo-4-methoxybenzene	96.76
8	$PdCl_2$(dppf). DCM complex	1-bromo-4-methoxybenzene	98.31
9	Pd2$(dba)_3$	1-bromo-4-methoxybenzene	97.54
10	Pd(btbpf)	1-bromo-4-methoxybenzene	97.34
11	Pd $(OAc)_2$	1-bromo-4-nitrobenzene	95.10
12	$PdCl_2$(dppf)	1-bromo-4-nitrobenzene	98.98
13	$PdCl_2$(dppf). DCM complex	1-bromo-4-nitrobenzene	97.20
14	Pd2$(dba)_3$	1-bromo-4-nitrobenzene	94.10
15	Pd(btbpf)	1-bromo-4-nitrobenzene	99.03

Reaction conditions **:** Halogen **(**H) (1.0 mmol), Boronate (B) (1.5 mmol), Pd (0.05 mmol) and surfactant (20 volumes) in water (10 volumes), base (3.0 mmol), Water, rt, 2h.

Table 2. Optimization of reaction condition with different organic and inorganic bases



Entry	Bases	Halogen (H)	*Conversion (%)
		Base optimization	
1	TEA	2-bromonaphthalene	99.93
2	DIPEA	2-bromonaphthalene	95.63
3	K_2CO_3	2-bromonaphthalene	86.59
4	Na_2CO_3	2-bromonaphthalene	83.52
5	K_3PO_4	2-bromonaphthalene	84.68
6	KTB	2-bromonaphthalene	89.55
7	TEA	1-bromo-4-methoxybenzene	99.90
8	DIPEA	1-bromo-4-methoxybenzene	98.49
9	K_2CO_3	1-bromo-4-methoxybenzene	98.12
10	Na_2CO_3	1-bromo-4-methoxybenzene	97.97
11	K_3PO_4	1-bromo-4-methoxybenzene	83.61
12	KTB	1-bromo-4-methoxybenzene	97.92
13	TEA	1-bromo-4-nitrobenzene	99
14	DIPEA	1-bromo-4-nitrobenzene	96.67
15	K_2CO_3	1-bromo-4-nitrobenzene	98.37
16	Na_2CO_3	1-bromo-4-nitrobenzene	97.90
17	K_3PO_4	1-bromo-4-nitrobenzene	98.47
18	KTB	1-bromo-4-nitrobenzene	99

Reaction Conditions : Halogen (H) (1.0 mmol), Boronate (B) (1.5 mmol), Pd (0.05 mmol), and surfactant (20 volumes) in water (10 volumes), base (3.0 mmol) ; reaction conducted in water at room temperature for 2 hours.

Catalyst Screening : A range of palladium catalysts, including Pd $(OAc)_2$, $PdCl_2$(dppf), $Pd_2(dba)_3$, and Pd(dtbpf), were evaluated under comparable reaction conditions [Table 1, entries 1–15]. All catalysts exhibited good reactivity, with $PdCl_2$(dppf) emerging as the most effective.

Base Screening : Various organic and inorganic bases, including Triethylamine (TEA), N, N-Diisopropylethylamine (DIPEA), potassium carbonate (K_2CO_3), caesium carbonate (Cs_2CO_3), sodium carbonate (Na_2CO_3), and potassium phosphate (K_3PO_4), were assessed for their influence on reaction efficiency. While all bases facilitated good conversion, TEA provided the highest yield and was subsequently employed for the preparation of all 67 target compounds.

Optimization of Reaction Conditions : The optimized reaction conditions were established using 0.5 mol% $PdCl_2$(dppf)·DCM complex and 3 equivalents of TEA, with sodium palmitate or sodium linoleate (20 volumes) serving as surfactants. The reaction proceeded efficiently at room temperature within 2 hours. Following extensive parameter screening, the optimized conditions were applied to the Suzuki coupling of various aryl boronic acids with halogenated compounds, primarily bromo derivatives, featuring diverse electronic effects.

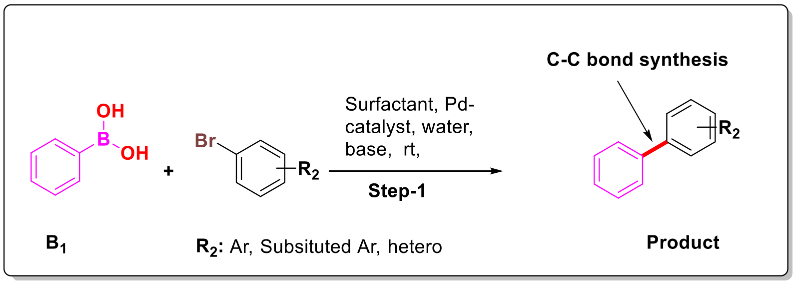
Scope and Functional Group Tolerance: The optimized protocol was successfully utilized for the synthesis of 67 compounds from commercially available aryl bromides and a range of aryl and heteroaryl boronic acids. The reaction demonstrated excellent efficiency across different electronic environments, accommodating both electron-donating and electron-withdrawing substituents at the ortho-, meta-, and para-positions of aryl bromides. These substrates underwent efficient Suzuki coupling, affording moderate to high yields.

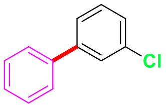
Optimization of the Suzuki Coupling Reaction and Model Substrate Selection: To optimize the reaction conditions, 4-methoxyphenylboronic acid (1.0 mmol) was selected as the model boronic acid substrate for screening.
Reaction Conditions : Halide Partner : 1-Bromo-4-methoxybenzene (1.1 mmol), Catalyst : Pd(dppf)Cl_2·DCM (0.05 mmol), Base : Triethylamine (TEA, 3 mmol), Surfactant : Sodium palmitate (4% in water, 20 volumes), Solvent : Aqueous medium, Temperature: Room temperature (rt), Reaction Time : 2 hours. The reaction was conducted in an aqueous medium containing a surfactant to enhance solubility and promote efficient coupling. This setup was designed to optimize the Suzuki coupling conditions across various substrates, allowing for an assessment of the impact of different electronic environments on reaction efficiency and yield.

Reaction Performance and Yield: The optimized reaction conditions yielded the isolated compound with a good isolated yield [Table 3-11]. This high conversion rate indicates efficient transformation of the starting materials into the desired product, with minimal side reactions.

Advantages of the Developed Method : The proposed methodology offers several key advantages: Cost and energy efficiency: Reduction in overall reagent and energy consumption. Sustainable reaction conditions : Use of aqueous media and surfactants minimizes environmental impact. Minimal catalyst loading: Efficient use of palladium catalysts reduces costs and waste. High isolated yields: Excellent product recovery and purity. Scalability: Demonstrated reproducibility from milligram to multigram scales. Streamlined purification : Simplified isolation procedures with minimal byproducts. Reduced waste generation: Eco-friendly approach aligned with green chemistry principles. Safer reaction conditions : Mild, room-temperature operation enhances safety and operational convenience. In summary, this study successfully establishes an optimized Suzuki coupling protocol with superior yield, high efficiency, and significant environmental and economic benefits. The findings highlight the reaction’s versatility, making it a valuable approach for the synthesis of structurally diverse molecules.

Table 3. Suzuki cross coupling reaction with Phenyl Boronic acid

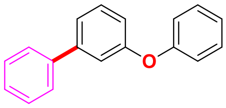




1

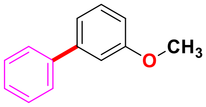
*

A) With Sodium Palmitate: 89%
B) With Sodium Linoleate: 79%
C) With TPGS=750-M: 88%
D) With Coventional heating: 61%



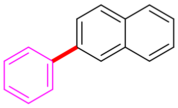
2

A) With Sodium Palmitate: 91%
B) With Sodium Linoleate: 92%
C) With TPGS=750-M: 89%
D) With Convetional heating: 65%



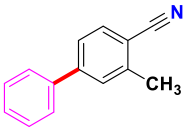
3

A) With Sodium Palmitate:95%
B) With Sodium Linoleate: 91%
C) With TPGS=750-M: 82%
D) With Convetional heating: 75%



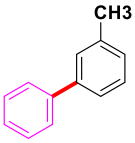
13

A) With Sodium Palmitate: 95%
B) With Sodium Linoleate: 93%
C) With TPGS 750M: 88%
D) With Convetional heating: 82%



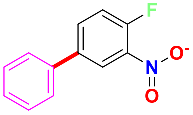
25

A) With Sodium Palmitate:82%
B) With Sodium Linoleate: 82%
C) With TPGS=750-M: 80%
D) With Convetional heating: 65%



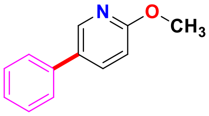
26

A) With Sodium Palmitate:82%
B) With Sodium Linoleate: 85%
C) With TPGS=750-M: 81%
D) With Convetional heating: 66%



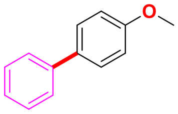
27

A) With Sodium Palmitate:80%
B) With Sodium Linoleate: 85%
C) With TPGS=750-M: 80%
D) With Convetional heating: 61%



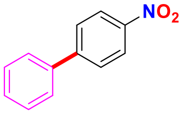
28

A) With Sodium Palmitate:93%
B) With Sodium Linoleate: 92%
C) With TPGS=750-M: 90%
D) With Convetional heating: 71%



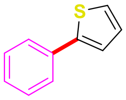
51

A) With Sodium Palmitate: 91%
B) With Sodium Linoleate: 95%
C) With TPGS=750-M: 88%
D) With Convetional heating: 82%



58

A) With Sodium Palmitate: 96%
B) With Sodium Linoleate: 93%
C) With TPGS=750-M: 90%
D) With Convetional heating: 81%

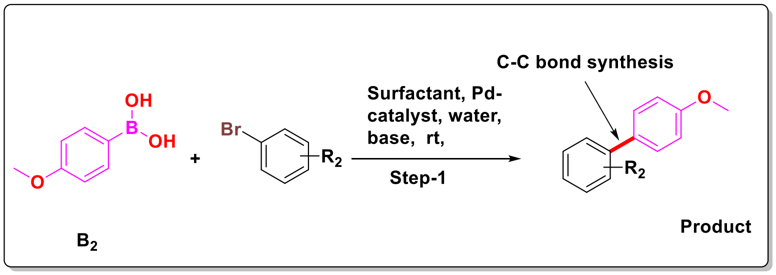


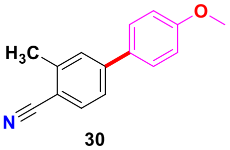
65

A) With Sodium Palmitate: 96%
B) With Sodium Linoleate: 93%
C) With TPGS=750-M: 89%
D) With Convetional heating: 79%

Unconventional surfactants like sodium palmitate and linoleate delivered superior yields in Suzuki cross-coupling reactions compared to TPGS-750M and conventional heating. These surfactants proved highly effective across diverse substrates, highlighting their potential as efficient, room-temperature alternatives.

