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Review article

Recent advances in β-cyclodextrin-based catalyst systems for the synthesis of heterocyclic compounds via multicomponent reactions (MCRs)

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ABSTRACT

Cyclodextrins (CDs) are essential compounds because of their wide applications in many fields. They are cyclic oligosaccharides with lipophilic internal cavities and hydrophilic external surfaces that link the α-D-glucopyranose portion. β-Cyclodextrin (β-CD) is a non-toxicity, cheap, green, and renewability macrocycle with excellent performance in organic transformation. β-CD is a green catalyst with satisfactory catalytic activity in diverse organic changes. Regarding green chemistry's goals, β-CD opens the way to effective catalysts for various reactions. Heterocyclic compounds are among the most prominent organic compounds in numerous organic materials, pharmaceuticals, and natural products. Heterocyclic compounds are essential in pharmaceutical products and show different biological activities in multiple illnesses. Thus, there is a tendency to develop novel, green, and helpful syntheses of heterocyclic systems, which has been a big challenge in synthetic organic chemistry. Using β-CD as catalysts for synthesizing heterocyclic compounds makes the procedure milder, easier, and less toxic, making it an eco-friendly substitute compared to the reported approaches. This review underlines the applications of β-CD-based catalysts for synthesizing heterocyclic compounds covering 2019–2023. This study will be helpful to researchers in the investigation areas of organic synthesis, medicinal chemistry, synthetic materials, and pharmacological medicine..

1. Introduction

The expansion of green and sustainable paths for the fabrication of chemical production is a fundamental target in modern organic synthesis. The striking plan for organic synthesis is to extend methods by exerting only green materials without creating chemical wastage. In this regard, reducing organic pollutants and developing new green catalysts are essential goals of green chemistry. One of the fast-developing fields of research related to expanding green catalysts for chemical conversions) Abdussalam-Mohammed et al., 2020; Anastas et al., 2002; Nozad et al., 2022; Mohammadi Dehcheshmeh et al., 2021; Zamani et al., 2019; Gozali Balkanloo et al., 2023).

β-CDs have drawn much notable among the various supramolecular catalysts due to their green biomimetic materials and unique nature. They are a well-known class of polysaccharides obtained from starch degradation using enzymatic conversion. These cyclic oligosaccharides have a hydrophilic outer surface and hydrophobic central cavity (Scheme 1) (Crini, 2014; Szejtli, 1998). They can make inclusion complexes with a wide range of guest compounds by including them in the cavity without forming covalent bonds (Fig. 1). The size of the central

Abbreviations: CD, Cyclodextrin; *β*-CD, β-Cyclodextrin; NPs, Nanoparticles; MCRs, Multicomponent reactions; TCR, Three-component response; BA, Barbituric acid; [β-CD/Im](OTs)$_2$, β-Cyclodextrin/imidazolium-based dicationic ionic liquid; Ils, Ionic liquids; SPIONs, Iron oxide nanoparticles; M-Starch, Magnetic starch; ChCl, Choline chloride; *p*-TSA, *p*-Toluenesulfonic acid; Er(OTF)$_3$, Erbium(III) trifluoromethanesulfonate; DABCO, 1,4-Diazabicyclo[2,2,2]octane; SDS, Sodium dodecyl sulfate; NWs, Nanowires; OMWCNTs, Oxidized multi-walled carbon nanotubes; SB-DBU, SB-DBU Cl, Silica-bonded 5-n-propyl-octahydro-pyrimido[1,2-*a*] azepinium chloride; SSA-MNPs, Silica sulfuric acid magnetic nanoparticle; PEI@Si–MNPs, Polyethyleneimine-modified superparamagnetic Fe_3O_4 NPs; CAN, Ceric ammonium nitrate; SO_3H, Sulfonic acid; MNPTC, Magnetic nano phase transfer catalyst; OPD, *o*-Phenylenediamine; CIT, Citric acid; β-CD.NS, β-Cyclodextrin-based nanosponges; MCM-41, Mobil Composition of Matter No. 41; GA, Guanidine; Met, Metformin; GO, Graphene oxide; β-CDH, β-CD hydrate; MNPs, Magnetic nanoparticles; VSM, Vibrating-sample magnetometer; TEM, Transmission electron microscopy; SEM, Scanning electron microscopy; TGA, Thermogravimetric Analysis; EDX, Electron-dispersive X-ray analysis; XRD, X-ray diffraction analysis; FT-IR, Fourier Transform Infrared; ^{13}C NMR, Carbon Nuclear Magnetic Resonance; ^{1}H NMR, Proton Nuclear Magnetic Resonance.

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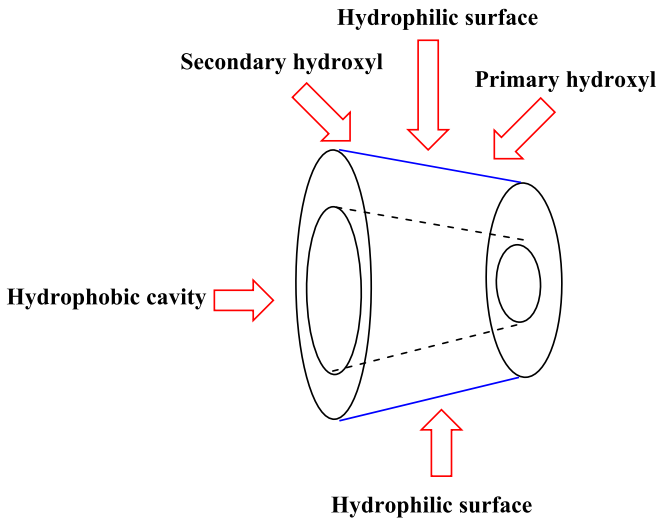


Scheme 1. Structure representation of native β-CD.

cavity plays a significant role in creating inclusion complexes (Khan et al.,1998; Engeldinger et al., 2003; Kfoury et al., 2018). CDs are somewhat low-price, non-toxic, biodegradable, and available, so developing catalysts based on these supramolecular oligosaccharides is highly advantageous. Modified CDs present many perfect opportunities and challenges for the chemistry community (Kfoury and Fourmentin, 2023, R Rao et al., 2010). Many reports have been published on using CDs in fields like pharmaceutical chemistry (Puskás et al., 2023), analytical chemistries (Cid-Samamed et al., 2022), and catalysis (Noël et al.,2021; Kanchana et al., 2020; Payamifar and Poursattar Marjani, 2023a, 2023b; Hapiot et al., 2017; Payamifar and Poursattar Marjani, 2024; Hapiot et al., 2017; Payamifar et al., 2021) during the past years indicate the interest of scientists in this field because it has outstanding properties. Developing a new and proper process for preparing β-CD-based catalysts with high catalytic performance, reusability, and simple workup is desirable. Recently, β-CDs and their derivatives as practical catalysts have drawn attention to synthesizing heterocycle compounds owing to their economical and straightforward accessibility (Mishra et al., 2018; Abbasi, 2017).

The progress of various chemicals of fascinating drug-like molecules utilizing multicomponent reactions (MCRs) permits high combinatorial

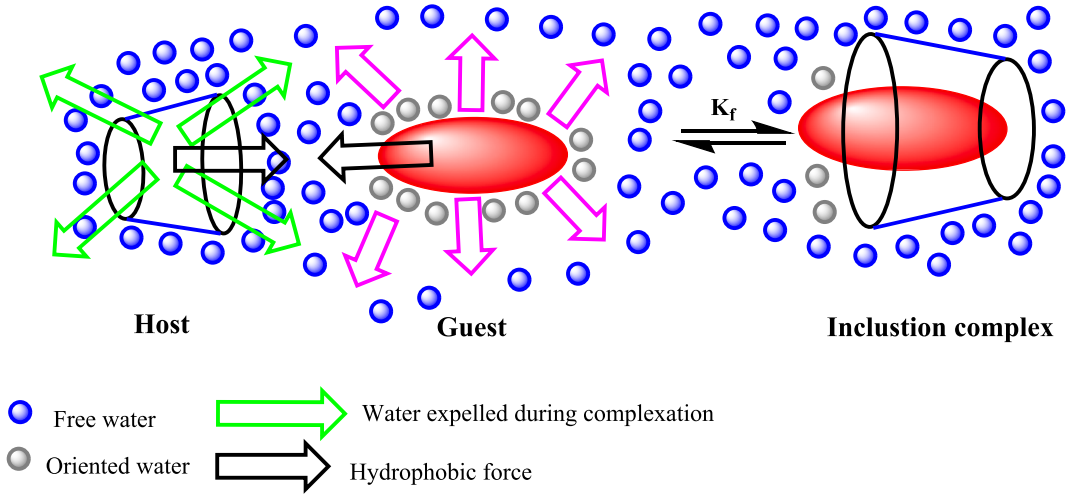
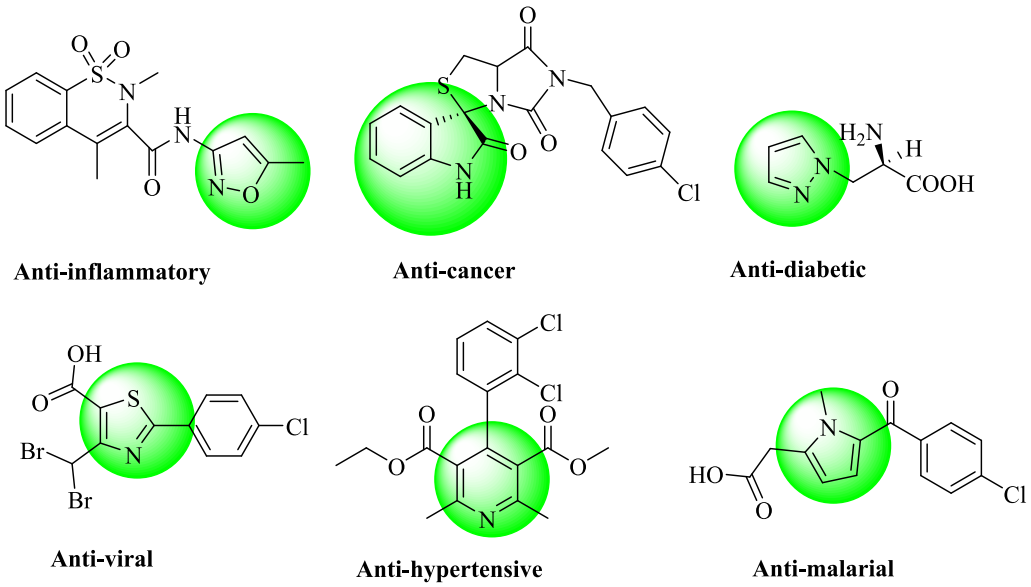
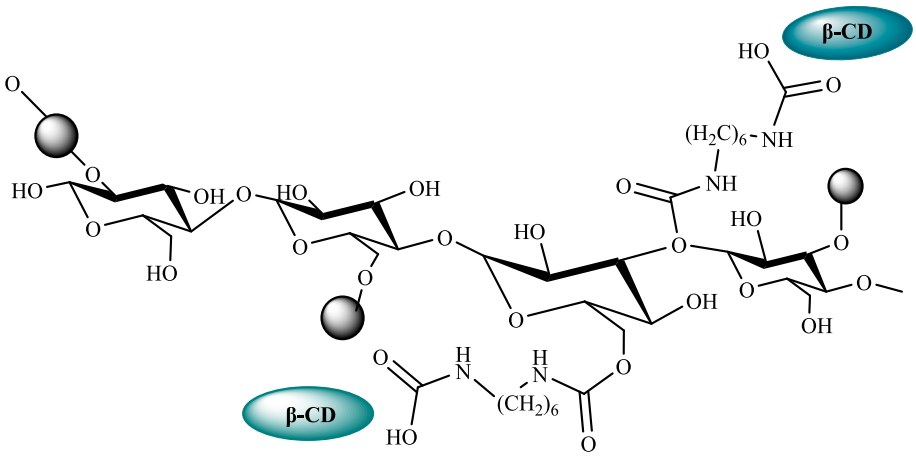


Fig. 1. The construction of an inclusion complex between β-CD and a guest molecular.



Scheme 2. Some structures of the biologically active fused heterocycles.



Scheme 3. The structure of β-CD-IL@M starch.

usefulness in making diversity. The conventional route for preparing complicated molecules was contrary to MCRs, allowing the complex molecules to lead to products in one pot. The role of MCRs is extensive in all areas of the chemistry world because they offer many products with the lowest effort in price- and time-effective methods. MCRs have been employed to prepare an extensive scope of various categories of heterocyclic compounds presenting various biologically active and medical applications. Some organic compounds that transform more than two components into their products *via* one-pot reactions are created by MCRs. This approach has advantages such as simplicity, economics, efficiency, and eco-friendliness compared to conventional chemical reactions (Cioc et al., 2014; Ugi, 2001; Meng et al., 2014; Younus et al., 2021; Zhi et al., 2019). The history of heterocyclic chemistry began in the 1800s, coinciding with the development of organic chemistry. The formation of heterocycle compounds is the most important reaction and is crucial to organic chemistry. They have one or more hetero atoms in their structure. They can be cyclic or noncyclic, vastly expanded in nature, and essential to our daily lives with an expansive scope of applications (Joule, 2020; Cabrele and Reiser, 2016; Jacobi, 2018; Bibak and Poursattar Marjani, 2023; Bikas et al., 2023; Payamifar et al., 2024a; 2024b; Khashaei et al., 2022; Bibak et al., 2024). They are found in pharmaceuticals (Kabir and Uzzaman, 2022), agrochemicals (Sanemitsu and Kawamura, 2008), and veterinary products. Also, they have applications such as sensitizers (AL-Adilee et al., 2016), anti-oxidants (Tsolaki et al., 2014), and corrosion inhibitors (Goni et al., 2021). They are employed as tools in the synthesis of other organic compounds.

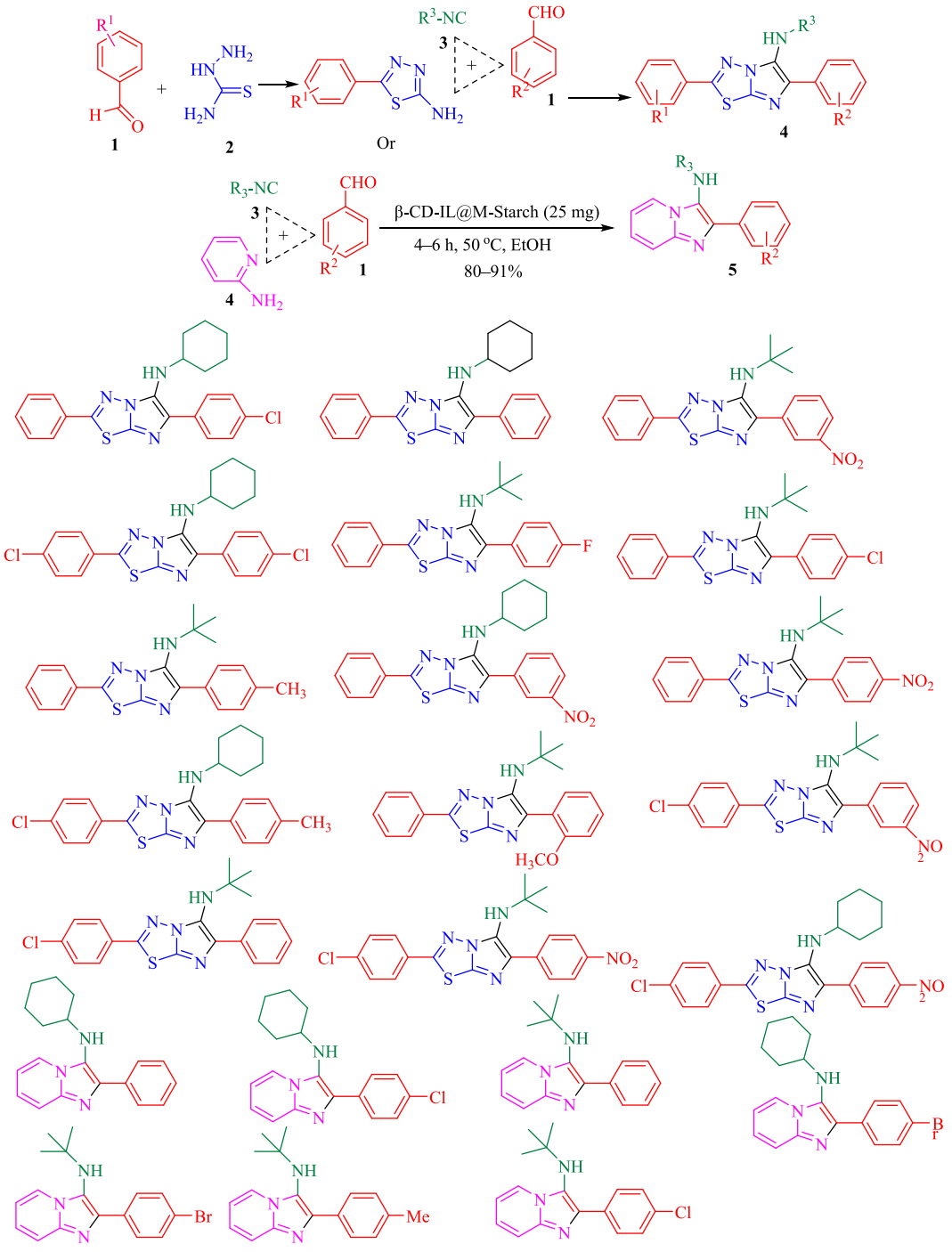
These excellent biological properties make heterocyclic compounds a suitable candidate for developing the pharmaceuticals field. They display an expansive range of biological activities, namely anti-cancer (Ali et al., 2015), anti-viral (De et al., 2021), anti-bacterial (Azab et al., 2013), anti-inflammatory (Sharma et al., 2020), anti-diabetic (Singh, 2022), and anti-tumor (El-Hag et al., 2019). Several heterocyclic compounds have been shown to have biological activity (Scheme 2).