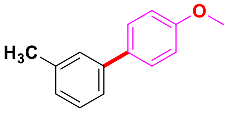
Table 4. Suzuki cross coupling reaction of (4-methoxyphenyl) boronic acid





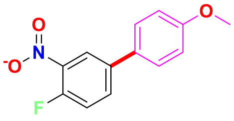
30

A) With Sodium Palmitate: 91%
B) With Sodium Linoleate: 87%
C) With TPGS=750-M: 88%
D) With Convetional heating: 71%



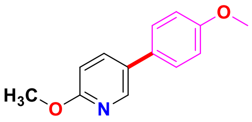
31

A) With Sodium Palmitate: 89%
B) With Sodium Linoleate: 89%
C) With TPGS=750-M: 86%
D) With Convetional heating: 78%



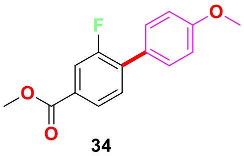
32

A) With Sodium Palmitate: 86%
B) With Sodium Linoleate: 82%
C) With TPGS=750-M: 81%
D) With Convetional heating: 71%



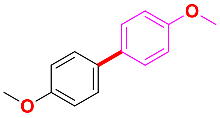
33

A) With Sodium Palmitate: 89%
B) With Sodium Linoleate: 88%
C) With TPGS=750-M: 87%
D) With Convetional heating: 77%



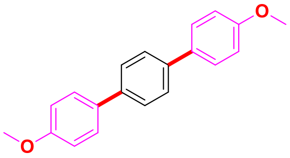
34

A) With Sodium Palmitate: 89%
B) With Sodium Linoleate: 84%
C) With TPGS=750-M: 83%
D) With Convetional heating: 75%



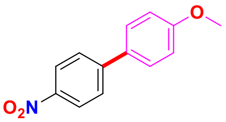
53

A) With Sodium Palmitate:90%
B) With Sodium Linoleate: 93%
C) With TPGS=750-M: 91%
D) With Convetional heating: 78%



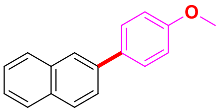
54

A) With Sodium Palmitate: 85%
B) With Sodium Linoleate: 82%
C) With TPGS=750-M: 89%
D) With Convetional heating: 80%



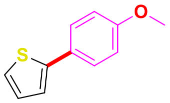
57

A) With Sodium Palmitate: 85%
B) With Sodium Linoleate: 86%
C) With TPGS=750-M: 78%
D) With Convetional heating: 69%



61

A) With Sodium Palmitate: 95%
B) With Sodium Linoleate: 93%
C) With TPGS=750-M: 89%
D) With Convetional heating: 78%

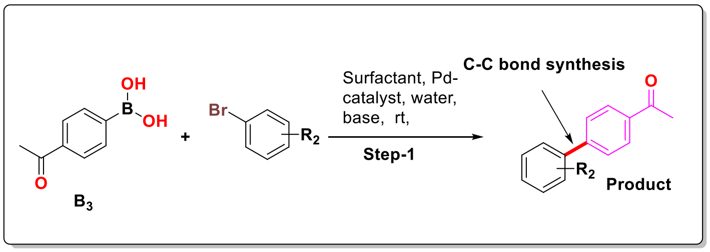


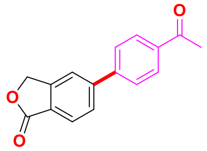
66

A) With Sodium Palmitate: 79%
B) With Sodium Linoleate: 81%
C) With TPGS=750-M: 82%
D) With Convetional heating: 61%

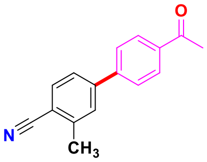
Suzuki cross-coupling with (4-methoxyphenyl) boronic acid showed high efficiency using sodium palmitate and linoleate, outperforming TPGS-750M and conventional heating. Electron-donating groups significantly enhanced yields across diverse substrates, confirming the effectiveness of these unconventional surfactants.

Table 5. Suzuki cross coupling reaction of (4-acetylphenyl) boronic acid

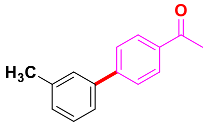




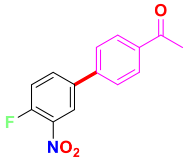
12
A) With Sodium Palmitate:97%
B) With Sodium Linoleate: 96%
C) With TPGS=750-M: 94%
D) With Convetional heating: 80%



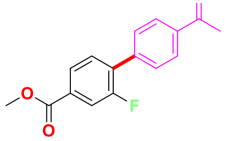
35
A) With Sodium Palmitate:87%
B) With Sodium Linoleate: 89%
C) With TPGS=750-M: 81%
D) With Convetional heating: 65%



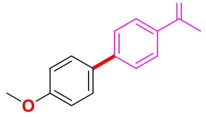
36
A) With Sodium Palmitate:90%
B) With Sodium Linoleate: 88%
C) With TPGS=750-M: 86%
D) With Convetional heating: 70%



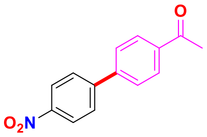
37
A) With Sodium Palmitate:87%
B) With Sodium Linoleate: 91%
C) With TPGS=750-M: 88%
D) With Convetional heating: 70%



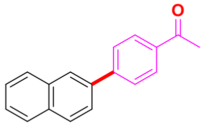
39
A) With Sodium Palmitate:80%
B) With Sodium Linoleate: 82%
C) With TPGS=750-M: 80%
D) With Convetional heating: 65%



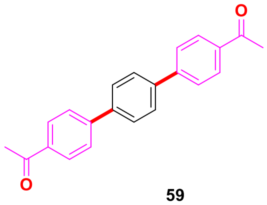
50
A) With Sodium Palmitate:83%
B) With Sodium Linoleate: 83%
C) With TPGS=750-M: 75%
D) With Convetional heating: 62%



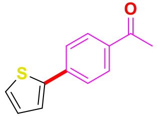
55
A) With Sodium Palmitate: 86%
B) With Sodium Linoleate: 90%
C) With TPGS=750-M: 82%
D) With Convetional heating: 69%



56
A) With Sodium Palmitate: 88%
B) With Sodium Linoleate: 89%
C) With TPGS=750-M: 78%
D) With Convetional heating: 60%



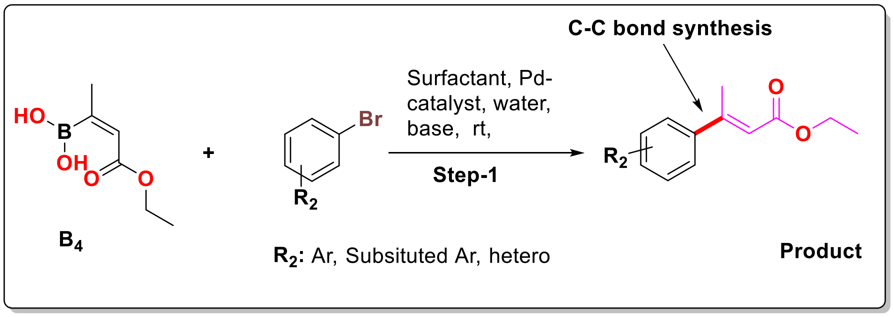
59
A) With Sodium Palmitate: 90%
B) With Sodium Linoleate: 88%
C) With TPGS=750-M: 78%
D) With Convetional heating: 63%

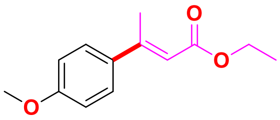


64
A) With Sodium Palmitate: 82%
B) With Sodium Linoleate: 80%
C) With TPGS=750-M: 83%
D) With Convetional heating: 61%

Suzuki reactions with (4-acetylphenyl) boronic acid delivered excellent yields, especially with sodium palmitate and linoleate, surpassing TPGS-750M and conventional heating. Electron-withdrawing groups significantly boosted efficiency across diverse substrates, affirming the strength of these surfactants.

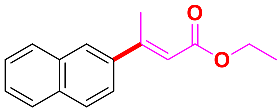
Table 6. Suzuki cross coupling reaction of (E)-(4-ethoxy-4-oxobut-2-en-2-yl) boronic acid





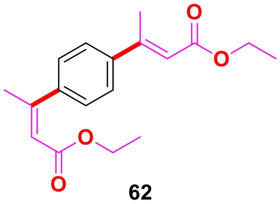
52

A) With Sodium Palmitate:92%
B) With Sodium Linoleate: 95%
C) With TPGS=750-M: 88%
D) With Convetional heating: 79%



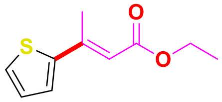
60

A) With Sodium Palmitate: 96%
B) With Sodium Linoleate: 94%
C) With TPGS=750-M: 91%
D) With Convetional heating: 79%



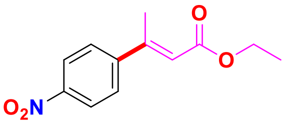
62

A) With Sodium Palmitate: 93%
B) With Sodium Linoleate: 91%
C) With TPGS=750-M: 85%
D) With Convetional heating: 71%



67

A) With Sodium Palmitate: 85%
B) With Sodium Linoleate: 83%
C) With TPGS=750-M: 82%
D) With Convetional heating: 62%

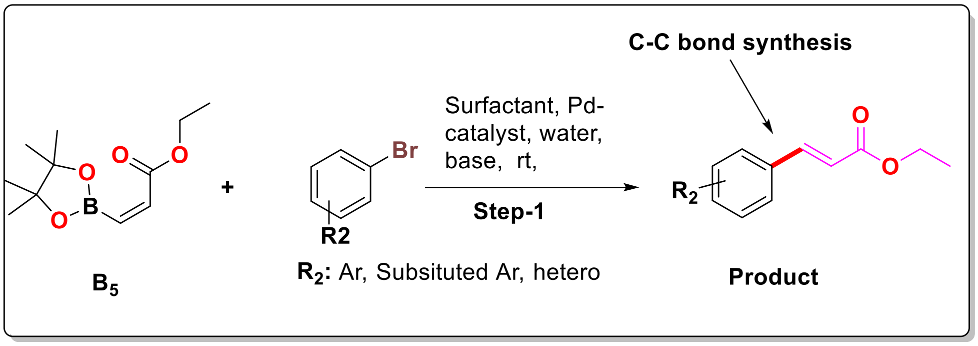


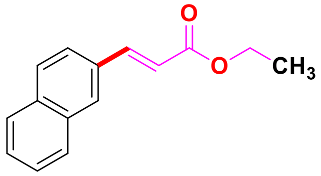
63

A) With Sodium Palmitate: 92%
B) With Sodium Linoleate: 91%
C) With TPGS=750-M: 81%
D) With Convetional heating: 75%

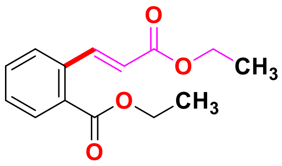
Suzuki cross-coupling with (E)-(4-ethoxy-4-oxobut-2-en-2-yl)boronic acid showed outstanding yields using sodium palmitate and linoleate, consistently outperforming TPGS-750M and conventional heating. The aliphatic side chain proved highly compatible across varied substrates, reinforcing the surfactants' broad applicability.

Table 7. Suzuki cross coupling reaction of ethyl (Z)-3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl) acrylate

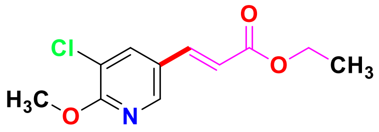




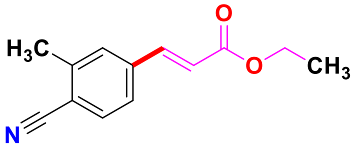
15
A) With Sodium Palmitate: 92%
B) With Sodium Linoleate: 93%
C) With TPGS=750-M: 89%
D) With Convetional heating: 70%



17
A) With Sodium Palmitate: 91%
B) With Sodium Linoleate: 91%
C) With TPGS=750-M: 90%
D) With Convetional heating: 71%

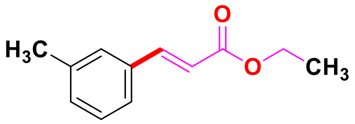


19
A) With Sodium Palmitate: 71%
B) With Sodium Linoleate: 72%
C) With TPGS=750-M: 72%
D) With Convetional heating: 55%

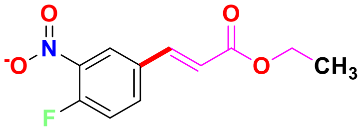


45

A) With Sodium Palmitate: 71%
B) With Sodium Linoleate: 72%
C) With TPGS=750-M: 72%
D) With Convetional heating: 55%

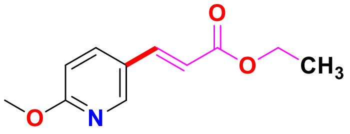


46
A) With Sodium Palmitate: 85%
B) With Sodium Linoleate: 81%
C) With TPGS=750-M: 83%
D) With Convetional heating: 56%

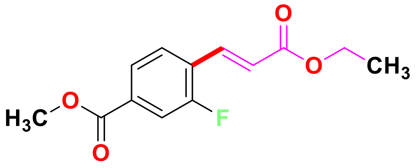


47

A) With Sodium Palmitate: 91%
B) With Sodium Linoleate: 89%
C) With TPGS=750-M: 85%
D) With Convetional heating: 65%



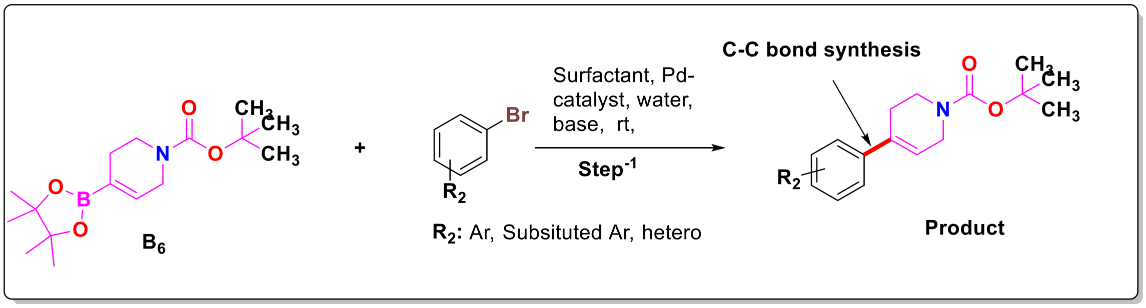
48
A) With Sodium Palmitate: 91%
B) With Sodium Linoleate: 89%
C) With TPGS=750-M: 85%
D) With Convetional heating: 65%

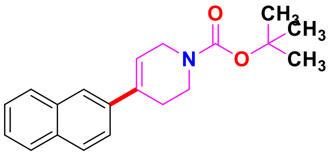


49
A) With Sodium Palmitate: 80%
B) With Sodium Linoleate: 83%
C) With TPGS=750-M: 79%
D) With Convetional heating: 57%

Suzuki cross-coupling using aliphatic ethyl acrylate-based boronic ester showed excellent yields with sodium linoleate and palmitate, confirming their efficiency across diverse substrates. These unconventional surfactants enable high-performance reactions under mild conditions.

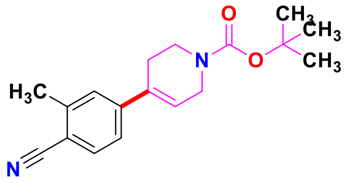
Table 8. Suzuki cross coupling reaction of tert-butyl 4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)-3,6-dihydropyridine-1(2H)-carboxylate





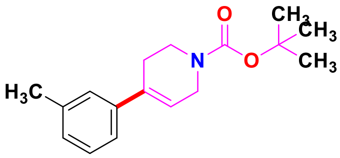
16

A) With Sodium Palmitate: 90%
B) With Sodium Linoleate: 90%
C) With TPGS=750-M: 89%
D) With Convetional heating: 61%



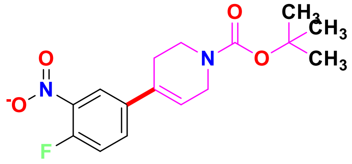
40

A) With Sodium Palmitate: 90%
B) With Sodium Linoleate: 90%
C) With TPGS=750-M: 89%
D) With Convetional heating: 61%



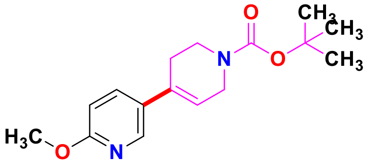
41

A) With Sodium Palmitate: 80%
B) With Sodium Linoleate: 78%
C) With TPGS=750-M: 75%
D) With Convetional heating: 61%



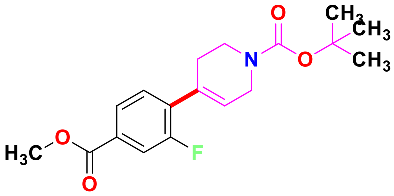
42

A) With Sodium Palmitate: 85%
B) With Sodium Linoleate: 83%
C) With TPGS=750-M: 82%
D) With Convetional heating: 66%



43

A) With Sodium Palmitate: 86%
B) With Sodium Linoleate: 86%
C) With TPGS=750-M: 82%
D) With Convetional heating: 62%

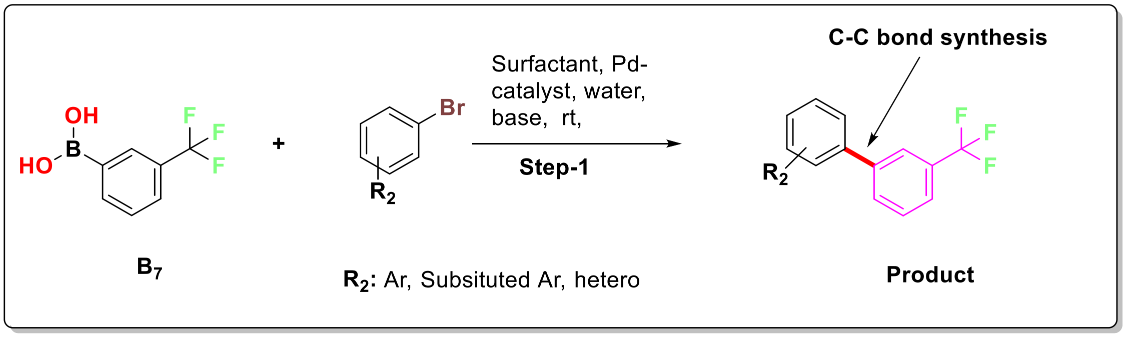


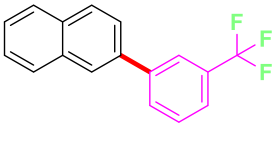
44

A) With Sodium Palmitate: 82%
B) With Sodium Linoleate: 83%
C) With TPGS=750-M: 82%
D) With Convetional heating: 70%

Suzuki cross-coupling with cyclic aliphatic tert-butyl boronic ester achieved strong yields, especially with sodium palmitate, demonstrating 82–86% yields across various substrates. Sodium linoleate also showed good performance, highlighting the versatility of unconventional surfactants in efficient reactions.

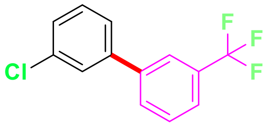
Table 9. Suzuki cross coupling reaction of (3-(trifluoromethyl)phenyl) boronic acid





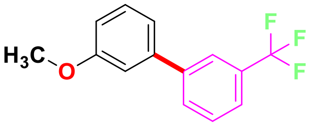
7

A) With Sodium Palmitate: 94%
B) With Sodium Linoleate: 85%
C) With TPGS=750-M: 74%
D) With Convetional heating: 80%



8

A) With Sodium Palmitate: 90%
B) With Sodium Linoleate: 85%
C) With TPGS=750-M: 90%
D) With Convetional heating: 93%

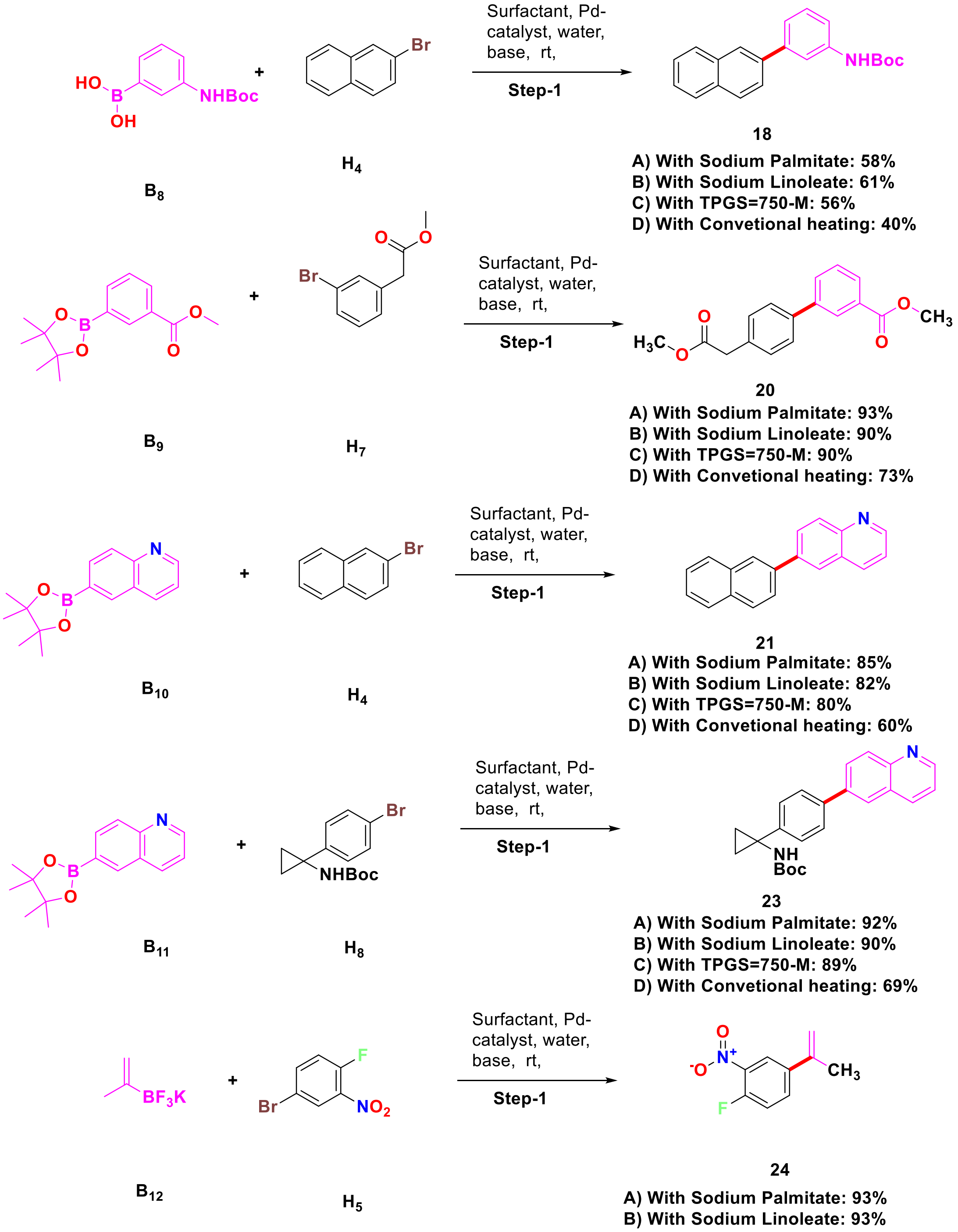


9

A) With Sodium Palmitate: 90%
B) With Sodium Linoleate: 91%
C) With TPGS=750-M: 82%
D) With Convetional heating: 80%

Suzuki cross-coupling with (3-(trifluoromethyl) phenyl) boronic acid demonstrated high yields, particularly with sodium palmitate outperforming TPGS-750M and conventional heating.