The preparation of myriad heterocycles was published *via* MCR. Still, these works do not deliver an acceptable result regarding reaction factors like pricey transition metal catalysts, long reaction times, and halogenated solvents. Given these drawbacks, diverse new procedures were regarded about environmental safety. Developing green and improved ways of synthesizing heterocycles remains a challenging objective. Expanding a profoundly efficient catalytic system is highly preferred with the rising need for practical organic conversion based on green sustainability in chemistry (Daştan et al., 2012; Safari et al., 2023; Nishanth Rao et al., 2021; Adhikari et al., 2022; Ameta and Dandia, 2014; Chaudhuri et al., 2021). Recently, β-CD, a powerful catalytic system, received significant attention for synthesizing heterocycles. β-CDs have been broadly employed as a valuable catalyst for synthesizing heterocycles that make many products (Abbasi, 2017; Cioc et al., 2014; Mishra et al., 2018). As mentioned earlier, the critical characteristic of CD compounds is that they can organize the inclusion of complex metal ions for hosting in their cavity with non-covalent bonding. This feature makes them water-soluble catalysts with high stability in water. Along this line, CD catalysts effectively conduct chemical transformations in aqueous media. CDs are gaining attention in greener synthetic routes due to their reactions being performed in neat, aqueous organic media, and it will inspire chemists to design and develop new heterocyclic compounds by using CDs as a perfect catalyst. This review highlights the advances in synthesizing heterocyclic compounds based on β-CD catalysts. We hope this discussion will be necessary and draw more interest in various research fields.

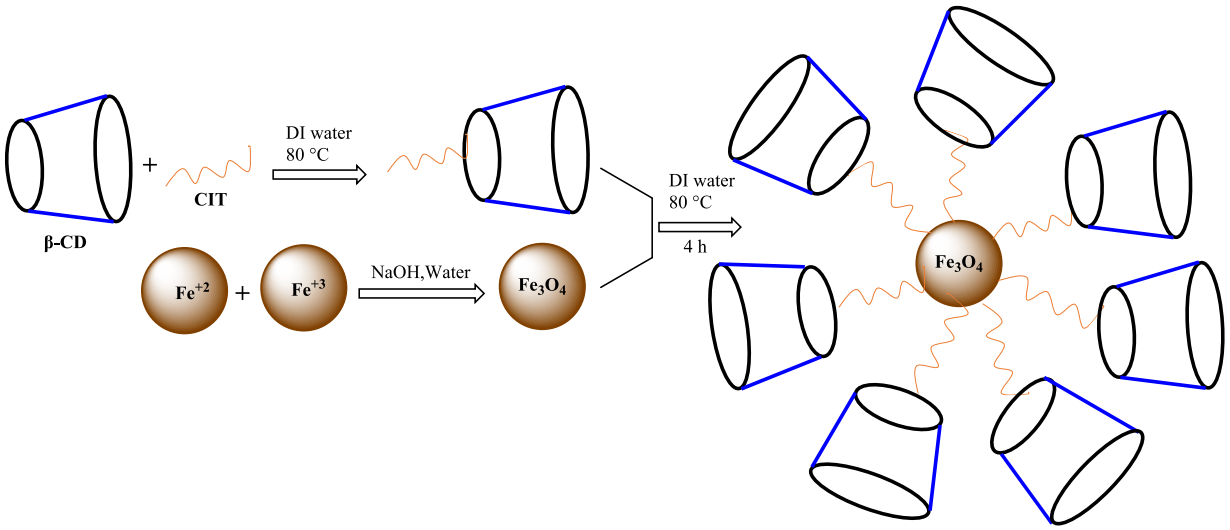
2. β-CD catalysts for synthesizing heterocycles

2.1. Synthesis of five-membered heterocycles

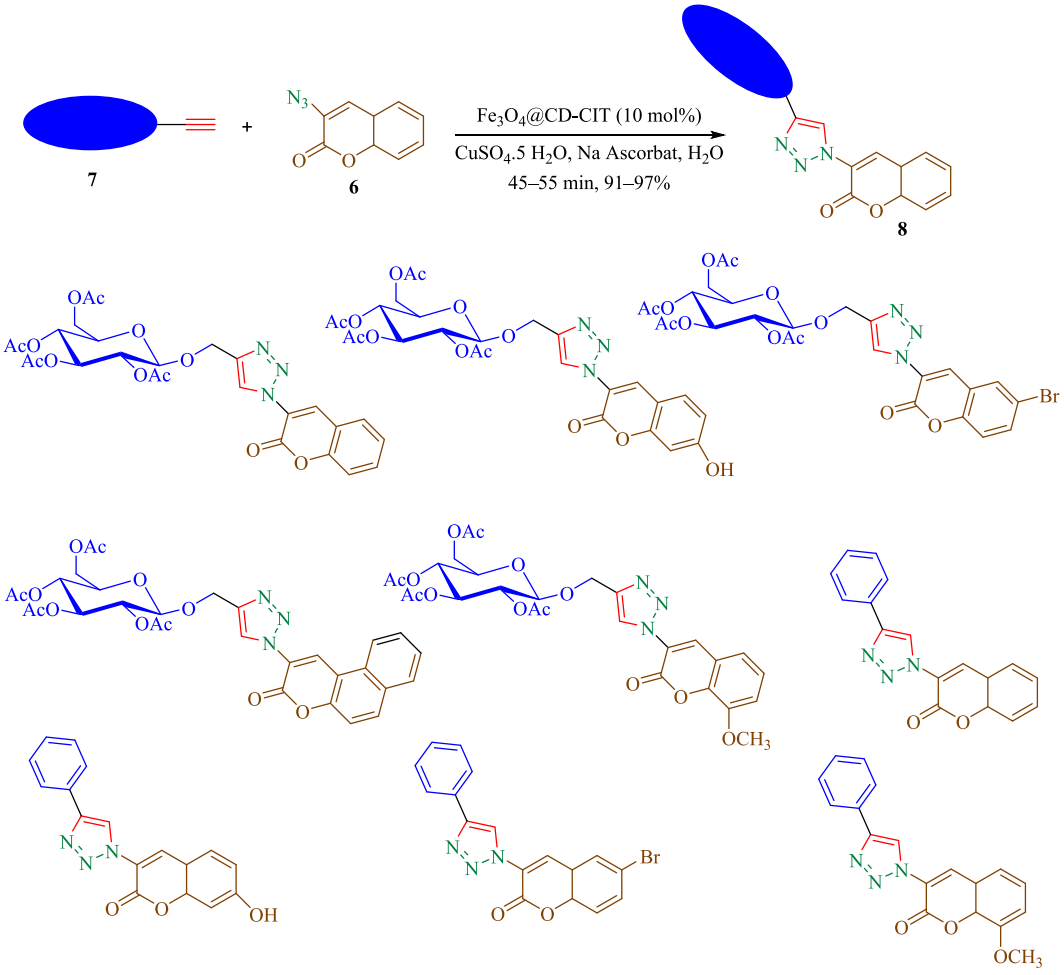
In 2019, Bahadorikhalili *et al.* reported a green procedure for the fabrication of imidazo[2,1-*b*][1,3,4]thiadiazol-5-amines **4** and imidazo[1,2-*a*]pyridines **5** from the corresponding benzaldehyde (**1**), semicarbazide (**2**), and isocyanides (**3**) using βCD-IL@MStarch in ethanol as an environmentally friendly solvent at 50 °C (Scheme 4) (Bahadorikhalili et al., 2019). Iron oxide nanoparticles (SPIONs) were obtained using co-precipitation of Fe^{3+} and Fe^{2+} ions for the preparation catalyst. Then, they were modified by starch (M−Starch). Separately, β-CD-IL was synthesized in two stages. Initially, a tosylation is the reaction of the tosylate β-CD by methyl imidazole. The polymerization of β-CD-IL with hexamethylene diisocyanate on M−Starch was obtained from the final β-CD-IL@M−Starch catalyst (Scheme 3). This new catalyst was identified using TGA, TEM, VSM, and FTIR techniques. In the TEM photo of this magnetic nanocatalyst, the dark spots represent the



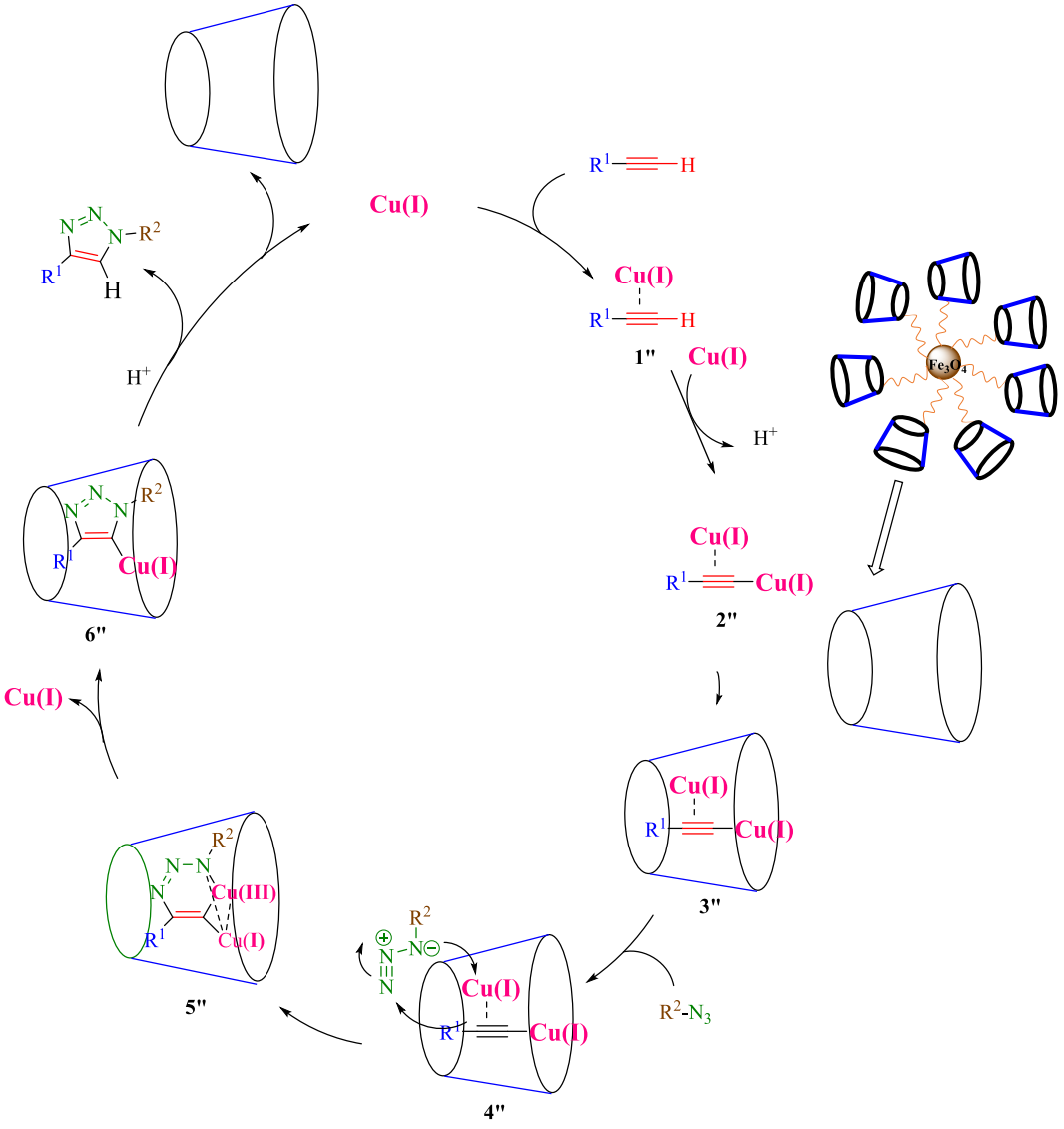
Scheme 4. Synthesis of imidazothiadiazolamine and imidazopyridinamine derivatives.



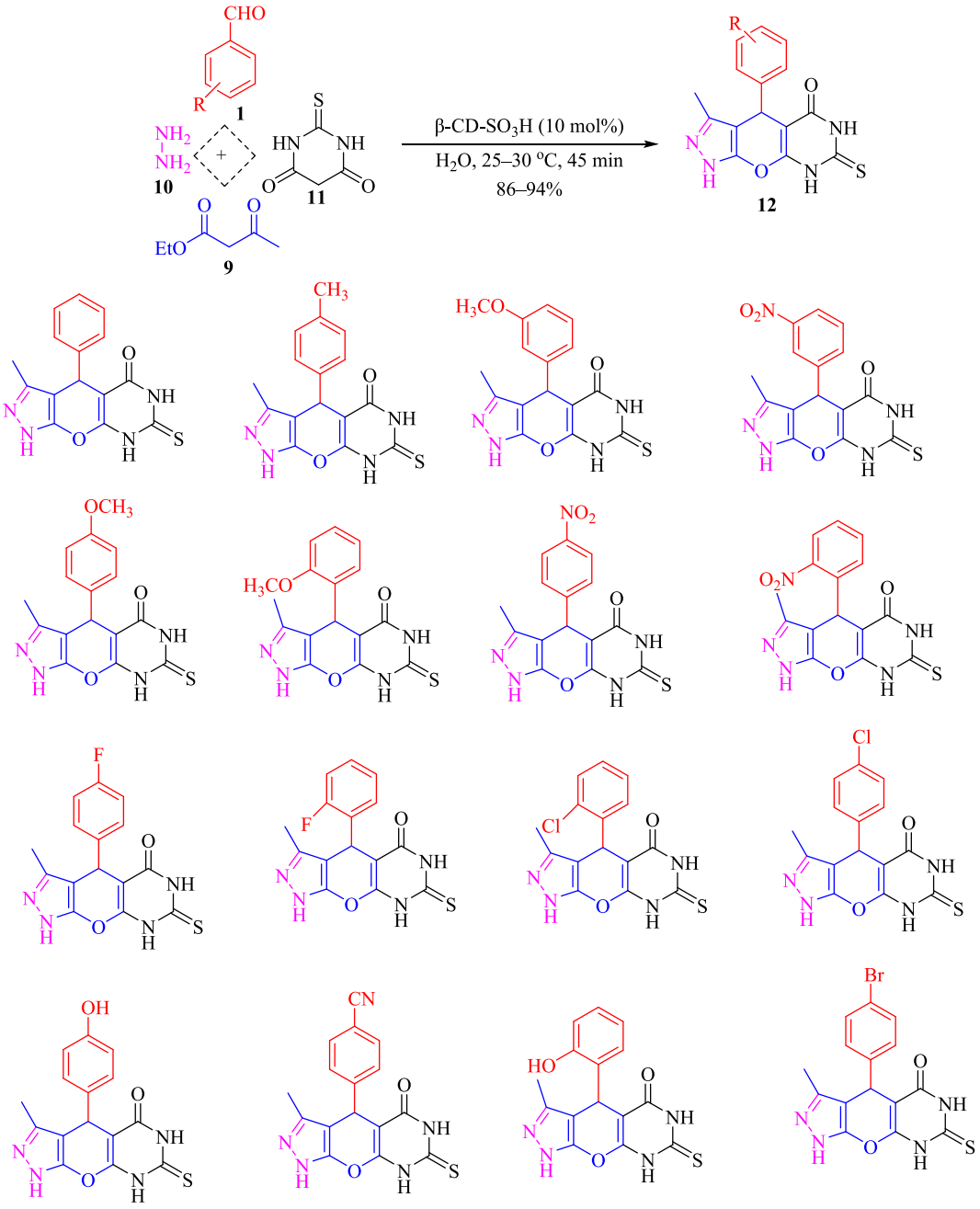
Scheme 5. The provision of Fe_3O_4@CD-CIT.