Table 10. Suzuki cross coupling reaction of some random boronate with different halogenated compounds



18
A) With Sodium Palmitate: 58%
B) With Sodium Linoleate: 61%
C) With TPGS=750-M: 56%
D) With Convetional heating: 40%

20
A) With Sodium Palmitate: 93%
B) With Sodium Linoleate: 90%
C) With TPGS=750-M: 90%
D) With Convetional heating: 73%

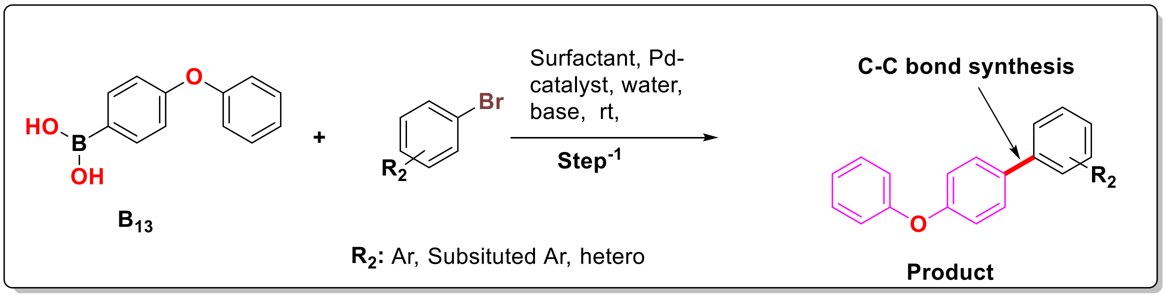
21
A) With Sodium Palmitate: 85%
B) With Sodium Linoleate: 82%
C) With TPGS=750-M: 80%
D) With Convetional heating: 60%

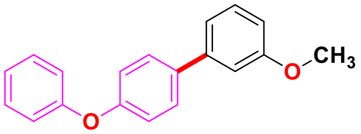
23
A) With Sodium Palmitate: 92%
B) With Sodium Linoleate: 90%
C) With TPGS=750-M: 89%
D) With Convetional heating: 69%

24
A) With Sodium Palmitate: 93%
B) With Sodium Linoleate: 93%
C) With TPGS=750-M: 90%
D) With Convetional heating: 71%

Random reactions of boronated compounds with halogenated compounds showed high yields, particularly with sodium palmitate and sodium linoleate, achieving 93% with methyl 3-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl) benzoate and potassium prop-1-en-2-yl trifluoroborate, demonstrating the effectiveness of unconventional surfactants.

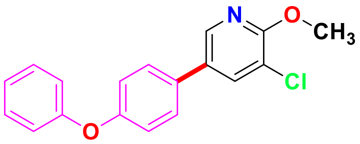
Table 11. Suzuki cross coupling reaction of (4-phenoxyphenyl) boronic acid





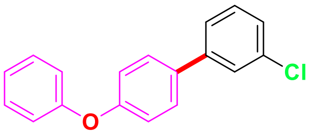
4

A) With Sodium Palmitate: 94%
B) With Sodium Linoleate: 90%
C) With TPGS=750-M: 90%
D) With Convetional heating: 78%



5

A) With Sodium Palmitate: 93%
B) With Sodium Linoleate: 93%
C) With TPGS=750-M: 82%
D) With Convetional heating: 70%

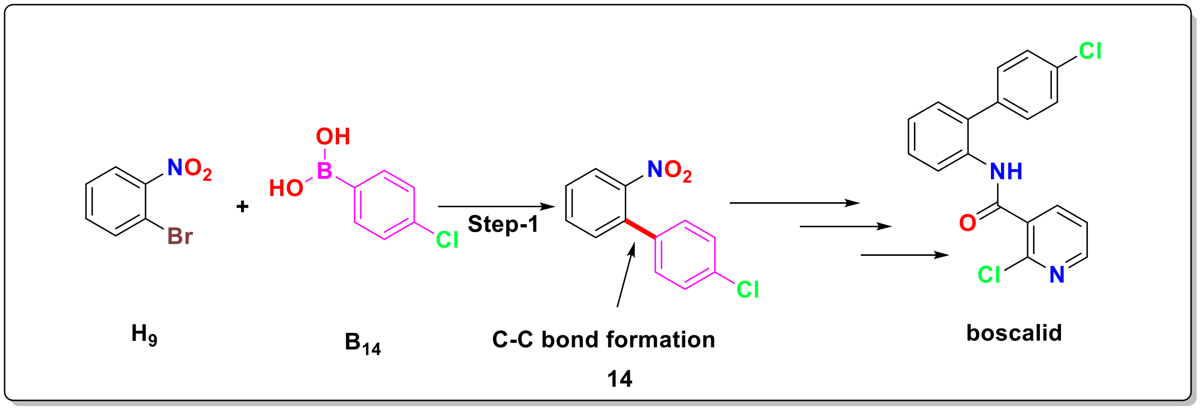


6

A) With Sodium Palmitate: 93%
B) With Sodium Linoleate: 92%
C) With TPGS=750-M: 96%
D) With Convetional heating: 65%

* Crude reaction conversion, ** isolated yields

Boscalid is an important pharma ingredient used as fungicide and prepared with the help of Suzuki cross coupling reaction (Scheme 1), We have applied our process of unconventional surfactant for the same and observed that the reaction of (4-chlorophenyl) boronic acid with 1-bromo-2-nitrobenzene gives **14** having 90% isolated yield with sodium palmitate (Condition A), 85% with sodium linoleate (Condition B), 88% with TPGS-750M (Condition c) and 73% with conventional heating reaction (Condition D). It means the use of unconventional surfactant for this Boscalid synthesis is good choice with respect to Yield, energy saving, cost, and process.



Scheme 1. Synthesis scheme for Boscalid.

Reaction condition-A : Bromo (1.0 eq), Boronate (1.5 eq), $PdCl_2$(dppf). DCM complex, 4% sodium palmitate (20 volumes), TEA (3.0 eq), rt, 2-3h, **Reaction condition-B :** Bromo (1.0 eq), Boronate (1.5 eq), $PdCl_2$(dppf) DCM complex, 4% sodium Linoleate (20 volumes), TEA (3.0 eq), rt, 2-3h, **Reaction condition-C :** Bromo (1.0 eq), Boronate (1.5 eq), $PdCl_2$(dppf) DCM complex, 4% TPGS-750M (20 volumes), TEA (3.0 eq), rt, 16h, **Reaction**

condition-D : Bromo (1.0 eq), Boronate (1.5 eq), $PdCl_2$(dppf) DCM complex, (20 volumes), Na_2CO_3 (3.0 eq), Dioxane : water, 90 °C, 2-16h

Overall comparison

This study demonstrates that unconventional surfactants—sodium palmitate and sodium linoleate—enable Suzuki cross-coupling reactions with yields comparable to TPGS-750M and significantly higher than conventional heating methods. These improvements stem from enhanced micellar organization and substrate solubilization. Aryl halides with electron-donating groups showed higher yields due to easier oxidative addition, while even less reactive electron-withdrawing groups and challenging substrates like heterocycles and polyhalogenated aromatics responded well, showcasing the method’s versatility. The use of these surfactants supports greener, more sustainable chemistry by reducing energy use and costs. The system showed broad substrate tolerance, efficiently handling boronic acids, boronate esters, potassium salt of boronates, and functional groups like lactones, N-Boc-amines, and halogens.

Extensive testing of palladium catalysts (e.g., Pd $(OAc)_2$, $PdCl_2$(dppf), $Pd_2(dba)_3$) and bases (e.g., TEA, DIPEA, K_2CO_3) confirmed high catalytic performance, with TEA yielding consistently superior results. Overall, 67 compounds were synthesized efficiently under ambient conditions with key green chemistry benefits: water as solvent, fast reactions, high yields and purity, minimal catalyst use, simple work-up, cost and eco-efficiency. This micellar approach offers a sustainable, high-performance alternative to traditional Suzuki methods, with broad applicability in pharmaceutical and industrial synthesis.

Conclusions

The Suzuki cross-coupling reaction has been successfully optimized using cost-effective, unconventional surfactants such as sodium palmitate and sodium linoleate in micellar catalysis, in conjunction with various palladium catalysts at room temperature. These readily available and affordable surfactants serve as viable alternatives to traditional high-cost surfactants while maintaining comparable catalytic efficiency. This innovative approach enables Suzuki cross-coupling under mild aqueous-phase conditions, eliminating the need for toxic organic solvents and significantly reducing energy-intensive reaction requirements. The success of this system underscores the potential of micellar catalysis in sustainable chemical processes. By adhering to green chemistry principles, this methodology offers several key advantages, including: Cost-effectiveness – Utilizing inexpensive and widely accessible surfactants. Environmental safety – Reducing reliance on hazardous solvents. Energy efficiency – Enabling reactions under mild conditions. Surfactant recyclability – Enhancing sustainability and minimizing waste. Overall, this scalable and environmentally friendly approach represents a significant advancement in organic synthesis, with promising implications for drug development and pharmaceutical applications.

Experimental Section

General. Commercially available chemicals, reagents, and surfactants, such as sodium palmitate and sodium linoleate, were used without further purification. Surfactant solutions were prepared using condensed, demineralized water. Products obtained after the workup were purified via combi flash chromatography or glass column chromatography using silica gel of various mesh sizes (60-120, 100-200, 350-400). Elution was typically done using n-hexane/ethyl acetate mixtures or MeOH/DCM. Product identification was carried out using spectroscopic methods, including ^{1}H NMR and ^{13}C NMR (using a 400 MHz Bruker system with solvents like DMSO-*d*$_6$, MeOD-d$_4$, or CDCl$_3$), and HRMS analysis. Melting points were measured with a Bucki instrument in an open capillary tube and are uncorrected. The melting points of all compounds and intermediates were determined using an Avi Scientific Blue Digital Melting Point Apparatus. The values are uncorrected. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker Ascend™ 400 spectrometer (400 MHz), with and without tetramethylsilane (TMS) as an internal standard. Chemical shifts (δ-values) are reported in parts per million (ppm) downfield from TMS. Splitting patterns are designated as follows: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet, for unresolved or overlapping signals), and br (broad signal). Solvents used are indicated in parentheses. Mass analysis and LCMS data were obtained using a Shimadzu LC-MS QP1000 EX instrument. Reagents, laboratory-grade (LR) solvents, and key starting materials were procured from BLD Pharma (Hyderabad, India), Spectrochem (Pune, India), Combi-Blocks Ltd. (Delhi, India), and Merck (Mumbai, India). Thin-layer chromatography (TLC) was performed using silica gel 60F-254 plates (Merck). Mobile phases included hexane : ethyl acetate or dichloromethane: methanol mixtures. Compound spots were visualized under UV light or by staining with potassium permanganate (KMnO$_4$), phosphomolybdic acid (PMA), or ninhydrin. Compound purification and intermediate isolation were carried out using a Combi-Flash chromatography system with a programmable injection spigot, capable of solvent flow rates of 1–200 mL/min and a maximum pressure of 200 psi.

Reaction Condition [A] : General experimental procedure for the synthesis of C-C bond in sodium palmitate as surfactant : The Boronate solution (0.612 moles, 1.20 equivalents) was combined with water (10 volumes), then halogen (0.510 moles, 1.00 equivalent) and a 4% surfactant solution (10-20 volumes, 1.5 moles, 3.00 equivalents) were added, along with an organic base (1.53 moles, 3.0 equivalents). The reaction mixture was purged with nitrogen or argon for 15 minutes. A palladium catalyst (0.025 moles, 0.05 equivalent) was then introduced, and the mixture was stirred at room temperature for 2 hours while progress was monitored by TLC. After completion, water and ethyl acetate were added to form an emulsion. The mixture was filtered through a Celite bed, washed with ethyl acetate, and the organic layer was separated and evaporated under vacuum to yield a crude product. This crude product was purified using column chromatography, and the collected fractions were evaporated to yield the desired product in good yield.