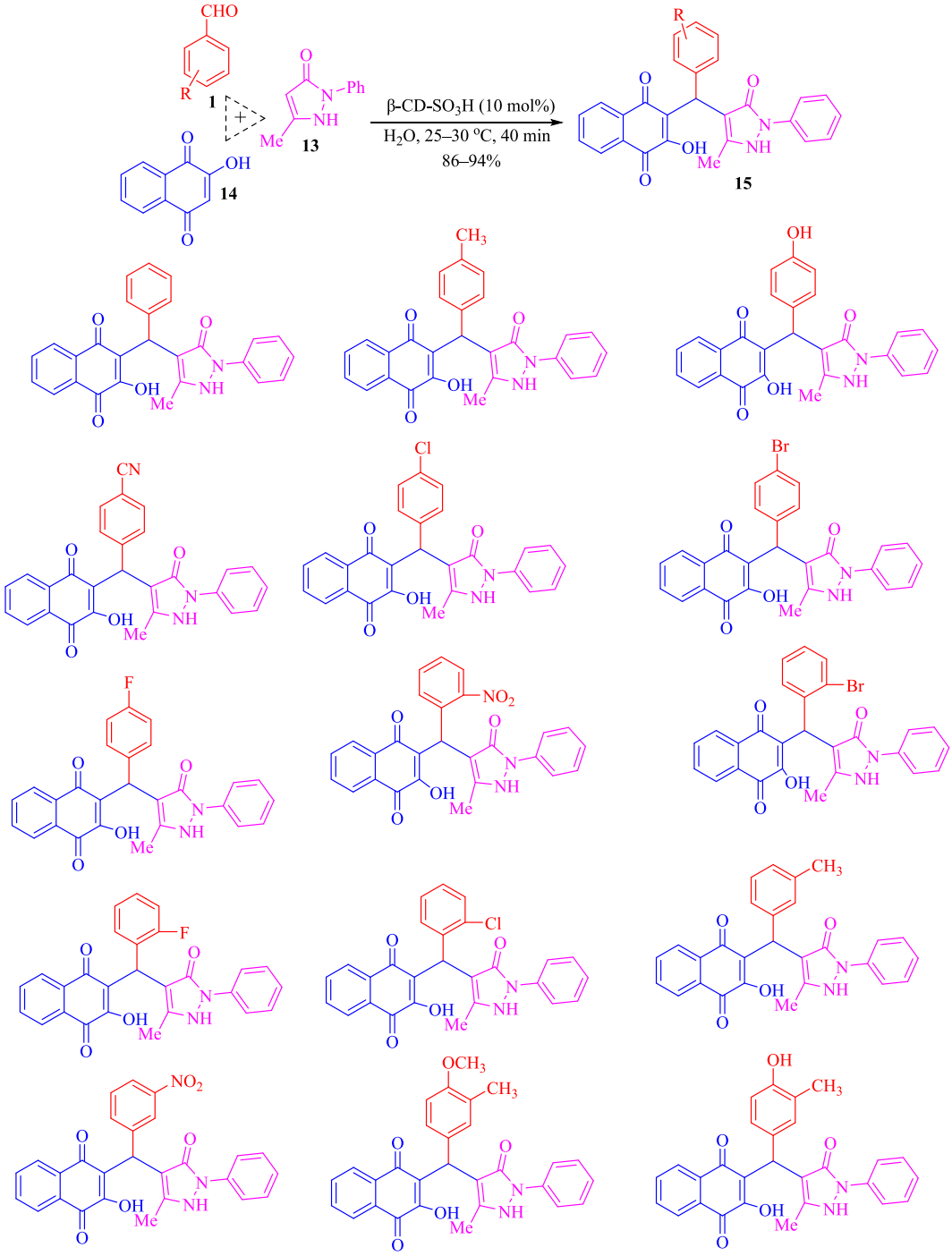
Scheme 6. The preparation of 1,2,3- triazoles derivatives by Fe_3O_4@CD-CIT.



Scheme 7. Mechanism for the preparation of 1,2,3- triazoles derivatives by Fe_3O_4@CD-CIT.



Scheme 8. Synthesis of pyrazolopyranopyrimidines **12**.



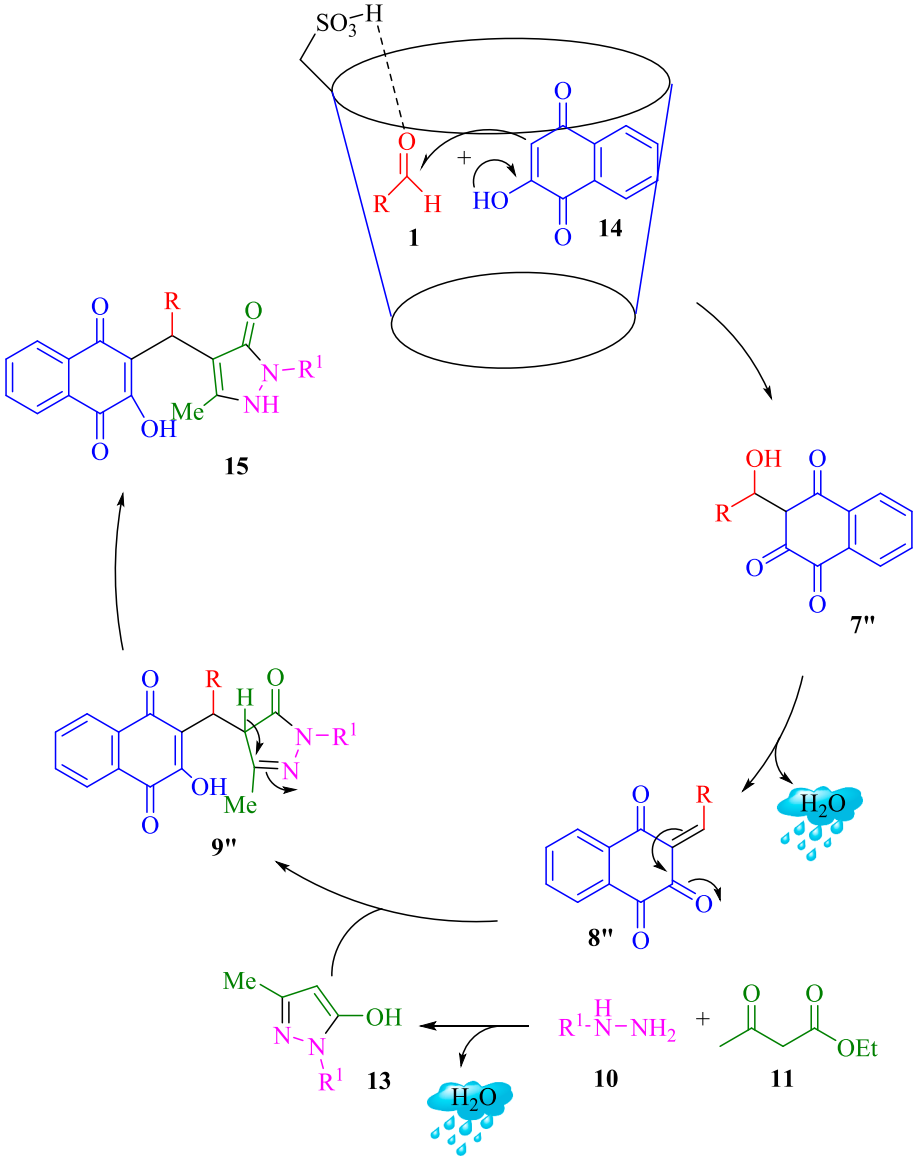
Scheme 9. Synthesis of benzylpyrazolyl naphthoquinones.

Table 1
A comparison of the catalytic efficiency of various catalysts was reported in the literature.

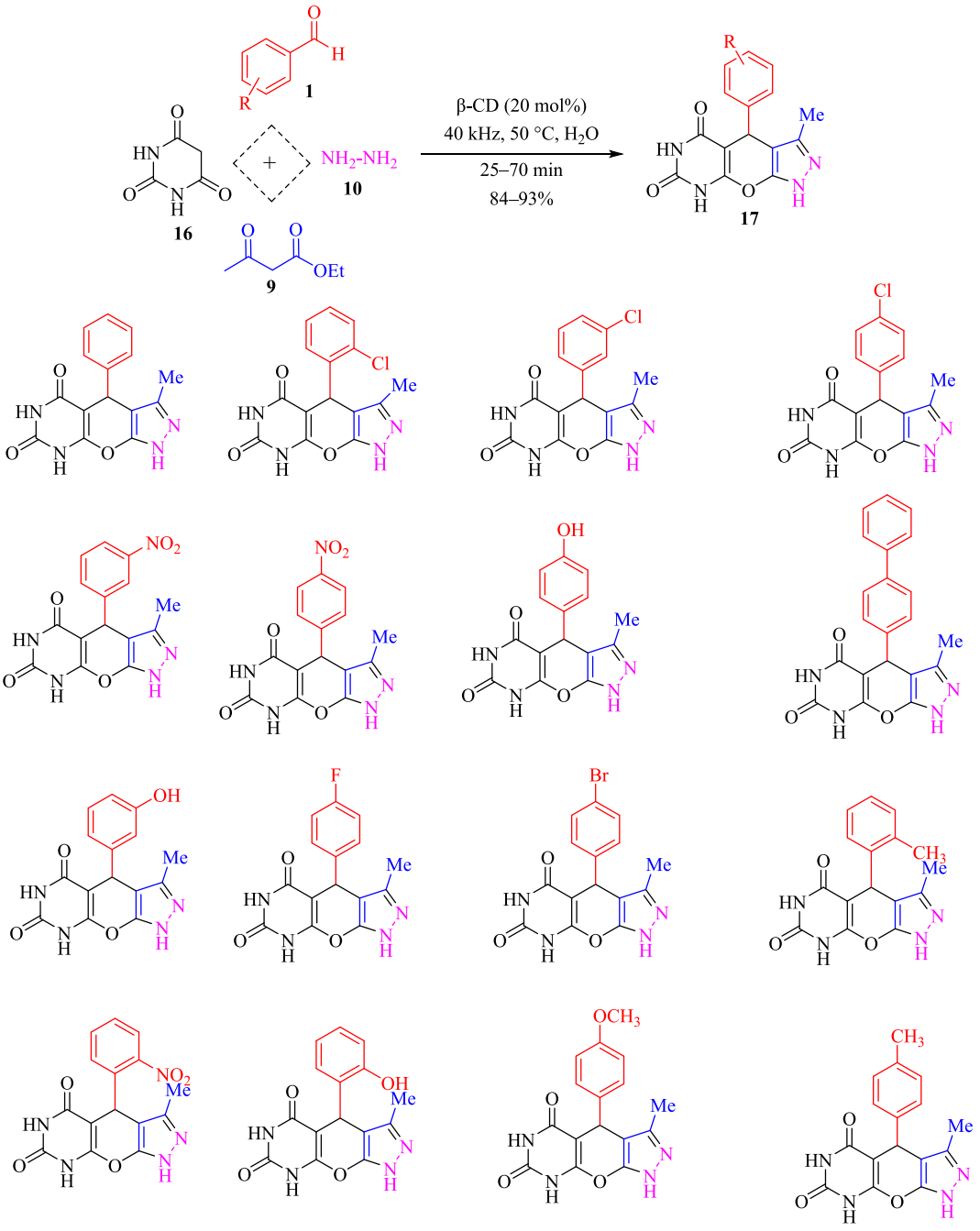
Entry	Catalyst	Solvent	Condition	Time (min)	Yield (%)	Reference
1	$Er(OTF)_3$	EtOH	Reflux	2 h	88–96	Kumar et al., 2017
2	*p*-TSA	H_2O	Reflux	24	75–84	Lakshmanan and Ramalakshmi, 2016
3	$MgCl_2$	EtOH	100 ℃	20–60	80–90	Fu et al., 2016
4	–	H_2O	MW, 120 ℃	7–9	70–90	Wang et al., 2012
5	β-CD-SO_3H	H_2O	Rt	40	88–94	Current discussed report

existence of Fe_3O_4 nanoparticles. The brighter parts correspond to the organic groups, including starch, ionic liquid β-CD, and the polymer linker. The magnetic behavior of the nanocatalyst was examined using the VSM analysis so that the superparamagnetic behavior of the catalyst could be confirmed. This catalyst was recycled successfully for ten consecutive runs. The imidazothiadiazolamine derivatives were attained in good isolated yields (80–91 %). Gadad *et al.* reported the synthesis of imidazothiadiazolamine derivatives by using MCRs. Reactions of 5-substituted-1,3,4-thiadiazol-2-amine with aldehydes and isocyanides in the presence of trimethylsilyl chloride or perchloric acid in dry ethanol at reflux for 14 h. are reported (Gadad et al., 2008). Compared with this method, employing β-CD-IL@M starch catalyst has some advantages like mild reaction conditions, easy reaction procedure, and high isolated yields.

Jain *et al.* declared a helpful route for the formation of 1,4-disubstituted 1,2,3-triazoles **8** from coumarin azides (**6**), terminal alkyl **7** *via* click reaction by using 2.5 mol% of Fe_3O_4@CD-CIT as a phase-transfer catalyst by lower Cu loading in H_2O under ultrasonic irradiation



Scheme 10. Plausible reaction mechanism.



Scheme 11. The preparation of pyrazolopyranopyrimidines by β-CD as a catalyst under ultrasound conditions.

Table 2
A comparison of the catalytic efficiency of different catalysts is presented in the literature.

Entry	Catalyst and solvent	Temperature	Time (min)	Yield (%)	References
1	DABCO, H_2O	Reflux	20–45	84–99	Heravi et al., 2014
2	SDS, H_2O	Reflux	40	87–96	Ahanthem et al., 2018
3	TiO_2 NWs, EtOH/H_2O	Reflux	45–100	83–95	Dastkhoon et al., 2015
4	OMWCNTs, EtOH/H_2O	Reflux	65–100	85–94	Khodabakhshi et al., 2016
5	Meglumine, H_2O	rt	15–360	83–95	Li et al., 2014
6	Cu^{2+}@MSNs-$(CO_2^-)_2$, H_2O	rt	60–120	75–92	Nasresfahani and Kassaee, 2017
7	ChCl:Urea, EtOH	80 ℃	60	75–92	Tipale et al., 2018
8	β-CD, H_2O	50 ℃	25–70	84–93	Current discussed report

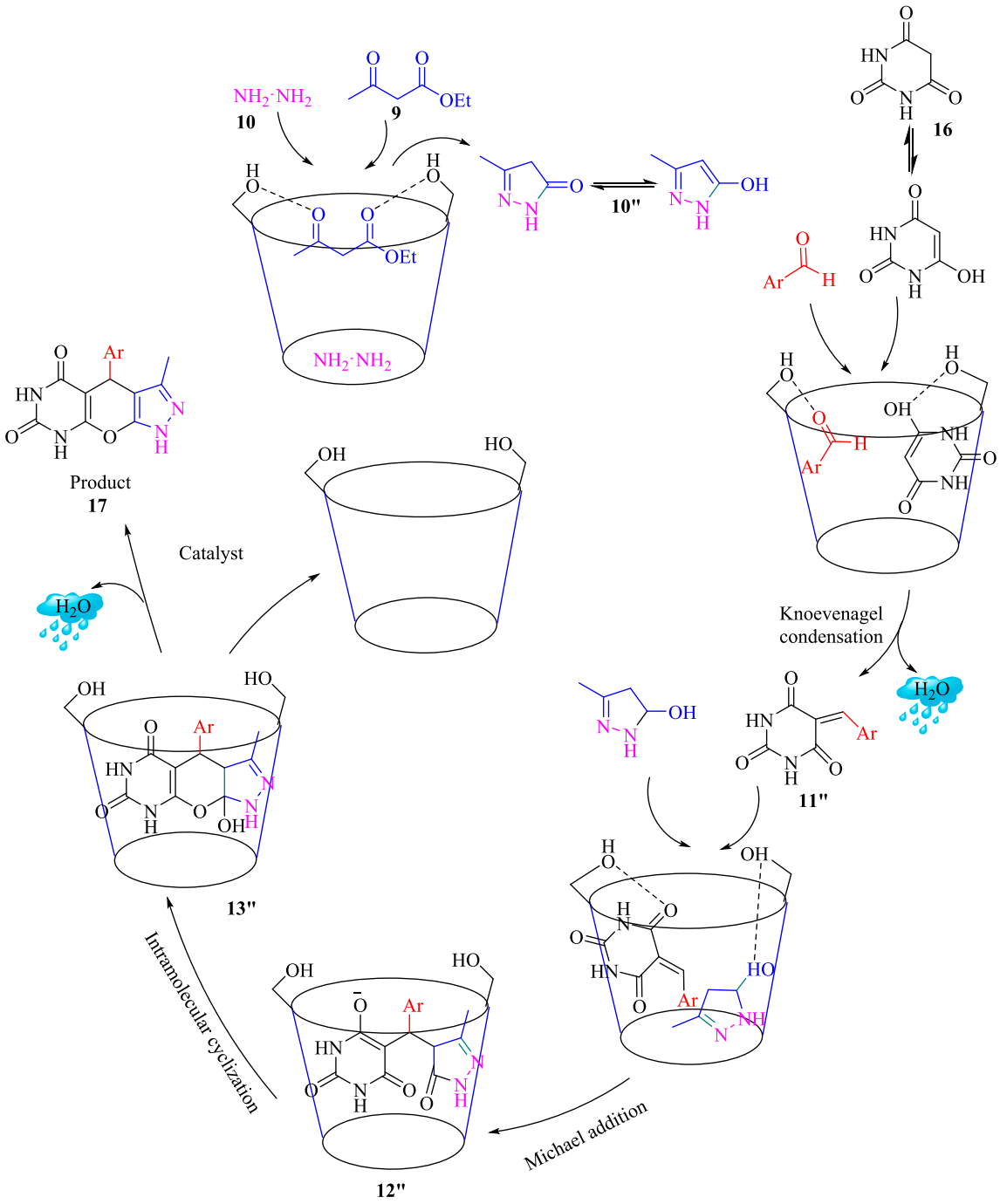
conditions at 40 °C (Scheme 6) (Jain et al., 2019). The steps of catalyst preparation are shown in Scheme 5. Diverse techniques, including FTIR, XRD, ^{1}H NMR, TEM, and VSM, identified the new magnetic compound Fe_3O_4@CD-CIT. TEM image indicated that particles were nearly spherical with a range of 5–10 nm. This reaction performed well, providing 1,4-disubstituted-1,2,3-triazoles were good to superior yields (91–97 %). Fe_3O_4@CD-CIT could be recycled by magnetic separation for 6 consecutive runs with a tiny reduction in performance. The SEM result of the recycled catalyst after the 7 times indicated no notable difference in the SEM photos of the fresh and the recovered catalyst, which furnished proof for the satisfactory structural stability of the catalyst under the utilized reaction conditions. In the same ultrasound irradiation method, Jiang *et al.* reported the synthesis of 1,4-disubstituted-1,2,3-triazoles derivatives by using 1,3-dipolar cycloaddition reaction employing $CuSO_4{\cdot}5H_2O$ (10 mol%) and sodium ascorbate (20 mol%) as the catalyst in t-BuOH/H_2O as reaction solvents and at room temperature (Jiang et al., 2011). Compared to the mentioned report, this method has some benefits like short-time reaction, recyclability, Gram scale synthesis, and high yield. The probable reaction mechanism for click reaction has been proposed in Scheme 7. At first, Cu(I) coordinated acetylide complexes created through the interaction of terminal alkyne and Cu(I) species (Complex **1**″). The host–guest complex is constructed by β-CD of MNPTC (Complex 3″) in the next step. The formed complex reacts with coumarin azide, creating complex **4**″, which converts into an intermediate **5**″. Then, complex E intermediate provides heterocyclic complex **6**″, which undergoes protonolysis and gets the related 1,2,3-triazole derivatives.