Reaction Condition [B]: General experimental procedure for the synthesis of C-C bond in sodium linoleate as surfactant: The reaction involved adding Boronates (0.612 moles, 1.20 eq) in water (10 vol) to halogen (0.510 moles, 1.00 eq) followed by a 4% surfactant solution (10 vol, 1.5 moles, 3.00 eq) and an organic base (1.53 moles, 3.0 eq). The mixture was purged with nitrogen or argon for 15 minutes. Palladium catalyst (0.025 moles, 0.05 eq) was added and stirred at room temperature for 2 hours while monitoring with TLC. After completion, water and EtOAc were added to form an emulsion, filtered through a Celite bed, and washed with EtOAc. The organic layer was separated, evaporated under vacuum to yield crude product, which was purified by column chromatography. The purified product was obtained by evaporating the eluted fractions, resulting in a good yield.

Reaction Condition [C]: General experimental procedure for the synthesis of C-C bond in TPGS-750M as surfactant: The synthesis process started with Boronates (0.612 moles, 1.2 eq) dissolved in water (5 volumes), to which halogen (0.510 moles, 1.0 eq) was added, followed by a 4% surfactant solution (10 volumes, 1.5 moles, 3.0 eq as solid) and an organic base (1.53 moles, 3.0 eq). The mixture was purged with nitrogen or argon gas for 10-15 minutes. Palladium catalyst (0.025 moles, 0.05 eq) was added, and the reaction mass was stirred at room temperature for 16 hours with TLC monitoring. Upon completion, water and EtOAc were added to form an emulsion. The mixture was filtered through a Celite bed and washed with EtOAc 2-3 times. The organic layer was separated and evaporated under vacuum to yield a crude product. This crude product was purified using column chromatography, and the purified fractions were evaporated to yield the desired product with good yield.

Reaction Condition [D]: General experimental procedure for the synthesis of C-C bond in Suzuki reaction via conventional heating reaction condition: The procedure began by dissolving Boronates (0.612 moles, 1.2 eq) in a mixture of dioxane (8 volumes) and water (2 volumes). Halogen (0.510 moles, 1.0 eq) was then added, followed by an inorganic base (1.53 moles, 3.0 eq). The reaction mass was purged with nitrogen or argon gas for 15 minutes before the addition of a Palladium catalyst (0.051 moles, 0.1 eq). The mixture was stirred at 90-100°C for 2-16 hours, with the reaction progress monitored by TLC. After completion, the reaction mass was filtered through a Celite bed, and the bed was washed with EtOAc. The organic layer was separated and evaporated under vacuum to yield a crude product, which was subsequently purified by column chromatography. The collected elutes were evaporated to obtain the desired product.

Scale up reaction:

Synthesis of 4-methoxy-1,1'-biphenyl (51) : To a solution of bromobenzene (10.0 g, 63.69 mmol, 1.0 equivalent) and (4-methoxyphenyl) boronic acid (11.2 g, 76.43 mmol, 1.2 equivalents) in water (50 mL), a sodium palmitate solution (100 mL, 4% w/v in water) was added. Triethylamine (19.2 g, 191.0 mmol, 3.0 equivalents) was introduced, and the reaction mixture was purged with nitrogen gas for 20 minutes. $PdCl_2$(dppf)·DCM complex (2.5 g, 3.185 mmol, 0.05 equivalent) was then added at room temperature, and the reaction mixture was stirred for 2 hours. Water (100 mL) and ethyl acetate (100 mL) were added to the reaction mixture, forming an emulsion. The mixture was filtered through a Celite bed, and the bed was washed with an additional 100 mL of ethyl acetate. The organic phase was separated, washed with brine, dried over anhydrous sodium sulphate, and concentrated under reduced pressure to afford a crude residue. The residue was purified by fractional column chromatography using silica gel (60–120 mesh) with 0–5% ethyl acetate in hexane as the mobile phase. The product, 4-methoxy-1,1'-biphenyl, was obtained as a white solid (14.0 g, 92% yield).

Synthesis of 4,4'-dimethoxy-1,1'-biphenyl (52) : To a solution of 1-bromo-4-methoxybenzene (10.0 g, 53.47 mmol, 1.0 equivalent) and (4-methoxyphenyl) boronic acid (9.6 g, 64.017 mmol, 1.2 equivalents) in water (50 mL), a sodium palmitate solution (100 mL, 4% w/v in water) was added. Triethylamine (16.2 g, 160.42 mmol, 3.0 equivalents) was introduced, and the reaction mixture was purged with nitrogen gas for 20 minutes. $PdCl_2$(dppf)·DCM complex (2.1 g, 2.674 mmol, 0.05 equivalent) was then added at room temperature, and the reaction mixture was stirred for 2 hours. Water (100 mL) and ethyl acetate (100 mL) were added to the reaction mixture, forming an emulsion. The mixture was filtered through a Celite bed, and the bed was washed with an additional 100 mL of ethyl acetate. The organic phase was separated, washed with brine, dried over anhydrous sodium sulphate, and concentrated under reduced pressure to afford a crude residue. The residue was purified by fractional column chromatography using silica gel (60–120 mesh) with 0–5% ethyl acetate in hexane as the mobile phase. The product, 4,4'-dimethoxy-1,1'-biphenyl (53), was obtained as a white solid (11.0 g, 96% yield).

Synthesis of 4-methoxy-4'-nitro-1,1'-biphenyl (57) : To a solution of 1-bromo-4-nitrobenzene (10.0 g, 49.50 mmol, 1.0 equivalent) and (4-methoxyphenyl) boronic acid (8.9 g, 59.40 mmol, 1.2 equivalents) in water (50 mL), a sodium palmitate solution (100 mL, 4% w/v in water) was added. Triethylamine (15 g, 148.51 mmol, 3.0 equivalents) was introduced, and the reaction mixture was purged with nitrogen gas for 20 minutes. $PdCl_2$(dppf)·DCM complex (2.02 g, 2.475 mmol, 0.05 equivalent) was then added at room temperature, and the reaction mixture was stirred for 2 hours. Water (100 mL) and ethyl acetate (100 mL) were added to the reaction mixture, forming an emulsion. The mixture was filtered through a Celite bed, and the bed was washed with an additional 100 mL of ethyl acetate. The organic phase was separated, washed with brine, dried over anhydrous sodium sulphate, and concentrated under reduced pressure to afford a crude residue. The residue was purified by fractional column chromatography using silica gel (60–120 mesh) with 0–5% ethyl acetate in hexane as the mobile phase. The product, 4-methoxy-4'-nitro-1,1'-biphenyl (**57**), was obtained as a white solid (9.59 g, 85% yield).

3-Chloro-1,1'-biphenyl (1) : Analytical data in accordance with literature.[26]

3-Phenoxy-1,1'-biphenyl (2): Melting Point: 123-126°C, ^{1}H NMR (400 MHz, DMSO-*d*$_6$): δ 7.66 (d, *J*=7.9 Hz, 2H, ArH), 7.53 – 7.34 (m, 7H, ArH), 7.32 – 7.29 (m, 1H, ArH), 7.17 (t, *J* = 7.3 Hz, 1H, ArH), 7.08 (d, *J* = 7.9 Hz, 2H, ArH), 7.02 – 6.97 (m, 1H, ArH), ^{13}C NMR (100 MHz, DMSO-*d*$_6$): δ 157.7, 157.0, 142.7, 139.8, 131.0, 130.5, 129.4, 128.2, 127.2, 124.0, 122.3, 119.2, 117.9, 117.2**,** HRMS: *m/z* calculated for $C_{18}H_{14}O$: 247.3090; [M+H] $^+$ found: 247.3059, Rf=0.53, TLC System Hexane: EtOAc, 9:1, Physical appearance: white solid

3-Methoxy-1,1'-biphenyl (3) : Analytical data in accordance with literature.[26]

3-Methoxy-4'-phenoxy-1,1'-biphenyl (4): ^{1}H NMR (400 MHz, DMSO-*d*$_6$): δ 7.74 – 7.67 (m, 2H, ArH), 7.47 – 7.34 (m, 3H, ArH), 7.24 – 7.14 (m, 3H, ArH), 7.12 – 7.04 (m, 4H, ArH), 6.92 (dd, *J* = 8.1, 2.5 Hz, 1H, ArH), 3.82 (s, 3H, -O$\underline{CH_3}$)**,** ^{13}C NMR (100 MHz, DMSO-*d*$_6$): δ 160.2, 156.97, 156.9, 141.5, 135.7, 130.5, 130.4, 128.8, 124.1, 119.3, 119.2, 113.2, 112.5, 55.3**,** HRMS: *m/z* calculated for $C_{19}H_{16}O_2$: 277.3350; [M+H] $^+$ found: 277.3345, Rf=0.57, TLC System Hexane: EtOAc, 9:1, Physical appearance: Colorless liquid

3-Chloro-2-methoxy-5-(4-phenoxyphenyl) pyridine (5): Melting Point: 85-88°C, ^{1}H NMR (400 MHz, DMSO-*d*$_6$): δ 8.47 (d, *J* = 2.3 Hz, 1H, ArH), 8.23 (d, *J* = 2.3 Hz, 1H, ArH), 7.75 (d, *J* = 8.6 Hz, 2H, ArH), 7.47 – 7.41 (m, 2H, ArH), 7.20 (t, *J* = 7.3 Hz, 1H, ArH), 7.11-7.08 (m, 4H, ArH), 4.00 (s, 3H, -O$\underline{CH_3}$), ^{13}C NMR (100 MHz, DMSO-*d*$_6$): δ 158.1, 157.2, 156.7, 142.9, 137.0, 131.1, 130.6, 130.4, 128.7, 124.2, 119.4, 119.3, 117.6, 54.6**,** **HRMS:** *m/z* calculated for $C_{18}H_{14}ClNO_2$: 311.7650, 313.7650; [M+H] $^+$ found: 311.7611, 313.7615, Rf=0.61, TLC System Hexane: EtOAc, 8:2, Physical appearance: Dark yellow solid

3-Chloro-4'-phenoxy-1,1'-biphenyl (6): Melting Point: 82-85 °C, ^{1}H NMR (400 MHz, DMSO-*d*$_6$): δ 7.79 – 7.70 (m, 3H, ArH), 7.65 (d, *J* = 7.7 Hz, 1H, ArH), 7.56 – 7.39 (m, 4H, ArH), 7.21 (t, *J* = 7.4 Hz, 1H, ArH), 7.14 – 7.07 (m, 4H, ArH), ^{13}C NMR (100 MHz, DMSO-*d*$_6$): δ 157.4, 156.7, 142.1, 134.2, 134.1, 131.1, 130.5, 128.9, 127.4, 126.9, 126.6, 125.5, 124.2, 119.5, 119.1, HRMS: *m/z* calculated for $C_{18}H_{13}ClO$: 280.7510, 282.7510; [M, M+2] $^+$ found: 281.7479, 283.7484, Rf=0.65, TLC System Hexane: EtOAc, 9.5:0.5, Physical appearance: Off white Solid

2-(3-(Trifluoromethyl) phenyl) naphthalene (7): Melting Point: 120-122°C, ^{1}H NMR (400 MHz, DMSO-*d*$_6$): δ 8.37 (s, 1H, ArH), 8.17-8.15 (m, 2H, ArH), 8.07 – 8.04 (m, 2H, ArH), 7.99 – 7.93 (m, 2H, ArH), 7.78– 7.77 (m, 2H, ArH), 7.60-7.55 (m, 2H, ArH)**,** ^{13}C NMR (100 MHz, DMSO-*d*$_6$): δ 141.5, 136.2, 133.7, 133.0 ($^2J_{(C–F)=}$ 30Hz), 131.4, 130.5, 129.1, 128.8, 127.9, 127.0, 126.9, 126.3, 125.3, 124.55, 124.51, ($^1J_{(C–F)=}$ 320.7Hz). 123.8, 123.7, HRMS : *m/z* calculated for $C_{17}H_{11}F_3$: 273.2702; [M+H] $^+$ found : 273.2681, Rf=0.67, TLC System Hexane : EtOAc, 9:1, Physical appearance : Off white Solid

3-Chloro-3'-(trifluoromethyl)-1,1'-biphenyl (8) : Melting Point : 40-43 °C**,** ^{1}H NMR (400 MHz, DMSO-*d*$_6$): δ 8.10 – 8.03 (m, 2H, ArH), 7.92 – 7.86 (m, 1H, ArH), 7.82 – 7.72 (m, 3H, ArH), 7.59 – 7.49 (m, 2H, ArH), ^{13}C NMR (100 MHz, DMSO-*d*$_6$): δ 145.4, 141.1, 140.1, 134.3, 131.3, 130.5, 128.5, 127.2, 126.1, 125.0 ($^1J_{(C–F)=}$ 30Hz), 123.8, HRMS : *m/z* calculated for $C_{13}H_8ClF_3$:256.6522, 258.6522; [M, M+2] $^+$ found: 256.6486, 258.6481, Rf=0.70, TLC System Hexane : EtOAc, 9.5:0.5, Physical appearance: white Solid

3-Methoxy-3'-(trifluoromethyl)-1,1'-biphenyl (9): ^{1}H NMR (400 MHz, DMSO-*d*$_6$): δ 8.04 – 7.95 (m, 2H, ArH), 7.81 – 7.68 (m, 2H, ArH), 7.42 (t, *J* = 8.2 Hz, 1H, ArH), 7.36 – 7.25 (m, 2H, ArH), 7.01 (dd, *J* = 8.2, 2.6 Hz, 1H, ArH), 3.85 (s, 3H, -O$\underline{CH_3}$)**,** ^{13}C NMR (100 MHz, DMSO-*d*$_6$): δ 160.3, 141.6, 140.5, 131.4, 130.6, 130.4 ($^2J_{(C–F)=}$ 30 Hz), 124.63, 124.60 ($^1J_{(C–F)=}$320.6 Hz), 123.7, 119.7, 114.4, 112.8, 55.6, HRMS: *m/z* calculated for $C_{14}H_{11}F_3O$: 253.2362; [M+H] $^+$ found: 253.2359**,** Rf=0.54, TLC System Hexane: EtOAc, 9:1, Physical appearance: Colorless liquid

5-(4-Phenoxyphenyl) isobenzofuran-1(3H)-one (10): Melting Point: 127-130°C, ^{1}H NMR (400 MHz, DMSO-*d*$_6$): δ 7.98 – 7.86 (m, 3H, ArH), 7.83 – 7.78 (m, 2H, ArH), 7.45 (t, *J* = 8.0 Hz, 2H, ArH), 7.21 (t, *J* = 7.5 Hz, 1H, ArH), 7.15 – 7.11 (m, 4H, ArH), 5.47 (s, 2H, -$\underline{CH_2}$-O-CO-)**,** ^{13}C NMR (100 MHz, DMSO-*d*$_6$): δ 170.9, 158.0, 156.4, 148.7, 145.7, 134.3, 130.6, 129.5, 127.9, 125.8, 124.5, 124.0, 121.0, 119.7, 119.1, 70.3**,** HRMS: *m/z* calculated for $C_{20}H_{14}O_3$: 303.3290; [M+H] $^+$ found: 303.3197, Rf=0.55, TLC System Hexane: EtOAc, 7:3, Physical appearance: off white Solid

1-(6-Methyl-4'-phenoxy-[1,1'-biphenyl]-3-yl) ethan-1-one (11): ^{1}H NMR (400 MHz, DMSO-*d*$_6$): δ 7.87 (d, *J* = 7.9 Hz, 1H, ArH), 7.78 (s, 1H, ArH), 7.52 – 7.38 (m, 5H, ArH), 7.20 (t, *J* = 7.4 Hz, 1H, ArH), 7.10 (t, *J* = 8.2 Hz, 4H, ArH), 3.86 (s, 3H, -O$\underline{CH_3}$), 2.34 (s, 3H, Ar-$\underline{CH_3}$), ^{13}C NMR (100 MHz, DMSO-*d*$_6$): δ 166.5, 156.8, 156.7, 141.4, 135.5, 131.4, 131.0, 130.6, 128.3, 127.9, 124.2, 119.5, 118.6, 52.5, 20.9, HRMS: *m/z* calculated for $C_{21}H_{18}O_3$: 319.3720; [M+H] $^+$ found: 319.3712, Rf=0.51, TLC System Hexane: EtOAc, 9:1, Physical appearance: Yellow colored gum

5-(4-Acetylphenyl) isobenzofuran-1(3H)-one (12): Melting Point: 179-181°C, ^{1}H NMR (400 MHz, DMSO-*d*$_6$): δ 8.15 – 8.12 (d, *J* = 8.2 Hz, 2H, ArH), 8.09 (s, 1H, ArH), 7.99 (s, 2H, ArH), 7.96 (d, *J* = 8.2 Hz, 2H, ArH), 5.52 (s, 2H, -$\underline{CH_2}$-O-CO-), 2.67 (s, 3H, -CO-$\underline{CH_3}$)**,** ^{13}C NMR (100 MHz, DMSO-*d*$_6$): δ 197.9, 176.7, 148.7, 145.1, 143.6, 136.9, 129.4, 128.4, 128.0, 125.9, 125.1, 121.9, 70.3, 27.1, HRMS: *m/z* calculated for $C_{16}H_{12}O_3$: 253.2690; [M+H] $^+$ found: 253.2595**,** Rf=0.52, TLC System Hexane: EtOAc, 7:3, Physical appearance: off white Solid

2-Phenylnaphthalene (13) : Analytical data in accordance with literature.[26]

4'-Chloro-2-nitro-1,1'-biphenyl (14): Melting Point: 62-65°C, ^{1}H NMR (400 MHz, DMSO-*d*$_6$): δ 8.03 (dd, *J* = 8.1, 1.3 Hz, 1H, ArH), 7.81-7.77 (m, 1H, ArH), 7.69-7.64 (m, 1H, ArH), 7.60 – 7.52 (m, 3H, ArH), 7.39 (d, *J* = 8.4 Hz, 2H, ArH)**,** ^{13}C NMR (100 MHz, DMSO-*d*$_6$): δ 149.1, 136.4, 134.4, 133.6, 132.3, 130.1, 129.3, 129.2, 124.7, HRMS: *m/z* calculated for $C_{12}H_8ClNO_2$: 233.6510, 235.6510; [M, M+2] $^+$ found: 233.6392, 235.6390**,** Rf=0.53, TLC System Hexane: EtOAc, 8:2, Physical appearance: White Solid

Ethyl (Z)-3-(naphthalen-2-yl) acrylate (15): ^{1}H NMR (400 MHz, DMSO-*d*$_6$): δ 8.23 (s, 1H, ArH), 7.98 – 7.90 (m, 4H, ArH), 7.82 (d, *J* = 16.0 Hz, 1H, Ar-$\underline{CH}$=CH-CO), 7.62 – 7.54 (m, 2H, , ArH), 6.78 (d, *J* = 16.0 Hz, 1H, Ar-CH=$\underline{CH}$-CO), 4.22 (q, *J* = 7.1 Hz, 2H, -$\underline{CH_2}$-CH$_3$), 1.29 (t, *J* = 7.1 Hz, 3H, -CH$_2$-$\underline{CH_3}$), ^{13}C NMR (100 MHz, DMSO-*d*$_6$): δ 166.7, 144.8, 134.2, 133.3, 132.1, 130.4, 128.9, 128.1, 127.7, 127.2, 124.3, 118.9, 60.5, 14.6, HRMS: *m/z* calculated for $C_{15}H_{14}O_2$: 227.2750; [M+H] $^+$ found: 227.2741, Rf=0.65, TLC System Hexane: EtOAc, 8:2, Physical appearance: Pale Yellow colored liquid

Tert-butyl 4-(naphthalen-2-yl)-3,6-dihydropyridine-1(2H)-carboxylate (16): ^{1}H NMR (400 MHz, DMSO-*d*$_6$): δ 7.95 – 7.82 (m, 4H, ArH), 7.68 (d, *J* = 8.8 Hz, 1H, ArH), 7.53 – 7.45 (m, 2H, ArH), 6.45-6.29 (m, 1H, ArH), 4.15-3.91 (m, 2H, =CH-$\underline{CH_2}$-*N*), 3.59-3.57 (m, 2H, -CH$_2$-$\underline{CH_2}$-*N*) 2.49-2.62 (m, 2H, -$\underline{CH_2}$-CH$_2$-*N*), 1.43 (s, 9H, O-C(CH$_3$)$_3$, Boc), ^{13}C NMR (100 MHz, DMSO-*d*$_6$): δ 154.3, 137.6, 134.6, 133.5, 132.7, 128.5, 128.2, 127.8, 126.7, 126.3, 123.7, 123.5, 122.2, 79.3, 46.2, 44.0, 28.5, 27.0, HRMS: *m/z* calculated for $C_{20}H_{23}NO_2$: 310.4090; [M+H] $^+$ found: 310.4083**,** Rf=0.55, TLC System Hexane: EtOAc, 6:4, Physical appearance: Colorless gum

Ethyl (E)-2-(3-ethoxy-3-oxoprop-1-en-1-yl) benzoate (17): ^{1}H NMR (400 MHz, DMSO-*d*$_6$): δ 8.31 (d, *J* = 15.9 Hz, 1H, Ar-$\underline{CH}$=CH-), 7.89 (t, *J* = 7.5 Hz, 2H, ArH), 7.65 (t, *J* = 7.5 Hz, 1H, ArH), 7.56 (t, *J* = 7.5 Hz, 1H, ArH), 6.54 (d, *J* = 15.9 Hz, 1H, Ar-CH=$\underline{CH}$-), 4.35 (q, *J* = 7.1 Hz, 2H, -$\underline{CH_2}$-CH$_3$), 4.22 (q, *J* = 7.1 Hz, 2H, $\underline{-CH_2}$-CH$_3$), 1.35 (t, *J* = 7.1 Hz, 3H, -CH$_2$-$\underline{CH_3}$), 1.28 (t, *J* = 7.1 Hz, 3H, -CH$_2$-$\underline{CH_3}$)**,** ^{13}C NMR (100 MHz, DMSO-*d*$_6$): δ 166.7, 166.3, 143.1, 135.2, 132.9, 130.7, 130.5, 130.3, 128.3, 121.1, 61.6, 60.6, 14.6, 14.4, HRMS: *m/z* calculated for $C_{14}H_{16}O_4$: 249.2780; [M+H] $^+$ found: 249.2777, Rf=0.69, TLC System Hexane: EtOAc, 8:2, Physical appearance: Colorless semisolid

Tert-butyl (3-(naphthalen-2-yl) phenyl) carbamate (18): Melting Point: 166-168 °C**,** ^{1}H NMR (400 MHz, DMSO-*d*$_6$): δ 9.49 (s, 1H, -$\underline{NH}$-CO-), 8.15 (s, 1H, ArH), 8.05-8.02 (m, 2H, ArH), 7.98-7.96 (m, 2H, ArH), 7.80 – 7.77 (m, 1H, ArH), 7.63 – 7.53 (m, 2H, ArH), 7.49-7.47 (m, 1H, ArH), 7.42-7.39 (m, 2H, ArH), 1.52 (s, 9H, O-C(CH$_3$)$_3$, Boc), ^{13}C NMR (100 MHz, DMSO-*d*$_6$): δ 153.3, 141.0, 140.6, 138.2, 133.7, 132.7, 129.7, 129.3, 128.9, 128.6, 127.9, 126.9, 126.5, 125.5, 121.3, 117.8, 117.1, 79.6, 28.6, HRMS: *m/z* calculated for $C_{21}H_{21}NO_2$: 320.4040; [M+H] $^+$ found: 320.4018**,** Rf=0.39, TLC System Hexane: EtOAc, 5:5, Physical appearance: Pale yellow solid