In 2020, Patil *et al.* represented an eco-friendly procedure for the synthesis of benzyl pyrazolyl naphthoquinones **15** (Scheme 9) and pyrazolopyranopyrimidines **12** (Scheme 8) by β-CD-SO_3H as a heterogeneous reusable catalyst in H_2O at 25–30 °C (Patil et al., 2020). β-CD-SO_3H was synthesized by a simple one-step process and then characterized by several analyses like TGA, FT-IR, XRD, ^{13}C NMR, ^{1}H NMR, EDAX, BET, and acid-base titration. The EDAX analysis of β-CD-SO_3H showed carbon and oxygen as the main elements related to the β-CD scaffolding. In contrast, the sulfur peak in its respective energy position at 2.2–2.4 keV also proved the construction of the expected catalyst. The loading of the SO_3H group was noticed to be 0.8625 mmol of the functional group per gram of catalyst. The results indicated the proper usage of β-CD-SO_3H and H_2O to prepare pyrazolo pyranopyrimidines (86–94 %). Compared to reported methods, this approach showed better results (Table 1). Simplicity, mild and fast way, a green solvent, an easy work-up, and an inexpensive catalyst cost are the main advantages of this procedure. They also suggested the mechanism of the reaction (Scheme 10). Initially, aryl hydrazine/hydrazine hydrate **10** is reacted with ethyl acetoacetate (**11**) to generate the pyrazolone ring **13**, an isomeric form. Simultaneously, the electrophilicity of carbonyl carbon of aldehyde 1 increases owing to the hydrogen bonding of β-CD-SO_3H. Nucleophilic attack of 2-hydroxy-1,4-naphthoquinone **14** to activated aldehyde leads to the creation of intermediate **7**″, followed by Knoevenagel condensation to construct intermediate **8**″. Then, Michael added intermediate **13** and unsaturated Knoevenagel product **8** to form intermediate **9**″, which undergoes a tautomeric proton shift to generate the desired product 15.

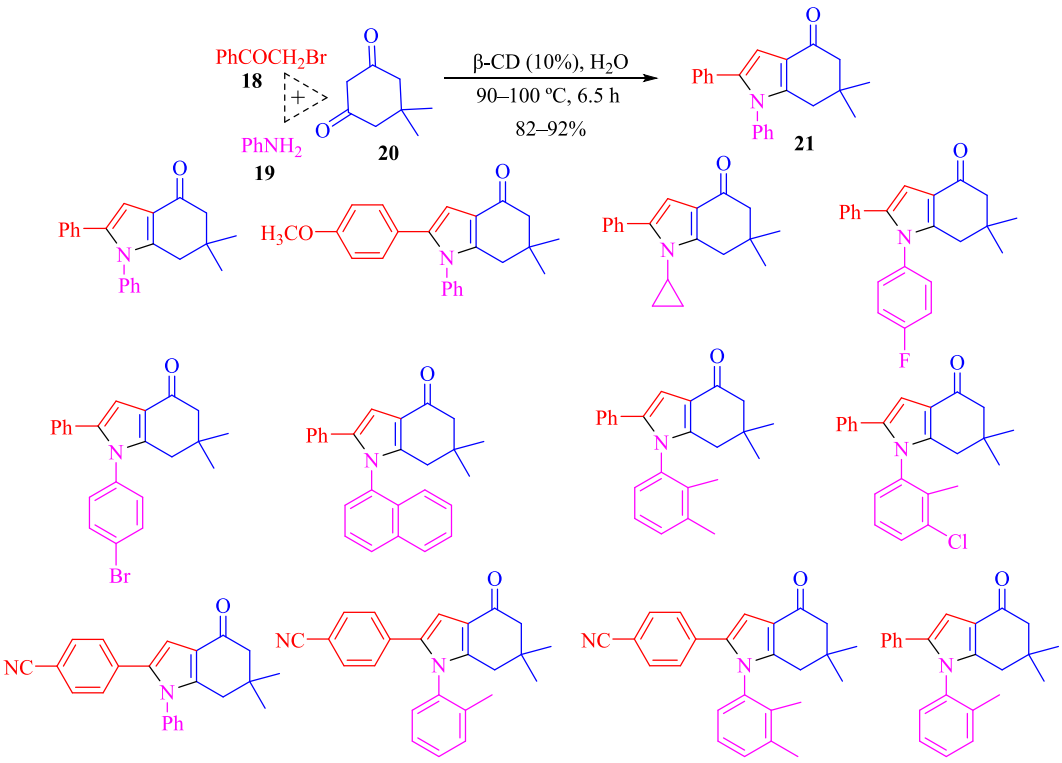
The one-pot reaction of aromatic aldehydes **1**, ethyl acetoacetate (**9**), hydrazine hydrate (**10**), and barbituric acid (BA, **16**) has been represented for the synthesis of pyrazolopyranopyrimidines **17** by Akolkar and *et al.* (Scheme 11) (Akolkar et al., 2020). This method is green, rapid, and ultrasound-assisted. They used β-CD as a recyclable catalyst in H_2O for synthesizing pyrazolopyranopyrimidines with superb yield (84–93 %). The result showed that β-CD was recycled for five runs. The comparative study with a former reported method for synthesizing pyrazolopyranopyrimidines is exhibited in Table 2. The authors proposed a possible mechanism demonstrated in Scheme 12. The first condensation of hydrazine hydrate and ethyl acetoacetate creates an intermediate equilibrium with its enolate **10**″. Then, intermediate **11**″ is made by Knoevenagel condensation of BA and aryl aldehyde. The intermediates **10**″ and **11**″ in the hollow of β-CD undergo Michael's addition to generate **12**″. Intramolecular cyclization by the nucleophilic addition of oxyanion to the C═O group provides intermediate **13**″. Eventually, the dehydrating intermediate D will convert to the expected product.

Dhananjaya *et al.* represented a preparation of 4-oxo-4,5,6,7-tetrahydroindoles **21** by β-CD (10 % w/w) in water (Dhananjaya et al.,2020). This reaction was conducted in a three-component response (TCR) of phenacyl bromide (**18**), primary amine (**19**), and dimedone (**20**). A spectrum of products was obtained using this green procedure with a high yield (82–92 %) (Scheme 13). The catalyst plays a critical role in raising the efficiency of the reaction, mainly by activating the bromo group of the phenacyl bromide and assisting the water solubility of all the reactants. β-CD could be recycled up to the third cycle with no notable product yield loss. This method has some advantages compared to previous approaches (Zhang et al., 2013; Caliskan et al., 2020), such as metal-free conditions, non-toxic catalysts, the ability to recycle, and active biological products. A reasonable reaction mechanism for this MCR reaction by β-CD is illustrated in Scheme 14. The nucleophilic attack by the dimedone **20** through its enol form on the bromo group having carbon the phenacyl bromide **18** was also facilitated by β-CD afforded the tri-keto intermediate **14**″. On further reaction with amine **19,** the **14**″ afforded an enamine intermediate **15**″ that on intramolecular cyclization furnished the desired product **21**.

Bahadorikhalili *et al.* prepared Cu@β-CD@MGO as a nanocatalyst for the preparation of *N*-(alkyl)-2-phenylimidazo[1,2-*a*]pyridin-3-amines **5**



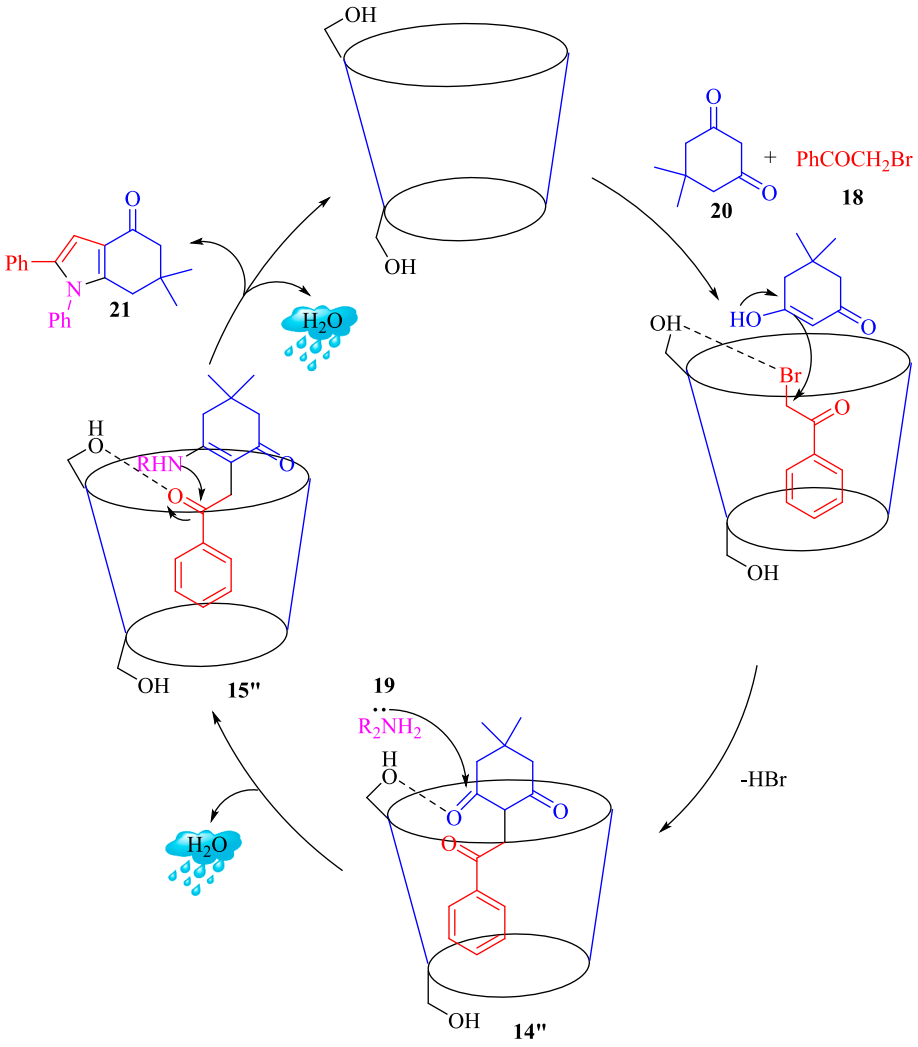
Scheme 12. The possible mechanism for one-pot synthesis of pyrazolopyranopyrimidines.



Scheme 13. The preparation of 4-oxo-4,5,6,7-tetrahydroindoles by β-CD catalyst.

via an efficient and atom-economical method (Bahadorikhalili et al.,2020). The TCR obtained aldehydes (from **22**), pyridin-2-amine (**4**), and isocyanides **3** were developed, and *N*-(alkyl)-2-phenylimidazo [1,2-*a*]pyridin-3-amine produced provided in high-yield (65–75 %) (Scheme 15). Techniques like XRD, TGA, TEM, SEM, VSM, FT-IR, and ICP analyzed this new nanocatalyst. SEM and TEM results showed that iron oxide nanoparticles (20–25 nm) were seen as dark spots within the graphene oxide nanosheets. The FTIR spectrum of βCD@MGO indicated the attachment of β-CD to the MGO. The bands at 3430 cm^{-1} are owing to the O—H groups of β-CD connected to the surface, and the band at 1691 cm^{-1} was allocated to the C═O. This nanocatalyst exhibited high reusability with no noticeable leaching detected after ten times. At the start of the reaction, aerobic oxidation of benzyl alcohol to the relevant aldehyde occurs in Cu@βCD@MGO catalyst. The fantastic benefit of this stage is that the oxidation reaction happens in the presence of oxygen in the air and without the requirement for other oxidizing reagents. A comparison between the present approach and formerly reported strategies for synthesizing phenylimidazo[1,2-*a*]pyridine derivatives demonstrated the excellent activity and high performance of Cu@βCD@MGO catalyst for synthesizing the cited compounds. In most cases, using non-reusable catalysts restricts these compounds' industrial production. Additionally, utilizing toxic or high-temperature conditions is one of the other disadvantages of some of the previous reports (Bharate et al., 2013, Bode et al., 2011). The possible mechanism was offered by the authors and displayed in Scheme 16. At the start of the reaction, aerobic oxidation of benzyl alcohol **22** to the corresponding aldehyde **1** occurs in the presence of Cu@βCD@MGO catalyst. The created aldehyde reacts to pyridine-2-amine **4**, forming **16″** intermediated. The reaction of 16″ intermediated with isocyanide derivative 3 leads to forming **17″** intermediated. An intramolecular cyclization reaction occurred in **17″** intermediated, which gives **18″** intermediated. The desired phenylimidazo[1,2-*a*]pyridines **5** will be formed by a 1,3-H-shift in the compound **18″**.

The Friedel-Crafts alkylation reaction of indoles **23** with aryl, heteroaryl as well, as alkyl aldehydes **1** by β-cyclodextrin hydrate (β-CDH) for the synthesis of bis-(indol-3-yl)-methanes **24** reported by Das *et al.* (Scheme 17) (Das et al., 2020). This reaction indicates good chemoselectivity under mild reaction conditions. The reactions were completed at 60 °C in H_2O as a safe solvent, providing products with good to high yields (87–94 %). The other supramolecular catalysts, like *α*- and *γ*- CD, were less helpful in this reaction. The β-CDH catalyst could be recycled six times without an appreciable drop in performance. Some of the previous studies listed in Table 3 involved the usage of pricey catalysts (Sun et al., 2015; Fu et al., 2020; Bahuguna et al., 2018; Huo et al., 2013), multistep synthesis of catalysts (Bahuguna et al., 2016) long reaction time (Sun et al., 2015; Fu et al., 2020; Bahuguna et al., 2018), and lack of chemoselectivity (Gao et al., 2017; Huo et al., 2013). The current β-cyclodextrin hydrate-catalyzed metal-free protocol in aqueous medium mainly does away with these weaknesses (entry 7). The possible mechanism was demonstrated in Scheme 18. The nucleophilic attack from 2-methylindole **23** to benzaldehyde **1** with the water molecules inside the cavity of β-CD hydrate) the intermediate **19″** is



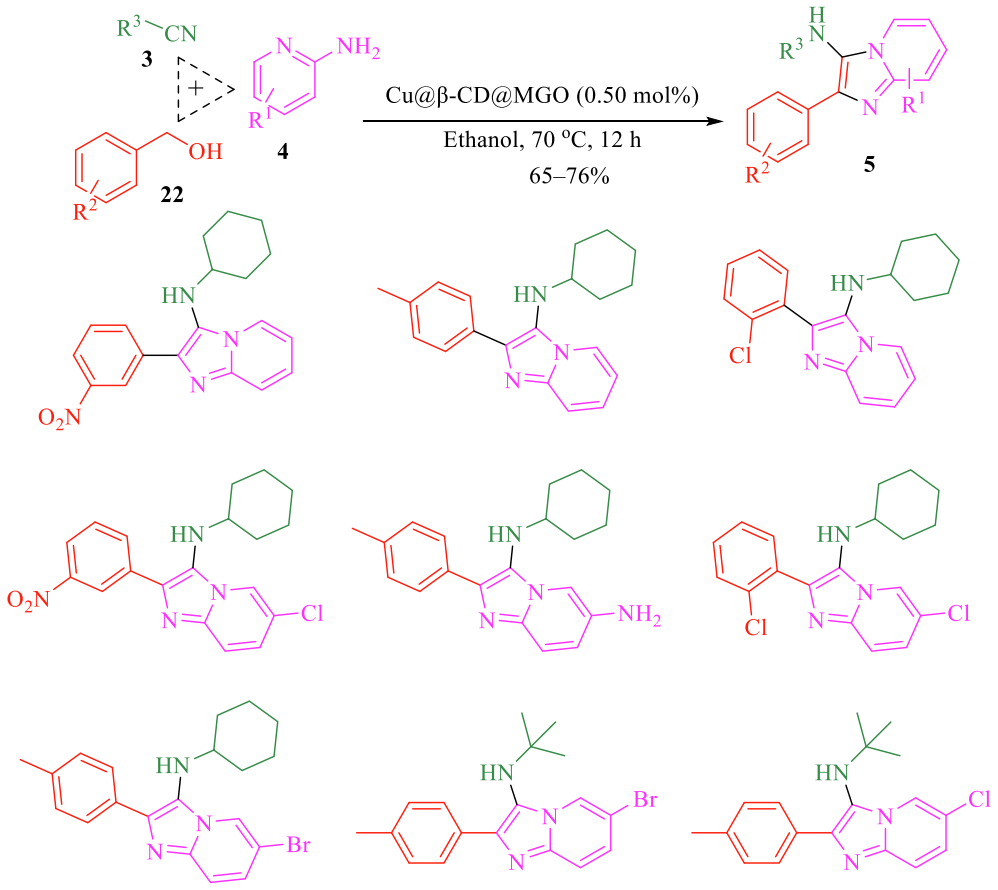
Scheme 14. Offered path for the construction of 4-oxo-4,5,6,7-tetrahydroindoles.

enhanced and rapidly created in the first stage. Subsequent dehydration of the intermediate **19**″ makes the corresponding **20**″, which, on further nucleophilic attack by another 2-methylindole **23,** affords the product **24**.