Ethyl (E)-3-(5-chloro-6-methoxypyridin-3-yl) acrylate (19): Melting Point: 110-112 °C**,** ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.49 – 8.38 (m, 2H, ArH), 7.66-7.58 (m, 1H, ArH), 6.76-6.68 (m, 1H, ArH), 4.20 – 4.15 (m, 2H, -O$\underline{CH_2}$-CH$_3$), 3.98 (s, 3H, Ar-O$\underline{CH_3}$), 1.29 – 1.20 (m, 3H, -OCH$_2$-$\underline{CH_3}$), ^{13}C NMR (100 MHz, DMSO-d_6): δ 165.4, 159.9, 146.9, 140.1, 137.1, 125.7, 119.1, 118.1, 66.8, 55.0, 14.6**,** HRMS: *m/z* calculated for $C_{11}H_{12}ClNO_3$: 241.6710, 243.6710; [M, M+2] $^+$ found: 241.6695, 243.6612**,** Rf=0.54, TLC System Hexane: EtOAc, 6:4, Physical appearance: White Solid

Methyl 4'-(2-methoxy-2-oxoethyl)-[1,1'-biphenyl]-3-carboxylate (20): ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.19 (s, 1H, ArH), 7.96 (d, *J* = 7.7 Hz, 2H, ArH), 7.73 – 7.60 (m, 3H, ArH), 7.40 (d, *J* = 7.7 Hz, 2H, ArH), 3.90 (s, 3H, ArCO-O$\underline{CH_3}$), 3.76 (s, 2H, Ar-$\underline{CH_2}$-CO), 3.64 (s, 3H, Ar-CH$_2$CO-O$\underline{CH_3}$), ^{13}C NMR (100 MHz, DMSO-d_6): δ 171.9, 166.6, 140.8, 138.0, 134.7, 131.8, 130.8, 130.5, 129.9, 128.5, 127.4, 127.2, 52.7, 52.2, 40.6**,** HRMS: *m/z* calculated for $C_{17}H_{16}O_4$: 285.3110; [M+H] $^+$ found: 285.3107, Rf=0.59, TLC System Hexane: EtOAc, 9:1**,** Physical appearance: Colourless semisolid

6-(Naphthalen-2-yl) quinoline (21): Melting Point : 125-127 °C, ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.94-8.93 (m, 1H, ArH), 8.50-8.43 (m, 3H, ArH), 8.28 (d, *J* = 8.8 Hz, 1H, ArH), 8.16 (d, *J* = 8.8 Hz, 1H, ArH), 8.12 – 8.02 (m, 3H, ArH), 8.02 – 7.98 (m, 1H, ArH), 7.64 – 7.53 (m, 3H, ArH), ^{13}C NMR : Solubility issue in DMSO-d_6 as well so ^{13}C NMR not recorded**,** HRMS: *m/z* calculated for $C_{19}H_{13}N$: 256.3200; [M+H] $^+$ found: 256.3185, Rf=0.53, TLC System Hexane: EtOAc, 6:4, Physical appearance: Dark yellow solid

5-(4-Fluoro-3-nitrophenyl)-2-methoxypyridin-3-amine (22): Melting Point: 178-180 °C, ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.25-8.22 (m, 1H), 8.04-7.99 (m, 1H, ArH), 7.74 (t, *J* = 2.3 Hz, 1H, ArH), 7.69-7.64 (m, 1H, ArH), 7.20 (t, *J* = 2.3 Hz, 1H, ArH), 5.18 (bs, 2H, Ar-$\underline{NH_2}$), 3.92 (s, 3H, -O$\underline{CH_3}$), ^{13}C NMR (100 MHz, DMSO-d_6): δ 155.4 ($^1J_{(C–F)=}$ 245.5Hz), 152.8, 152.5, 137.7, 137.6 ($^4J_{(C–F)=}$ 30Hz), 134.8, 134.1, 134.0, 133.2, 130.8, 127.3, 123.4, 119.5, 119.3, 116.9, 53.5, HRMS: *m/z* calculated for $C_{12}H_{10}FN_3O_3$: 264.2284; [M+H] $^+$ found: 264.2271, Rf=0.66, TLC System Hexane: EtOAc, 5:5, Physical appearance: Brown solid

Tert-butyl (1-(4-(quinolin-6-yl) phenyl) cyclopropyl) carbamate (23): ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.92 (s, 1H, -$\underline{NH}$-CO-), 8.45 (d, *J* = 8.4 Hz, 1H, ArH), 8.30 (s, 1H, ArH), 8.11-8.13 (m, 2H, ArH), 7.83 – 7.75 (m, 3H, ArH), 7.62 – 7.55 (m, 1H, ArH), 7.29 (d, *J* = 8.0 Hz, 2H, ArH), 1.43 (s, 9H, O-C(CH$_3$)$_3$, Boc), 1.21 (s, 4H, -$\underline{CH_2}$-$\underline{CH_2}$-), ^{13}C NMR (100 MHz, DMSO-d_6): δ 155.9, 150.9, 147.5, 144.6, 138.2, 136.8, 136.7, 129.6, 128.9, 128.6, 127.1, 125.6, 125.4, 122.3, 78.2, 34.6, 28.7, 18.9, 18.6, HRMS: *m/z* calculated for $C_{23}H_{24}N_2O_2$: 361.4570; [M+H] $^+$ found: 361.4565**,** Rf=0.50, TLC System Hexane: EtOAc, 6:4, Physical appearance: pale yellow semisolid

1-Fluoro-2-nitro-4-(prop-1-en-2-yl) benzene (24) : ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.18 (m, 1H, ArH), 8.00-7.96 (m, 1H, ArH), 7.60 (m, 1H, ArH), 5.60 (s, 1H, =C$\underline{H}$), 5.28 (s, 1H, =C$\underline{H}$), 2.16 (s, 3H, -$\underline{CH_3}$)**,** ^{13}C NMR (100 MHz, DMSO-d_6): δ 152.9 ($^1J_{(C–F)=}$ 248.8 Hz), 140.2, 138.0, 137.3, 133.4, 122.8, 118.9, 115.6, 21.6, HRMS : *m/z* calculated for $C_9H_8FNO_2$: 182.1664; [M+H] $^+$ found: 181.9837, Rf=0.67, TLC System Hexane : EtOAc, 9 :1, Physical appearance: Yellow gum

3-Methyl-[1,1'-biphenyl]-4-carbonitrile (25) : Melting Point : 60-65 °C, ^{1}H NMR (400 MHz, DMSO-d_6): δ 7.91 – 7.81 (m, 2H, ArH), 7.78-7.70 (m, 3H, ArH), 7.56 – 7.52 (m, 2H, ArH),7.49-7.45 (m, 1H, ArH), 2.58 (s, 3H, Ar-$\underline{CH_3}$)**,** ^{13}C NMR (100 MHz, DMSO-d_6) : δ 144.9, 142.6, 138.8, 133.5, 129.5, 129.1, 128.9, 127.5, 125.2, 118.5, 110.9, 20.5, HRMS : *m/z* calculated for $C_{14}H_{11}N$: 194.2490; [M+H] $^+$ found: 194.2481**,** Rf=0.49, TLC System Hexane : EtOAc, 9.5:0.5, Physical appearance: White Solid

3-Methyl-1,1'-biphenyl (26): Analytical data in accordance with literature.[26]

4-Fluoro-3-nitro-1,1'-biphenyl (27): ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.37-8.34 (m, 1H, ArH), 8.14 -8.10(m, 1H, ArH), 7.78 – 7.73 (m, 2H, ArH), 7.69-7.67 (m, 1H, ArH), 7.53-7.49 (m, 3H, ArH), ^{13}C NMR (100 MHz, DMSO-d_6) : δ

155.7 ($^{1}J_{(C–F)=}$ 246.9 Hz), 137.7, 137.6, 134.7, 134.6 ($^{4}J_{(C–F)=}$ 30Hz, Para to F (C-1, biphenyl), 129.6, 128.9, 127.3, 124.2, 119.5, 119.3**,** HRMS : *m/z* calculated for $C_{12}H_8FNO_2$: 218.2000; [M+H] $^+$ found: 218.1981, Rf=0.51, TLC System Hexane : EtOAc, 9.5:0.5, Physical appearance: Yellow Liquid

2-Methoxy-5-phenylpyridine (28) : Analytical data in accordance with literature.[27]

Methyl 2-fluoro-[1,1'-biphenyl] -4-carboxylate (29) : Analytical data in accordance with literature.[28]

4'-Methoxy-3-methyl-[1,1'-biphenyl] -4-carbonitrile (30) : Analytical data in accordance with literature.[29]

4'-Methoxy-3-methyl-1,1'-biphenyl (31) : Analytical data in accordance with literature.[26]

4-Fluoro-4'-methoxy-3-nitro-1,1'-biphenyl (32): Melting Point: 72-73°C**,** ^{1}H NMR (400 MHz, DMSO-*d*$_6$): δ 8.31-8.29 (m, 1H, ArH), 8.09-8.05 (m, 1H, ArH), 7.74 – 7.60 (m, 3H, ArH), 7.06 (d, *J* = 8.8 Hz, 2H, ArH), 3.81 (s, 3H, -O$\underline{CH_3}$)**,** ^{13}C NMR (100 MHz, DMSO-*d*$_6$): δ 166.1, 155.2 ($^{1}J_{(C–F)=}$ 247 Hz), 152.6, 137.6, 137.4, 134.08, 134.0, 129.6, 128.5, 123.4, 119.4, 119.2, 115.0, 55.7, HRMS: *m/z* calculated for $C_{13}H_{10}FNO_3$: 248.2260; [M+H] $^+$ found: 248.2251, Rf=0.56, TLC System Hexane: EtOAc, 9:1, Physical appearance: Pale yellow Solid

2-Methoxy-5-(4-methoxyphenyl) pyridine (33) : Analytical data in accordance with literature.[29]

Methyl 2-fluoro-4'-methoxy-[1,1'-biphenyl]-4-carboxylate (34): Melting Point=80-85 °C, ^{1}H NMR (400 MHz, DMSO-*d*$_6$): 7.88 (d, *J* = 8.0 Hz, 1H, ArH), 7.80 (d, *J* = 8.0 Hz, 1H, ArH), 7.71 (t, *J* = 8.0 Hz, 1H, ArH), 7.60 (d, *J* = 8.3 Hz, 2H, ArH), 7.11 (d, *J* = 8.3 Hz, 2H, ArH), 3.91 (s, 3H, -O$\underline{CH_3}$), 3.84 (s, 3H, -OCO$\underline{CH_3}$)**,** ^{13}C NMR (100 MHz, DMSO-*d*$_6$): δ 165.5, 160.0, 157.8 ($^{1}J_{(C–F)=}$ 247 Hz), 133.0, 131.2, 131.2, 130.3, 130.2, 126.5, 125.9, 117.1, 116.9, 114.3, 55.6, 52.8, HRMS: *m/z* calculated for $C_{15}H_{13}FO_3$: 261.2644; [M+H] $^+$ found: 261.2623, Rf=0.61, TLC System Hexane: EtOAc, 8:2, Physical appearance: yellow Solid

4'-Acetyl-3-methyl-[1,1'-biphenyl]-4-carbonitrile (35): Melting Point: 109-111 °C, ^{1}H NMR (400 MHz, DMSO-*d*$_6$): δ 8.06 (d, *J* = 8.1 Hz, 2H, ArH), 7.89 (dd, *J* = 8.1, 4.1 Hz, 4H, ArH), 7.81 – 7.72 (m, 1H, ArH), 2.62 (s, 3H, -CO-$\underline{CH_3}$), 2.56 (s, 3H, Ar-$\underline{CH_3}$)**,** 13C NMR (100 MHz, DMSO-*d*$_6$): δ 197.9, 143.6, 143.0, 142.7, 136.9, 133.6, 129.3, 129.2, 127.7, 125.5, 118.3, 111.8, 27.2, 20.5, HRMS: *m/z* calculated for $C_{16}H_{13}NO$: 236.2860; [M+H] $^+$ found: 236.2689, Rf=0.51, TLC System Hexane: EtOAc, 8:2, Physical appearance: off white Solid

1-(3'-Methyl-[1,1'-biphenyl]-4-yl) ethan-1-one (36): Analytical data in accordance with literature.[30]

1-(4'-Fluoro-3'-nitro-[1,1'-biphenyl]-4-yl) ethan-1-one (37): Melting Point: 90-93 °C, ^{1}H NMR (400 MHz, DMSO-*d*$_6$): δ 8.44-8.42 (m, 1H, ArH), 8.21-8.17 (m, 1H, ArH), 8.05 (d, *J* = 8.0 Hz, 2H, ArH), 7.90 (d, *J* = 8.0 Hz, 2H, ArH), 7.74-7.69 (m, 1H, ArH), 2.61 (s, 3H, -CO-$\underline{CH_3}$)**,** ^{13}C NMR (100 MHz, DMSO-*d*$_6$): δ 197.9, 156.5, 153.6 ($^{1}J_{(C–F)=}$ 247 Hz), 141.5, 137.9, 136.8, 136.4, 135.0, 134.9, 129.4, 127.6, 124.7, 119.7, 119.5, 27.2, HRMS: *m/z* calculated for $C_{14}H_{10}FNO_3$: 260.2370; [M+H] $^+$ found: 260.2281, Rf=0.67, TLC System Hexane: EtOAc, 8:2**,** Physical appearance: yellow Solid

1-(4-(6-Methoxypyridin-3-yl) phenyl) ethan-1-one (38) : Analytical data in accordance with literature.[31]

Methyl 4'-acetyl-2-fluoro-[1,1'-biphenyl]-4-carboxylate (39): Melting Point: 88-90 °C, ^{1}H NMR (400 MHz, DMSO-*d*$_6$): δ 8.11 (d, *J* = 8.0 Hz, 2H, ArH), 7.94 (d, *J* = 11.3 Hz, 1H, ArH), 7.87 (d, *J* = 11.3 Hz, 1H, ArH), 7.82-7.79 (m, 3H, ArH), 3.93 (s, 3H, O-$\underline{CH_3}$), 2.66 (s, 3H, -CO-$\underline{CH_3}$)**,** ^{13}C NMR (100 MHz, DMSO-*d*$_6$): δ 197.9, 165.3, 160.4, 158.0 ($^{1}J_{(C–F)=}$ 249 Hz), 138.8, 136.7, 132.2, 131.7, 129.6, 129.6, 128.9, 126.1, 117.3, 117.0, 53.0, HRMS: *m/z* calculated for $C_{16}H_{13}FO_3$: 273.2754; [M+H] $^+$ found: 273.2736, Rf=0.62, TLC System Hexane: EtOAc, 9:1**,** Physical appearance: white solid

Tert-butyl 4-(4-cyano-3-methylphenyl)-3,6-dihydropyridine-1(2*H*)-carboxylate (40): Melting Point: 122-125 °C**,** ^{1}H NMR (400 MHz, DMSO-*d*$_6$): δ 7.75 (d, *J* = 8.1 Hz, 1H, ArH), 7.55 (s, 1H, ArH), 7.45 (d, *J* = 8.1 Hz, 1H, ArH), 6.47-6.27 (m, 1H Piperidine, =$\underline{CH}$), 4.14-3.95 (m, 2H), 3.75-3.45 (s, 2H,), 2.54 – 2.51 (m, 5H, Ar-$\underline{CH_3}$+ Piperidine $\underline{CH_2}$), 1.44 (s, 9H,-O-C(CH$_3$)$_3$, Boc), ^{1}H NMR (400 MHz, **MeOD-d$_4$**): δ 7.63 (d, *J* = 8.1 Hz, 1H, ArH), 7.50 (s, 1H, ArH), 7.43 (d, *J* = 8.1 Hz, 1H, ArH), 6.40-4.21 (m, 1H, Piperidine, =$\underline{CH}$), 4.18-4.10 (m, 2H), 3.77-3.57 (m, 2H), 2.65-2.45 (m, 5H, Ar-CH$_3$+ Piperidine CH$_2$), 1.52 (s, 9H, -O-C(CH$_3$)$_3$, Boc), ^{13}C NMR (100 MHz, DMSO-*d*$_6$): δ 154.2, 144.8, 142.0, 133.8, 133.0, 126.9, 124.8, 123.3, 118.5, 110.5, 79.6, 44.1, 28.5, 26.7, 20.4, HRMS: *m/z* calculated for $C_{18}H_{22}N_2O_2$: 299.3860; [M+H] $^+$ found: 299.3798, Rf=0.47, TLC System Hexane: EtOAc, 6:4, Physical appearance: off white solid.

Tert-butyl 4-(m-tolyl)-3,6-dihydropyridine-1(2*H*)-carboxylate (41) : Analytical data in accordance with literature.[32]

Tert-butyl 4-(4-fluoro-3-nitrophenyl)-3,6-dihydropyridine-1(2*H*)-carboxylate (42): ^{1}H NMR (400 MHz, MeOD-d$_4$) δ 8.14-8.12 (m, 1H, ArH), 7.85-7.83 (m, 1H, ArH), 7.44-7.40 (m, 1H, ArH), 6.38-6.16 (m, 1H, =CH, piperidine), 4.22-4.06 (m, 2H, ArH), 3.79-3.55 (m, 2H, ArH), 2.67-2.49 (m, 2H, ArH), 1.52 (s, 9H, -O-C(CH$_3$)$_3$, Boc), ^{13}C NMR (100 MHz, MeOD-d$_4$): δ 174.9, 155.2, 154.2, 137.7, 137.3, 132.8, 132.3, 122.2, 119.8, 118.8, 79.4, 34.1, 31.7, 29.0, 28.5, HRMS: *m/z* calculated for $C_{16}H_{19}FN_2O_4$: 323.3364; [M+H] $^+$ found: 323.3312**,** Rf=0.50, TLC System Hexane: EtOAc, 6:4, Physical appearance: yellow gum

Tert-butyl 6-methoxy-3',6'-dihydro-[3,4'-bipyridine]-1'(2'*H*)-carboxylate (43) : Analytical data in accordance with literature.[32]

Tert-butyl 4-(2-fluoro-4-(methoxycarbonyl) phenyl)-3,6-dihydropyridine-1(2*H*)-carboxylate (44): ^{1}H NMR (400 MHz, DMSO-*d*$_6$): 7.80 (d, *J* = 8.1 Hz, 1H, ArH), 7.71 (d, *J*= 8.1 Hz, 1H, ArH), 7.56 (t, *J* = 8.0 Hz, 1H, ArH), 6.06-5.91 (m, 1H, =CH, piperidine), 4.15-3.93 (m, 2H), 3.89 (s, 3H), 3.57-3.57 (m, 2H), 2.48-2.36 (m, 2H), 1.46 (s, 9H, -O-C(CH$_3$)$_3$, Boc), ^{13}C NMR (100 MHz, DMSO-*d*$_6$): δ 165.4, 158.0, 154.2, 138.2, 133.9, 130.7, 127.3, 125.7, 116.9, 79.4, 52.8, 28.5, HRMS: *m/z* calculated for $C_{18}H_{22}FNO_4$: 336.3754; [M+H] $^+$ found: 336.3741**,** Rf=0.51, TLC System Hexane: EtOAc, 6:4, Physical appearance: Colorless gum.

Ethyl (E)-3-(4-cyano-3-methylphenyl) acrylate (45) : Analytical data in accordance with literature.[33]

Ethyl (E)-3-(m-tolyl) acrylate (46) : Analytical data in accordance with literature.[34]

Ethyl (E)-3-(4-fluoro-3-nitrophenyl) acrylate (47) : Analytical data in accordance with literature.[35]

Ethyl (E)-3-(6-methoxypyridin-3-yl) acrylate (48): Melting Point: 115-118 °C**,** ^{1}H NMR (400 MHz, DMSO-*d*$_6$): δ 8.51 – 8.44 (m, 1H, ArH), 8.16 (d, *J*=8.6 Hz, 1H, ArH), 7.64 (d, *J* = 16.6 Hz, 1H, =CH), 6.87 (d, *J* = 8.6 Hz, 1H=CH), 6.61 (d, *J* = 16.6 Hz, 1H, =CH-CO), 4.19 (q, *J* = 7.0 Hz, 2H, -OCH$_2$-CH$_3$), 3.89 (s, 3H, -OCH$_3$), 1.25 (t, *J* = 7.0 Hz, 3H, -OCH$_2$-CH$_3$), ^{13}C NMR (100 MHz, DMSO-*d*$_6$): δ 166.6, 165.1, 149.1, 141.4, 137.7, 124.2, 117.5, 111.5, 60.4, 53.9, 14.6**,** HRMS: *m/z* calculated for $C_{11}H_{13}NO_3$: 208.2290; [M+H] $^+$ found: 208.2281, Rf=0.31, TLC System Hexane: EtOAc, 9:1, Physical appearance: white solid.

Methyl (E)-4-(3-ethoxy-3-oxoprop-1-en-1-yl)-3-fluorobenzoate (49) : Analytical data in accordance with literature.35

1-(4'-Methoxy-[1,1'-biphenyl] -4-yl) ethan-1-one (50) : Analytical data in accordance with literature.[27]

4-Methoxy-1,1'-biphenyl (51) : Analytical data in accordance with literature.[30]

Ethyl (Z)-3-(4-methoxyphenyl) but-2-enoate (52) : Analytical data in accordance with literature.[36]

4,4'-Dimethoxy-1,1'-biphenyl (53) : Analytical data in accordance with literature.[37]

4,4''-Dimethoxy-1,1':4',1''-terphenyl (54) : Analytical data in accordance with literature.[38]

1-(4'-Nitro-[1,1'-biphenyl]-4-yl) ethan-1-one (55) : Analytical data in accordance with literature.[27]

1-(4-(Naphthalen-2-yl) phenyl) ethan-1-one (56) : Analytical data in accordance with literature.[39]

4-Methoxy-4'-nitro-1,1'-biphenyl (57) : Analytical data in accordance with literature.[40]

4-Nitro-1,1'-biphenyl (58) : Analytical data in accordance with literature.[26]

1,1'-([1,1' :4',1''-Terphenyl] -4,4''-diyl) bis(ethan-1-one) (59) : Analytical data in accordance with literature.[27]

Ethyl (Z)-3-(naphthalen-2-yl) but-2-enoate (60) : Analytical data in accordance with literature.[36]

2-(4-Methoxyphenyl) naphthalene (61) : Analytical data in accordance with literature.[41]

Ethyl (Z)-3-(4-((E)-4-ethoxy-4-oxobut-2-en-2-yl) phenyl) but-2-enoate (62) : Analytical data in accordance with literature.[42]

Ethyl (Z)-3-(4-nitrophenyl) but-2-enoate (63) : Analytical data in accordance with literature.[42]

1-(4-(Thiophen-2-yl) phenyl) ethan-1-one (64) : Analytical data in accordance with literature.[43]

2-Phenylthiophene (65) : Analytical data in accordance with literature.[27]

2-(4-Methoxyphenyl) thiophene (66) : Analytical data in accordance with literature.[29]

Ethyl (Z)-3-(thiophen-2-yl) but-2-enoate (67) : Analytical data in accordance with literature.[36]

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Supplementary Material

Supplementary materials include spectral analytical data for synthesized novel compounds

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