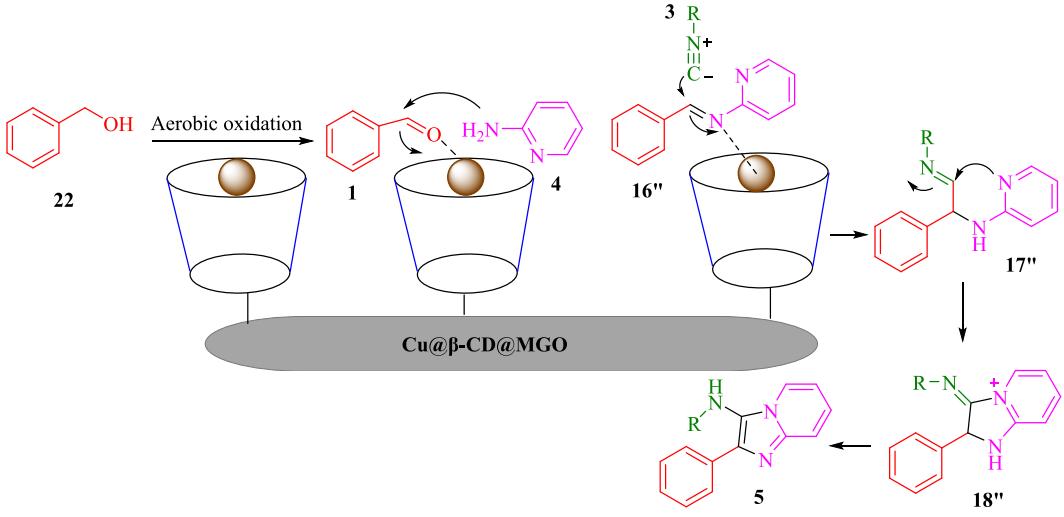
In 2022, Bhadke *at el.* illustrated a green protocol for the synthesis of pyrazolones **26** from ethyl acetoacetate (**9**) and phenylhydrazine (**25**) *via* the β-CD (12 mol%) catalyzed in H_2O at 80 °C for 20–30 min. β-CD acts as an effective host and powerful catalyst (Scheme 19) (Bhadke et al., 2022). This procedure reduced the restrictions of former traditional strategies, some of which needed transition metal catalysts, long reaction times, harsh reaction conditions, or costly reagents. β-CD furnishes such innate advantages as eco-friendly conditions, low prices, simplification of protection-deprotection stages, and decreased waste.

Among the various procedures famous in the literature for the synthesis of triazoles utilizing click chemistry, one that meets the goal of the green strategy is the usage of CDs as a phase transfer catalyst. In 2022, for the first time, Madhuri and at el. illustrated the synthesis of spirochromanone attached 1,2,3-triazoles **29** and spirochromanone conjugates, including bis-1,2,3-triazoles (Scheme 20) (Madhuri et al., 2022). Spirochromanone linked 1,2,3-triazoles and spirochromanone conjugates comprising bis-1,2,3-triazoles were prepared for the first time and are assessed for probable anti-bacterial activity. The process applies easy, practical click chemistry, a low-cost phase transfer catalyst, and water as an eco-friendly solvent.

In 2022, Tajbakhsh *et al.* described a facile method for synthesizing isoxazole derivatives **31** through a TCR from aryl aldehydes **1**, ethyl acetoacetate (**9**), and hydroxylamine hydrochloride (**30**) using Cu@Met-β-CD as a biodegradable, and reusable catalyst (Scheme 22) (Tajbakhsh et al., 2022). The steps of this catalyst preparation are provided in Scheme 21. This catalyst was analyzed using EDX, SEM, FT-IR, TGA, and

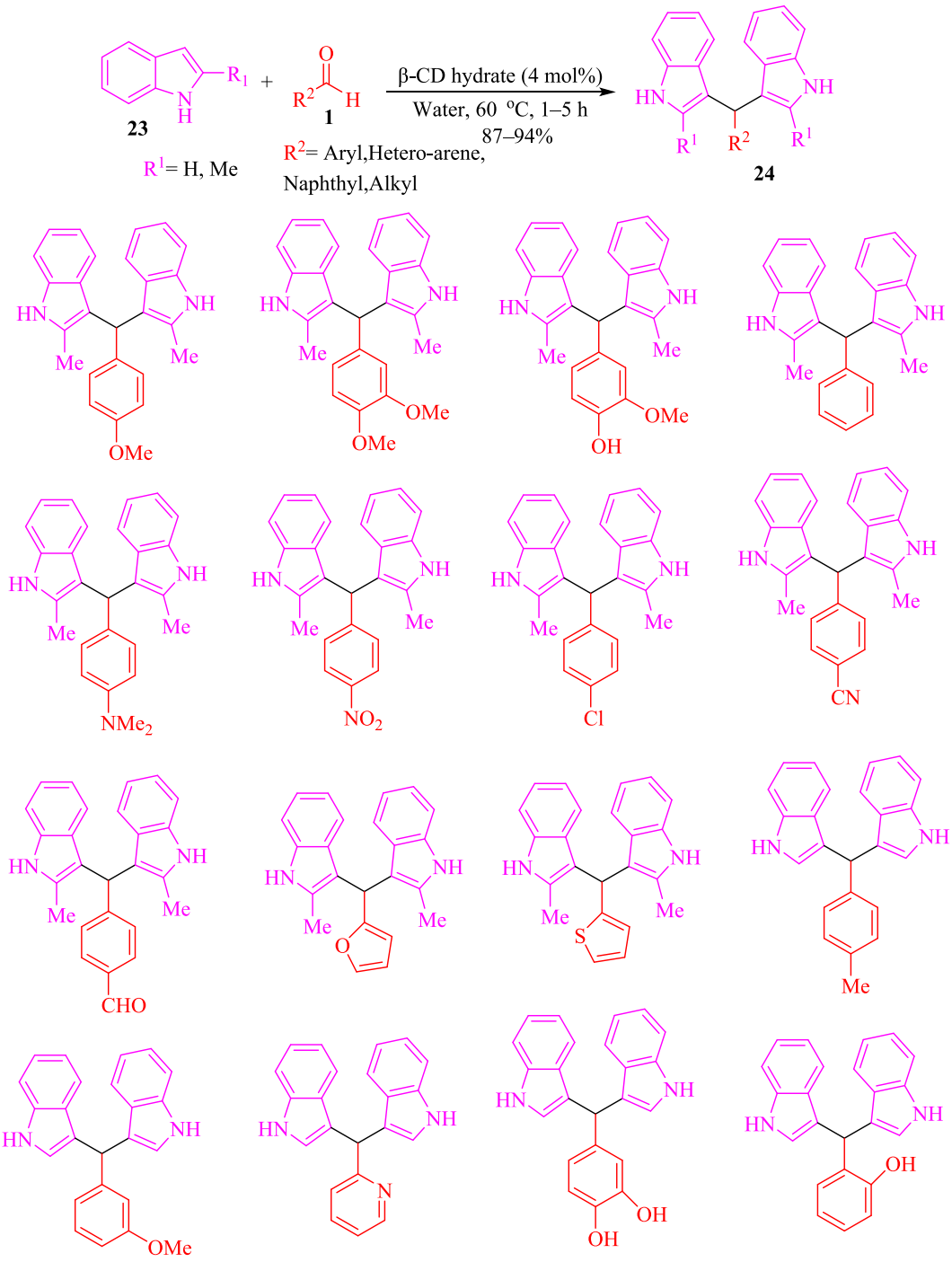


Scheme 15. Synthesis of phenylimidazo[1,2-*a*]pyridines by Cu@β-CD@MGO.



Scheme 16. The suggested way for this reaction is using Cu@β-CD@MGO.

XRD analyses. In the FT-IR analysis, the strong absorption bands at 3380 cm^{-1} and 1640 cm^{-1} correspond to OH groups' stretching and bending vibrations, respectively, were observed. The aliphatic CH absorption bands of β-CD can be seen at 2925 cm^{-1}. The peak at 1370 cm^{-1} corresponds to the characteristic bands of the S═O tosyl group. The peak of 1624 cm^{-1} corresponds to stretching bonds C═N of metformin, which shifted to 1650 cm^{-1} and varied the form of the peak in Cu@Met-β-CD upon complexation with copper. This work has advantages like



Scheme 17. Synthesis of bis-(indolyl)methanes by β-CD hydrate catalyzed.

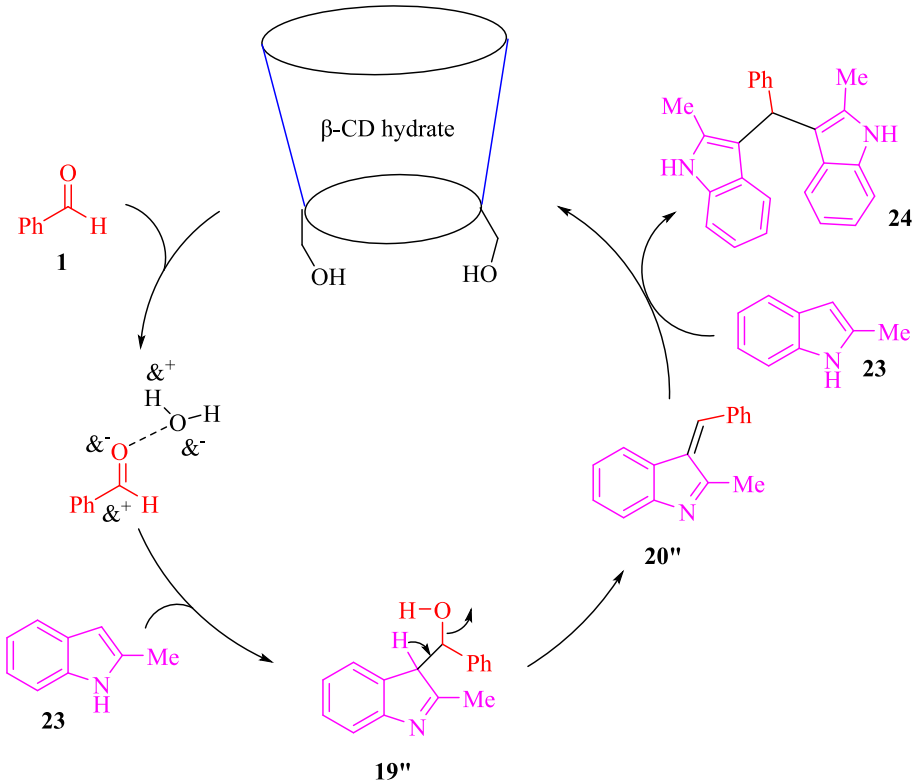
Table 3
A comparison of the catalytic efficiency of diverse catalysts is introduced in the literature.

Entry	Catalyst	Solvent	Time (h)	Temp (℃)	Yield (%)	Reference
1	–	Ethyl lactate: H_2O	0.5	rt	91	Gao et al., 2017
2	[DABCOH][HSO_4]	–	2	90	79	Tong et al., 2016
3	α-Chymotrypsin	H_2O	24	70	90	Sun et al., 2015
4	Lipase enzyme	H_2O	36	55	95	Fu et al., 2020
5	ZnO-RGO	EtOH: H_2O	12	rt	86	Bahuguna et al., 2018
6	TPPMS/CBr_4	CH_3CN	4	rt	72	Huo et al., 2013
7	β-CD hydrate	H_2O	3	60	89	Current discussed report

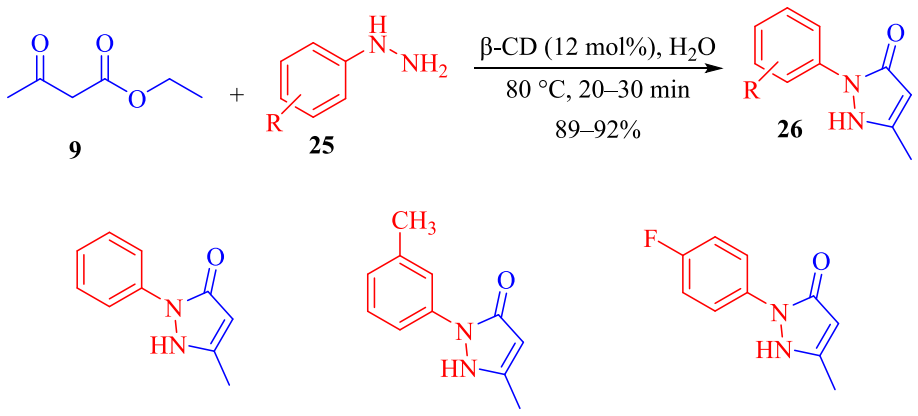
clean workup, short reaction times, excellent yields, and an environmentally friendly process. The recovery of the Cu@Met-β-CD was tested, and the results showed that catalysts were successfully recycled seven consecutive times with little activity reduction. The SEM images of the catalyst indicated round shape morphology in average size, mainly in the range of < 50 nm. Table 4, compares Cu@Met-β-CD and various catalysts in synthesizing 4-(4-hydroxybenzylidene)-3-methylisoxazol-5 (4*H*)-one. The Authors also suggested the mechanism for the reaction (Scheme 23). Firstly, the Cu immobilized in functionalized β-CD works as a Lewis acid and enhances the electrophilic properties of the carbonyl groups in ethyl acetate. Then, the nucleophilic attack of the NH_2 group **30** occurs at the activated carbonyl carbon of **9**, resulting in an oxime intermediate **21″**. The condensation provides isoxazol-5-ones as the heterocyclic compound **22″**. The obtained carbanions can also be utilized in condensation reactions with aldehydes to produce electrophilic arylidene isoxazole-5-ones **23″**.

In 2023, Morita *et al.* designed a green procedure for synthesizing 2,3-dihydrobenzofurans **34** (Scheme 25) and 1,2,3-trisubstituted indanes **37** (Scheme 26) stereoselectively using permethylated β-CD-tagged NHC–gold(I) (Morita et al., 2023). The structure of β-CD-NHC-AuCl is displayed in Scheme 24. The gold(I) catalyst performs perfectly in these reactions, which could be recovered in five runs. The suggested reaction mechanism for preparing 1,2,3-trisubstituted indanes is shown in Scheme 27. At first, the OH group of benzylic alcohol **36** is activated by coordinating a gold center, possibly inner the hydrophobic cavity of the β-CD, creating an active intermediate **24″**. The addition of *trans*-anethole **35** to the intermediate **24″** provides the intermediate **25″** with an electron-rich aromatic ring and an electron-deficient ring. After cyclization between the aromatic ring and the benzylic part in the intermediate **25″**, product **37** was afforded.

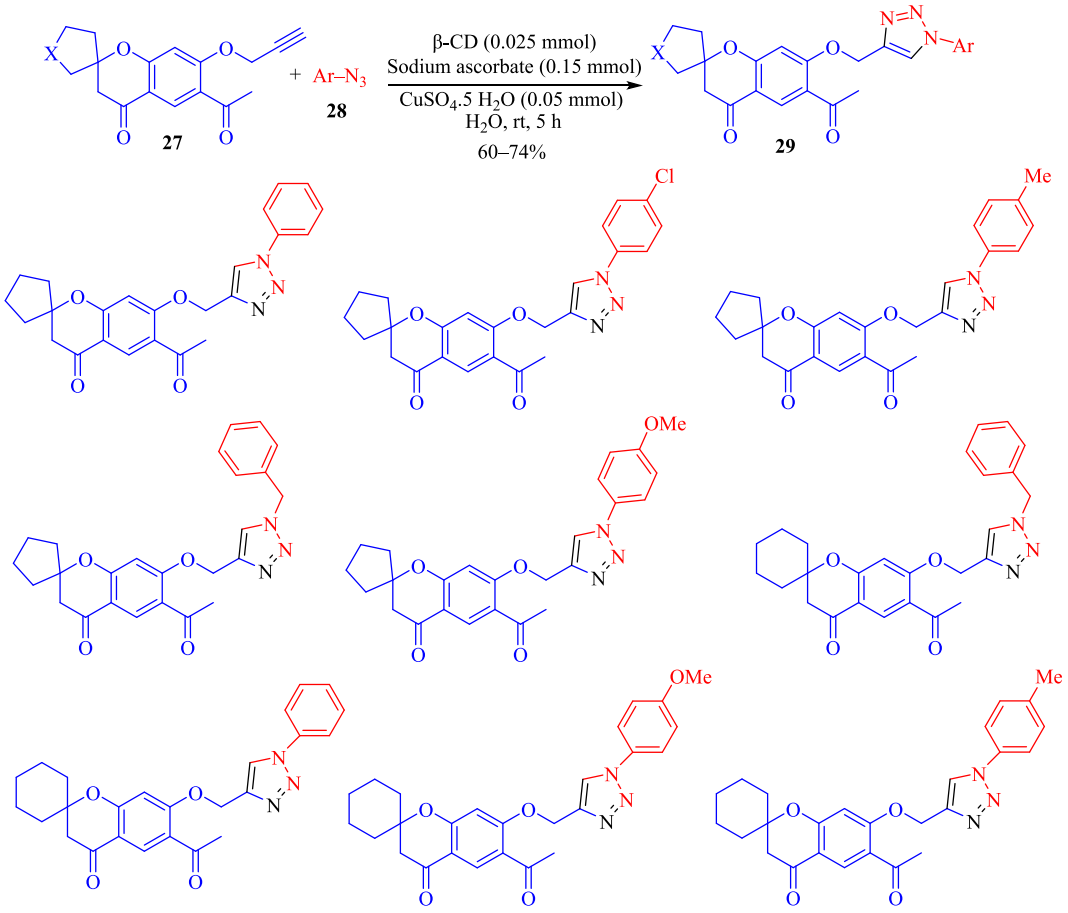
Paul *et al.* reported a green method for the preparation of 1,2,3-functionalized 4-hydroxy pyrrolidine-5-ones **40** from aldehydes **1**, amines **38**, and dimethylacetylenedicarboxylate (**39**) employing a supramolecular catalyst β-CD (Scheme 28) (Paul et al., 2023). This reaction was performed without metal salt in a water/ethanol medium. The reactions were completed in a time (8 h), and the product was established with satisfactory yields (64–90 %). The offered mechanism for synthesizing pyrrolidine-2-one derivates is indicated in Scheme 29. β-CD activates both aryl aldehyde **1** and di(methyl/ethyl) acetylene dicarboxylate **39** compounds simultaneously as the active electrophilic species,



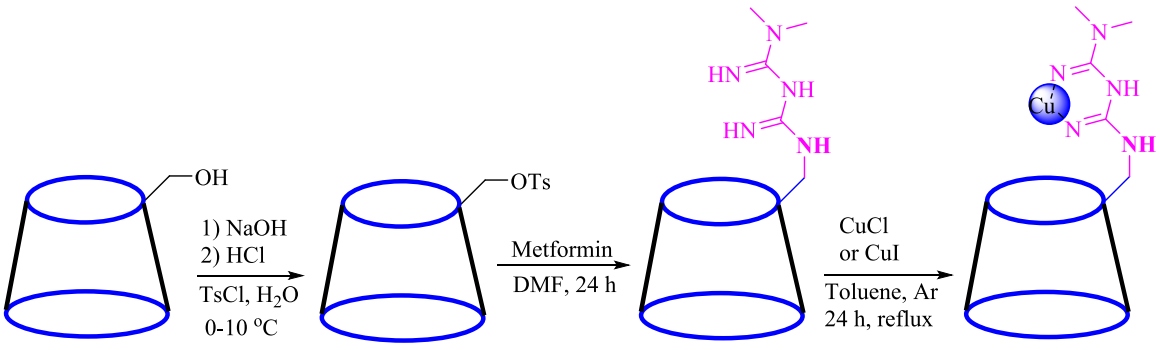
Scheme 18. The suggested mechanism for reactions of indoles with aldehydes catalyzed by β-CD hydrate.



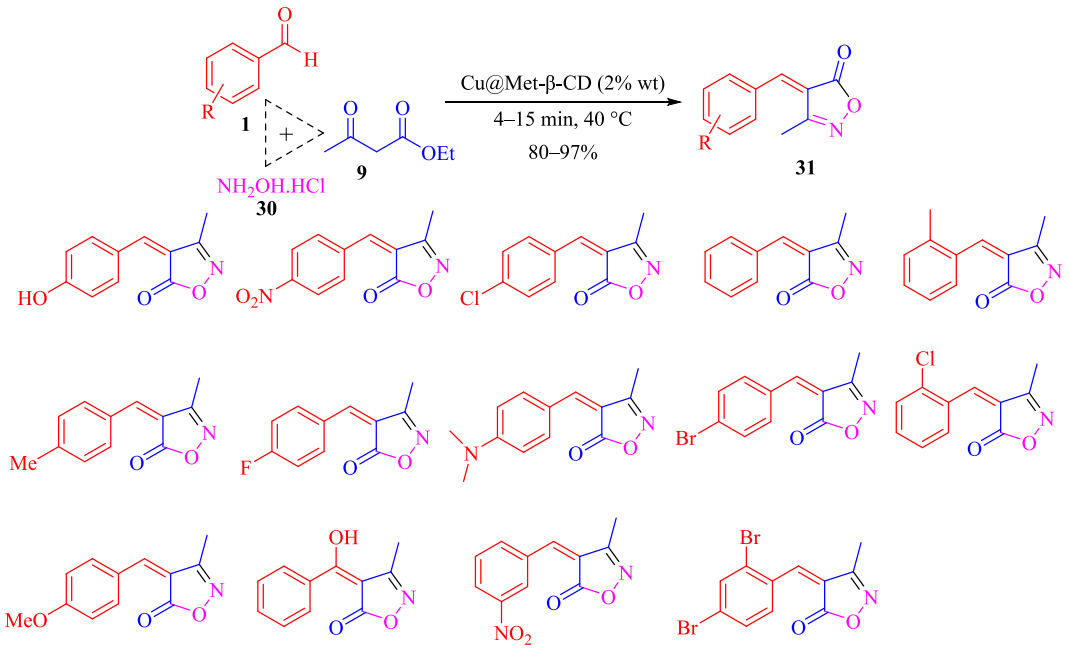
Scheme 19. Synthesis of pyrazolones derivates **26**.



Scheme 20. The preparation of triazole moieties by β-CD catalyst.



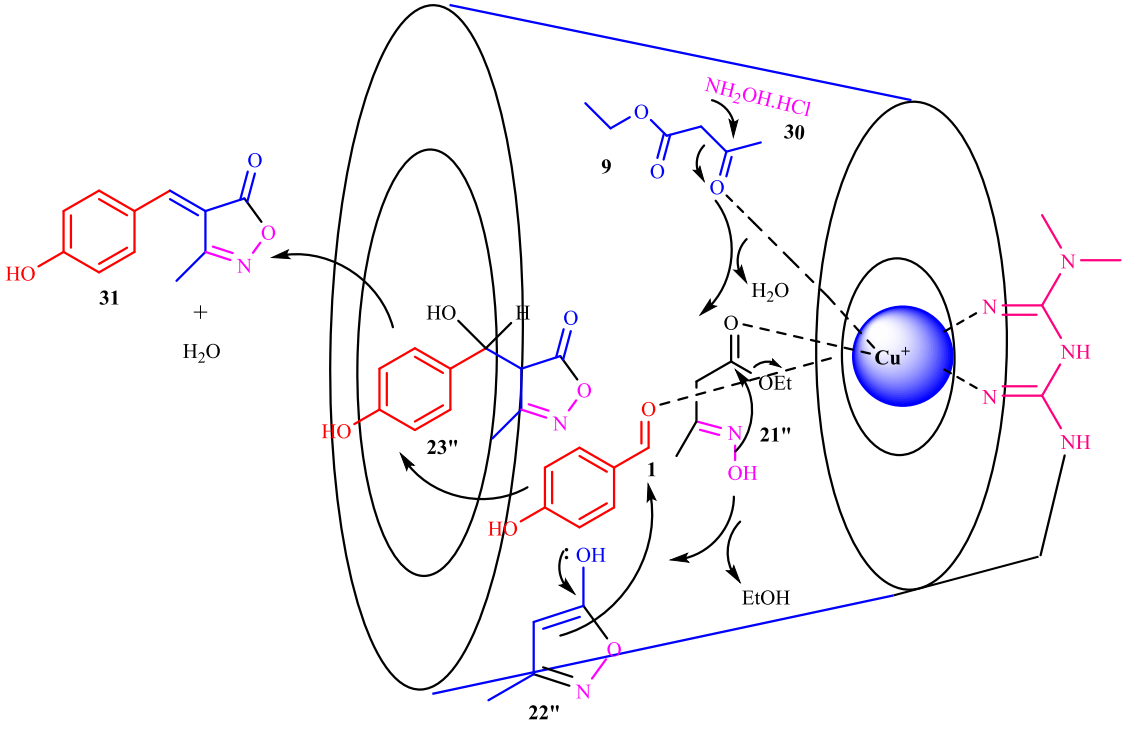
Scheme 21. Steps catalyst preparation.



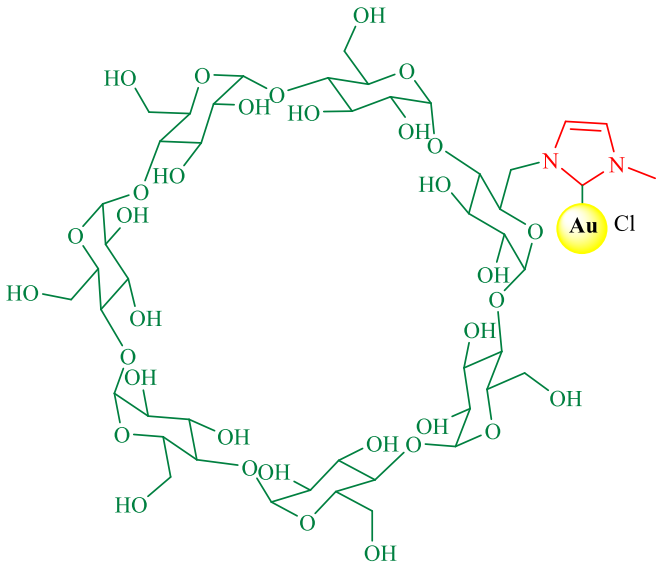
Scheme 22. The synthesis of isoxazole derivatives using Cu@Met-β-CD.

Table 4
Comparison of Cu@Met-β-CD and different catalysts in synthesizing 4-(4 hydroxybenzylidene)-3-methylisoxazol-5(4H)-one.

Entry	Catalyst	Condition	Time (min)	Yield (%)	Reference
1	Sodium acetate	EtOH/H_2O, rt	30	90	Aslam et al., 2020
2	DABCO	EtOH, rt	30–180	87	Kim et al., 2018
3	Modified-MMT	H_2O, 70 °C	7–70	92	Mashhadinezhad et al., 2018
4	Cu/TCH-pr@SBA-15	Solvent-free, 80 °C	8	95	Kalhor et al.,2020
5	L-Valine	EtOH, reflux	1–240	95	Kour et al., 2020
7	Cu@Met-β-CD	H_2O, rt. up to 50 °C	2–5	97	Current discussed report



Scheme 23. Suggested path for the reaction by Cu@Met-β-CD.



Scheme 24. The structure of β-CD-NHC-AuCl catalyst.

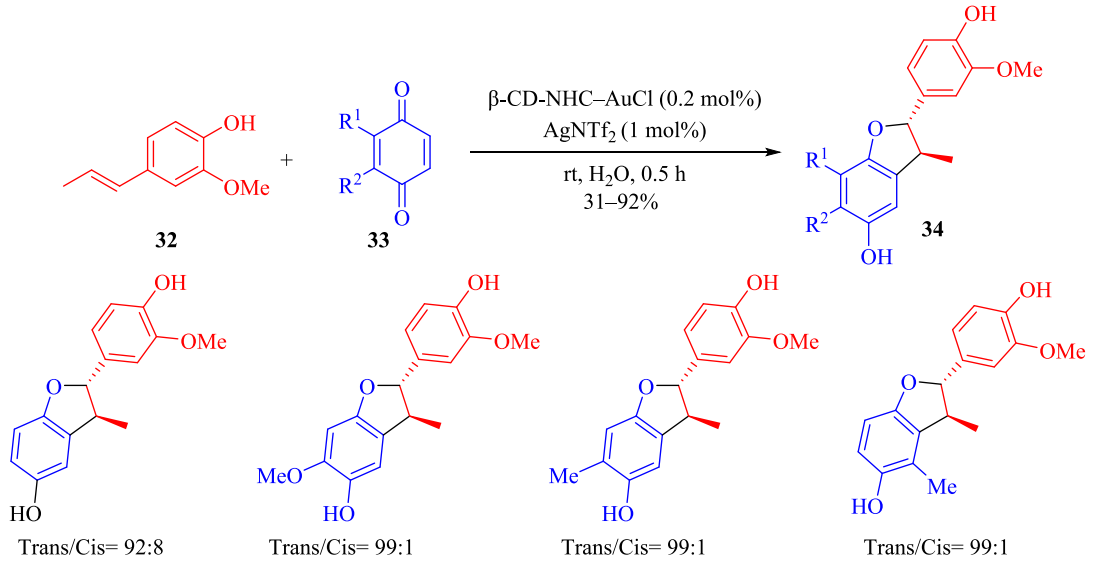
facilitating the reaction. The condensation of aryl amines **38** with these activated electrophilic aldehydes with the loss of H_2O, along with the reaction of H_2O with activated electrophilic di(methyl/ethyl) acetylene dicarboxylate compound, may then produce the corresponding intermediates **26**″ and **27**″, respectively. After that, the reaction between these two intermediates **26**″ and **27**″ will yield the corresponding intermediate **28**″. Eventually, a series of tautomerization, cyclization, and aromatization reactions create the intermediates **29**″, and **30**″, and finally, the product **40**.

2.2. Synthesis of six-membered heterocycles

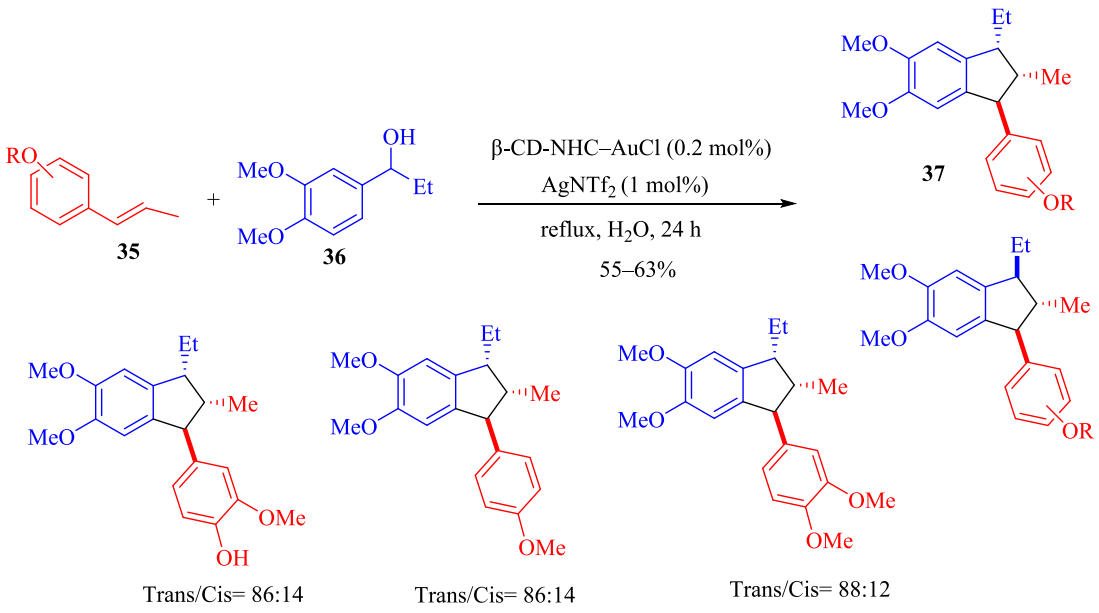
Ahadi *et al.* introduced MNPs@β-CD@Cu(OAc)$_2$ as an impressive catalyst for the preparation of spiropyrans **43** from isatin (**41**), dimedone (**20**), and 2-substituent-acetonitrile **42** (Scheme 31) (Ahadi et al., 2019). This nanocatalyst was prepared by surface-modified magnetic support by the Cu(II)-β-CD complex. The preparation step of MNPs@β-CD@Cu(OAc)$_2$ is shown in Scheme 30. TGA, FT-IR, XRD, SEM, and VSM characterized the structure of the nanocatalyst. The result obtained from the SEM analyst showed that the average diameter of the nanocatalyst was about 33 nm. The reusability of the nanocatalyst was examined, and it turned out that the catalyst recycled six runs with no significant loss in performance. The chief properties in this work are green condition, short reaction period, facile workup, and reusability catalytic by using an external magnet. Table 5, compares MNPs@β-CD@Cu(OAc)$_2$ catalyst with other catalysts reported for the preparation of spiropyrans. The suggested mechanism for the reaction is exhibited in Scheme 32. The carbonyl group at isatin **41** could be activated by MNPs-β-CD@Cu(OAc)$_2$ in the β-CD cavity, so the nucleophilic attack by the CH acidic group of 2-substituent-acetonitrile **42** (*Knoevenagel* condensation) could be carried out to attained the intermediate **31**″. Then, Michael's addition is done by enolized dimedone **20** to create the intermediate **32**″. Then, nucleophilic attack through oxygen onto the CN group, and next, tautomerization provides the **43** product.

An efficient method of the preparation of pyrano[2,3-*d*]pyrimidine-2,4,7-triones **45** from aryl aldehydes **1**, BA (**16**), and Meldrum acid (**44**) has been developed by β-CD catalyst by Bhosle *et al.* (Bhosle et al., 2019). This approach obtained various pyrano[2,3-d] pyrimidine-2,4,7-triones in superb yields (Scheme 33). This reaction was conducted under mild water at 65 °C *via* a one-pot MCR. Compared to *α*-and *γ*-CD, β-CD gives excellent results as a catalyst. *α*- and *γ*- CD yield 45 and **39**, respectively

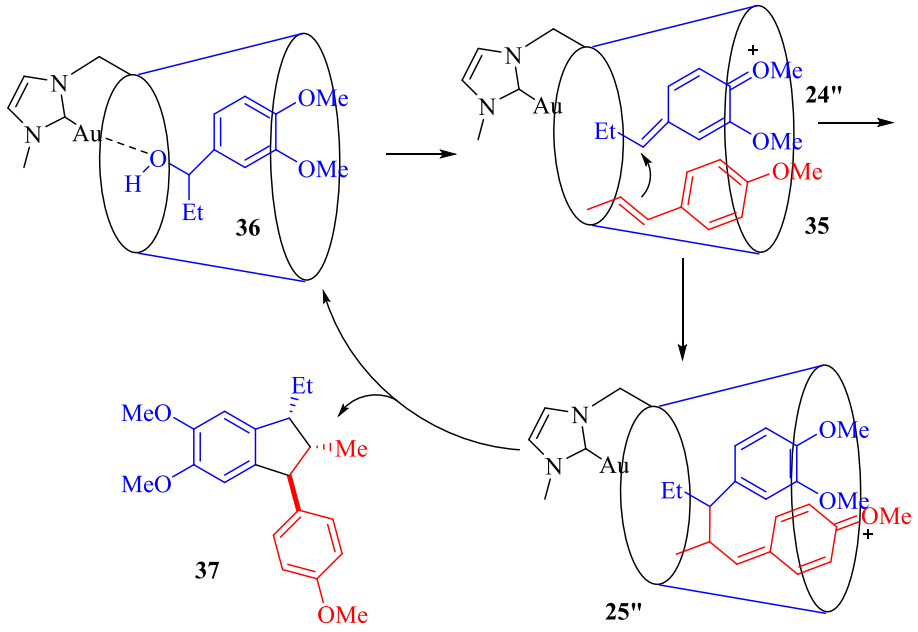
Mohammadian and Akhlaghinia introduced Fe_3O_4/COS@β-CD-SO_3H NPs as highly efficient and recoverable magnetic nanocatalysts for the preparation of spirooxindoles **43** *via* one-pot, MCR from isatin (**41**), dimedone (**20**), and malononitrile (**42**) in water (Scheme 35) (Mohammadian and Akhlaghinia, 2019). The structure of magnetic nanocatalysts is indicated in Scheme 34. This new magnetic nanocatalyst was characterized by different techniques comprising XRD, FT-IR, EDX, SEM, VSM, TGA, and TEM analyses. The TEM image of magnetic nanocatalysts showed the presence of sphere particles with a size of 16–25 nm. Spirooxindole products were synthesized by reacting various isatins, ethyl cyanoacetate or malononitrile, and 1,3-dicarbonyl compounds. The recoverability of the nanocatalyst was executed eight consecutive times with no noticeable drop in activity. The significant prominent benefits of this approach are easy workup, short-time reaction, catalyst reusability, and high product yields. Table 6 compares the performance of Fe_3O_4/COS@β-CD-SO_3H NPs and other catalysts to prepare spirooxindoles **43**. The presented mechanism is shown in



Scheme 25. Preparation of 2,3-dihydrobenzofurans derivates by β-CD-NHC-AuCl.



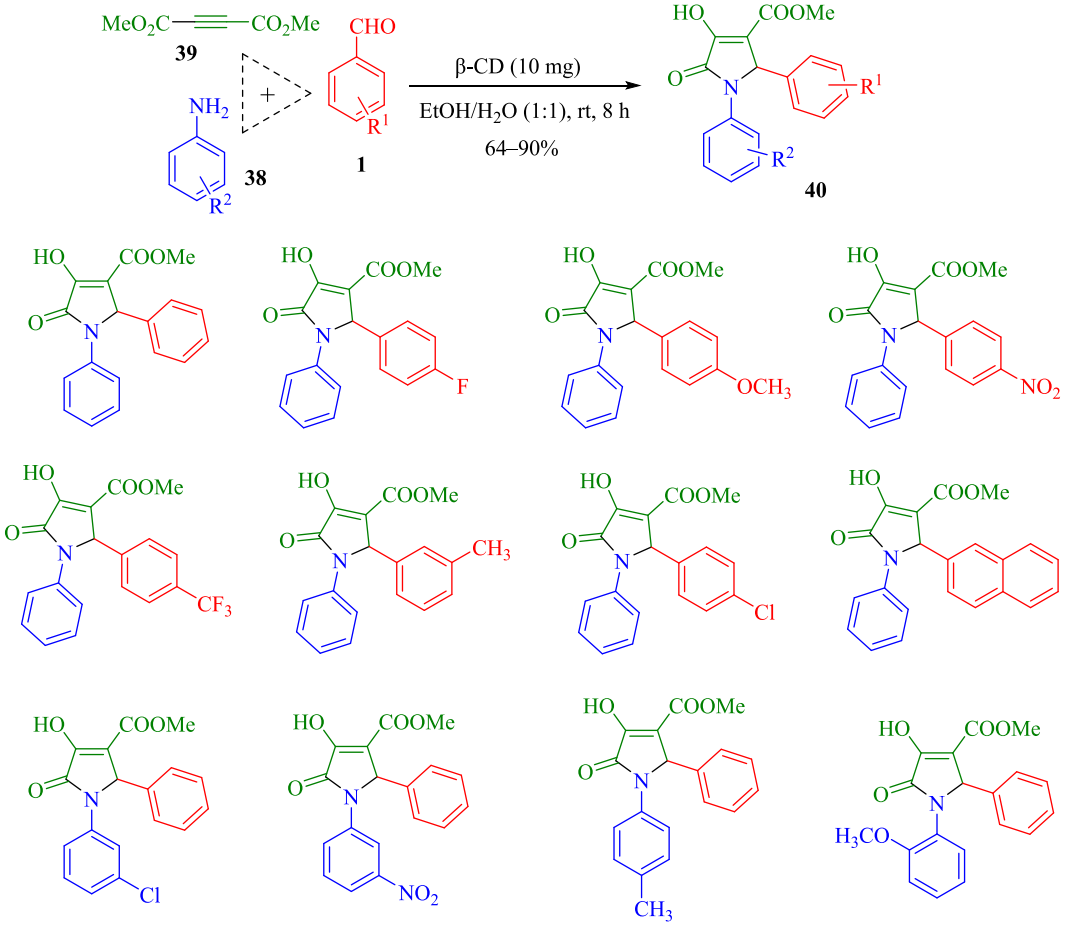
Scheme 26. Preparation of 1,2,3-trisubstituted indanes products by β-CD-NHC-AuCl.



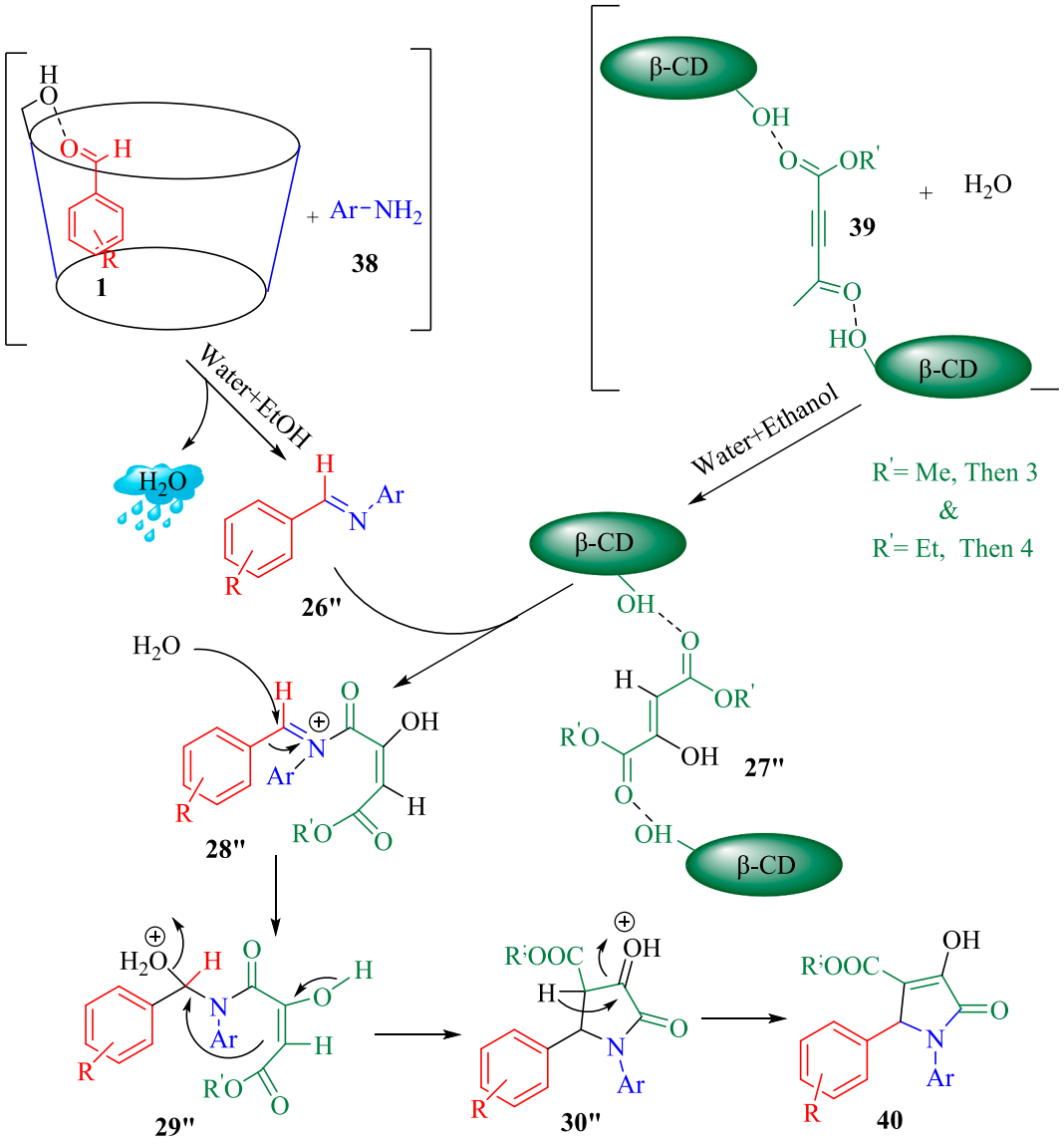
Scheme 27. Suggested reaction mechanism for preparation of 1,2,3- trisubstituted indanes.

Scheme 36. The condensation of isatin **41** with nitrilo-active methylene components **42** to afford **33**″. In the next stage, the electron-deficient adduct **33**″ is attacked *via* acid-promoted Michael addition of activated **20** (1,3-dicarbonyl compound) to give intermediate **34**″. Further, intramolecular cyclization provides the product **43**.

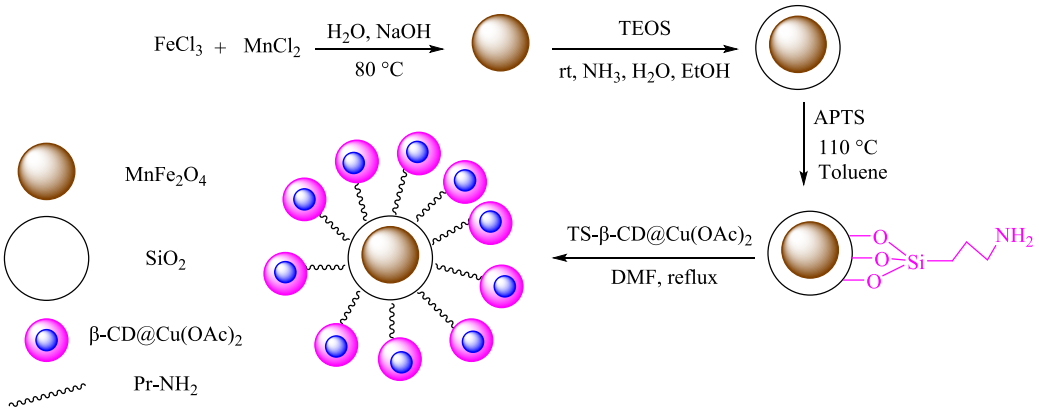
Bhosle *et al.* explained a procedure for the preparation of novel isoniazid fused chromeno[4,3-*b*]quinolins **48** from isoniazid (**46**), dimedone (**20**), 4-hydroxycoumarin (**47**), and aryl aldehydes **1** using β-CD in the H_2O at 60–65 °C (Scheme 37) (Bhosle et al., 2020). This approach provided high yields of the products (71–94 %) *via* one-pot conditions. The catalyst recovered four runs without a substantial performance drop. The probable reaction mechanism is displayed in Scheme 38. The reaction is possibly performed by forming **35**″ and **36**″ in the inner cavity of β-CD. Michael's addition happens with the in situ-created intermediate **37**″, followed by intramolecular cyclization and dehydration to provide the final product **48**.



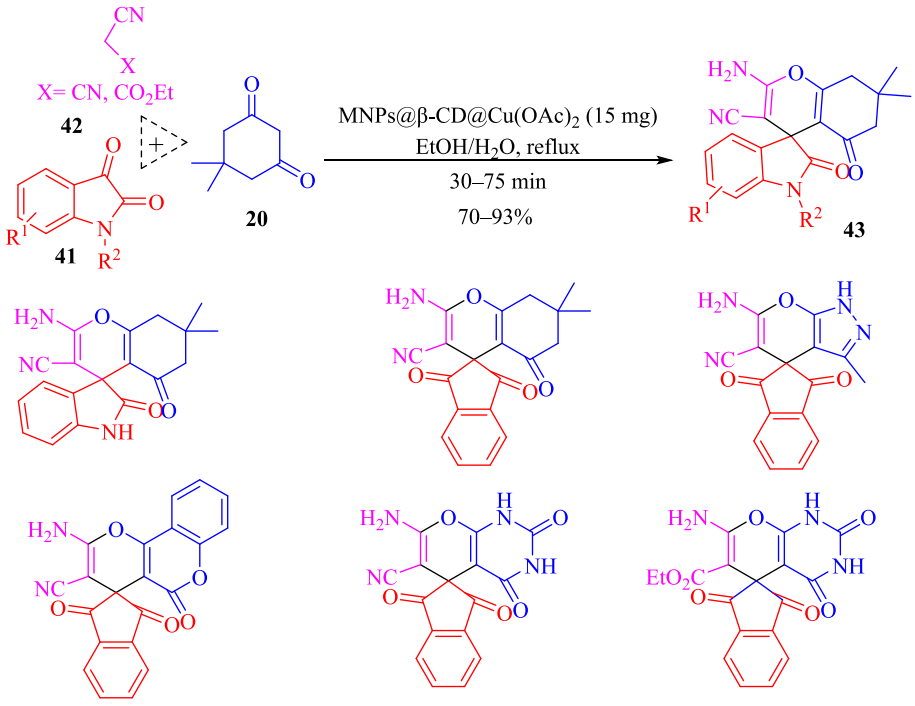
Scheme 28. Synthesis of functionalized pyrrolidine-2-ones using β-CD.



Scheme 29. Recommend mechanism for this reaction by β-CD.



Scheme 30. Synthesis of this new nanocatalyst.



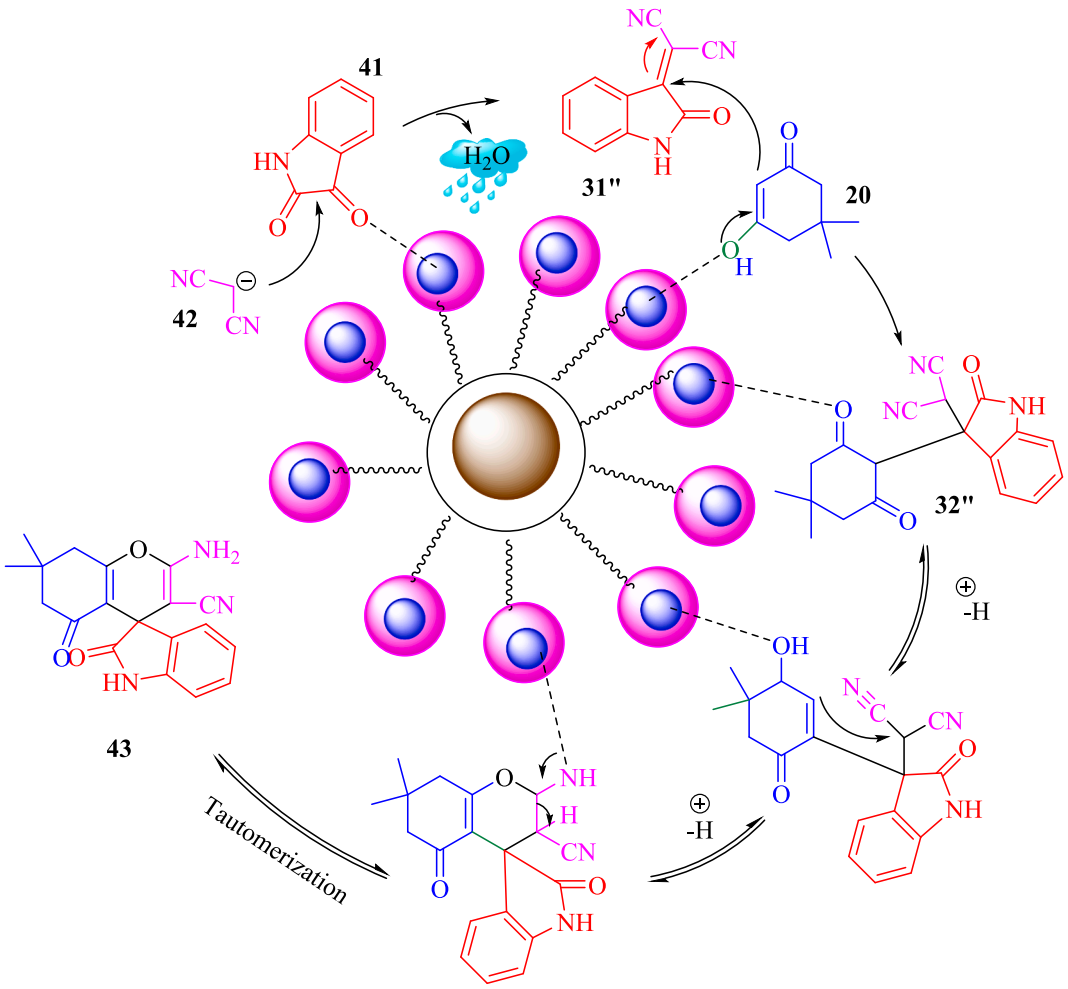
Scheme 31. Preparation of the spiropyrans by MNPs@β-CD@Cu(OAc)$_2$.

Table 5
Compares MNPs@β-CD@Cu(OAc)$_2$ catalyst with various catalysts presented to prepare spiropyrans.

Entry	Catalyst	Condition	Time (min)	Yield (%)	Reference
1	SB-DBU	EtOH:H_2O, 50 °C	150	97	Hasaninejad et al., 2013
2	SSA-MNPs	EtOH:H_2O, 60 °C	80	95	Karimi et al., 2015
3	PEI@Si–MNPs	H_2O, 40 °C	40	92	Khoobi et al., 2015
4	Sodium stearate	H_2O, 60 °C	3 h	95	Wang et al., 2010
5	MNPs@β-CD@Cu(OAc)$_2$	EtOH:H_2O, 80 °C	35	93	Current discussed report

In 2020, Nipate *et al.* described for the first time an eco-friendly strategy for synthesizing 2-phenyl-3,4-dihydroimidazo[4,5-*b*] indoles **50** *via* a one-pot, TCR of aldehyde **1**, isatin (**41**), and NH_4OAc (**49**) by β-CD as a reusable and biodegradable catalyst in EtOH/H_2O at 80 °C within 90 min (Scheme 39) (Nipate et al., 2020). This catalyst recycled for four consecutive runs with a slight decrease in activity. This protocol has some advantages, such as a green catalyst, clean reaction, easy recovery of catalyst, and shorter reaction time. The corresponding compounds were gained in 71–95 % yields under mild conditions. The probable reaction mechanism is presented in Scheme 40. In the first stage, the aldehyde that attaches to the OH group of the β-CD cavity raises the electrophilicity of the carbonyl group reaction, letting it react with ammonia, providing an intermediate **38″**. This intermediate **38″** could again react with ammonia, undergoing dehydration and quickly creating a key intermediate **39″**. Then cyclocondensation, cyclization, dehydration, and tautomerization generate produce **50**.

Naeimi and Rahmatinejad reported a convenient approach for the fabrication of 3,4-dihydropyrano[3,2-*c*]chromenes **52** from aryl aldehyde **1**, 4-hydroxycoumarin (**47**) and ethyl cyanoacetate (**51**) using Fe_3O_4 nanoparticles supported on β-CD-guanidine as a reusable, and heterogeneous catalyst (Scheme 42) (Rahmatinejad and Naeimi, 2020). The step of preparation of Fe_3O_4-β-CD-GA is shown in Scheme 41. FTIR, X-ray diffraction, SEM, TGA, and VSM methods were used to analyze this heterogeneous catalyst. The SEM photo of the catalyst exhibits semi-spherical particles with a moderate size of almost 49 nm. The three-component coupling ethyl cyanoacetate, aryl aldehyde, and 4-hydroxycoumarin were conducted under clean and mild status. The recycling of Fe_3O_4-β-CD-GA nanoparticles under optimized reaction conditions is assessed. The result indicated that the nanocatalyst was recovered for five successive runs with a minor loss of catalyst activity. This procedure has benefits like easy workup, high yields, low catalyst loading, mild conditions, and catalyst reusability. The various amounts of catalyst were examined for the synthesis **52**, and the results are summarized in Table 7. The suggested reaction by the authors is exhibited in Scheme 43. Knoevenagel condensation of activated ethyl cyanoacetate **51** and aldehyde **1** followed by Michel's addition of 4-hydroxycoumarin **47** with the Knoevenagel product **40″** gives intermediate **41″**. Then, cyclization of the intermediate **41″** in the existence of the catalyst produces the intermediate **42″**, which, through tautomerization, forms the final

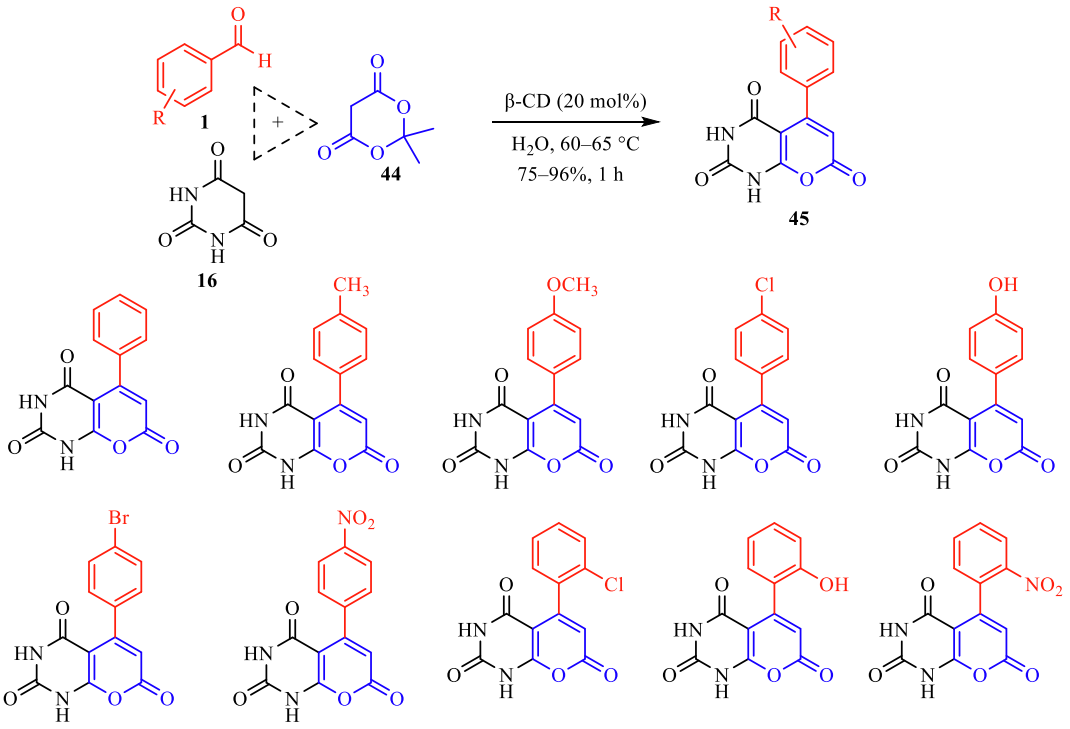


Scheme 32. The proposed way to synthesize spirogyra is by using a nanomagnetic catalyst.

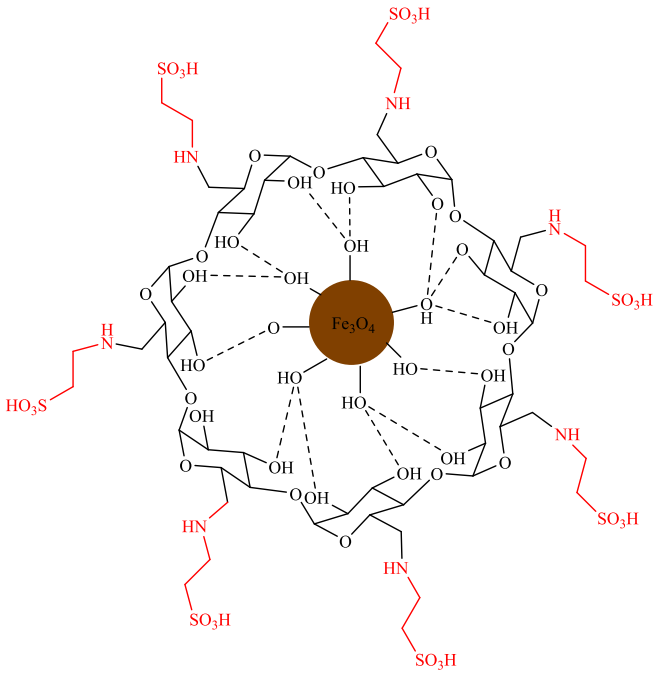
product **52**.

An efficient route to the synthesis of hexahydro-4*H*-indazol-4-ones **53** was developed by Singh *et al.* through the MCR of aryl aldehydes **1**, hydrazine/phenylhydrazine (**25**), and 1,3-diketone **20** using β-CD (113 mg, 10 mol%) as a supramolecular catalyst in H_2O at 60 °C for 20 min (Scheme 44) (Tiwari et al., 2020). The recycling of β-CD as a catalyst is evaluated. The result displayed that the catalyst was reused five consecutive times with little decrease in its activity. The plausible mechanism for synthesizing hexahydro-4*H*-indazol-4-ones **53** is presented in Scheme 45. The reaction is performed *by* activating the C═O bond of benzaldehyde **1** through β-CD-assisted construction of the anion **43**″. Hydrazine **25** attacks the anion **43**″ *via* the Knoevenagel condensation to create an intermediate **44**″, which undergoes Michael's addition of dimedone **20,** resulting in cyclization, leading to the building of the product **53**.

Kardooni *et al.* discussed a one-pot synthesis of Kojic acid-based heterocycles **57** from kojic acid (**56**), aryl aldehydes **54**, and aryl amines **55** using β-CD-based nanosponges as an efficient and biodegradable nanocatalyst *via* Mannich-type condensation reaction in ethanol (Scheme 47) (Kardooni et al., 2020). This catalyst was synthesized by a reaction of β-CD and dicarbonyl imidazole (as a crosslinker agent) in DMF and was stirred for four hours at 100 °C (Scheme 46). The SEM image showed that β-CDNS has an almost smooth and regular surface with several hollows and cavities. The authors found that the usage of ethanol in the presence of β-CD.NS (0.01 mol%) afforded the Kojic acid-based heterocycles an excellent yield (80–95 %) within 25–50 min. This procedure has advantages: superior results, a short reaction period, catalyst reusability, and green reaction conditions. The catalyst recyclability was tested, and the performance of the β-CD-based nanosponge was without considerable shifts after three times. The authors suggested the mechanism of Mannich-type tri-compound condensation as displayed in Scheme 48. Here, CD units of nanosponges work as a nanocontainer, which could create the inclusion complex with the starting compound. The reaction selectivity was controlled by intermolecular



Scheme 33. The preparation of pyrano[2,3-*d*]pyrimidine-2,4,7-triones by β-CD.

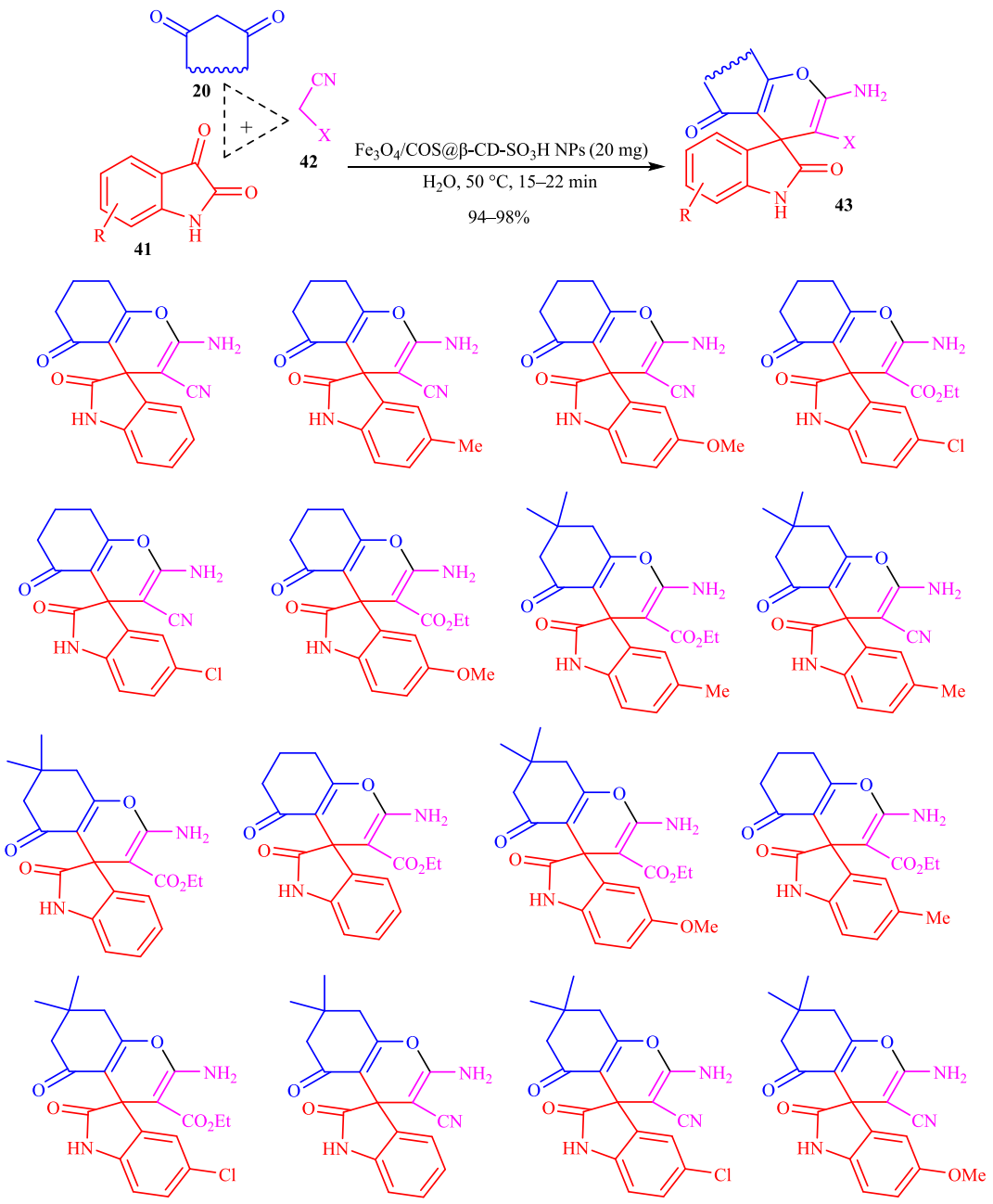


Scheme 34. Structure of Fe_3O_4/COS@β-CD-SO_3H NPs catalyst.

hydrogen bonding between CD units of nanosponges with the guest molecules, which promoted the condensation reaction.

Chate *et al.* disclosed a straightforward preparation of 5-phenyl-5,6-dihydropyrido[2,3-*d*]pyrimidine-2,4,7(1*H*,3*H*,8*H*)-triones **59** from aryl aldehyde **1**, 6-amino uracil (**58**), and Meldrum acid (**44**) through host–guest β-CD complex in water at 100 °C (Scheme 49) (Chate et al., 2020). Other CDs were examined, like α-CDs and γ-CD showed 45 and 65 % yield, respectively. This poorer yield is owing to the small or large cavity, which couldn't make inclusion complexes with the substrates. The synthesis of dihydropyrido[2,3-*d*]pyrimidine-2,4,7(1*H*,3*H*,8*H*)-triones was done in excellent yield (79–97 %) *via* efficient and environmental protocol. β-CD catalyst was reused several times without a noteworthy drop in catalytic performance. The probable pathway of MCRs depicted in Scheme 50 comprises condensation, Michael addition, cyclization, and elimination steps. A proton from Meldrum acid **44** active by β-CD catalyzes a Knoevenagel condensation with the C═O group to provide the arylidene **46**″ intermediate. Then adding arylidene intermediate **46**″ with **58** cyclized presents the intermediate product **47**″. The release of CO_2 and acetone generates the product **59**.

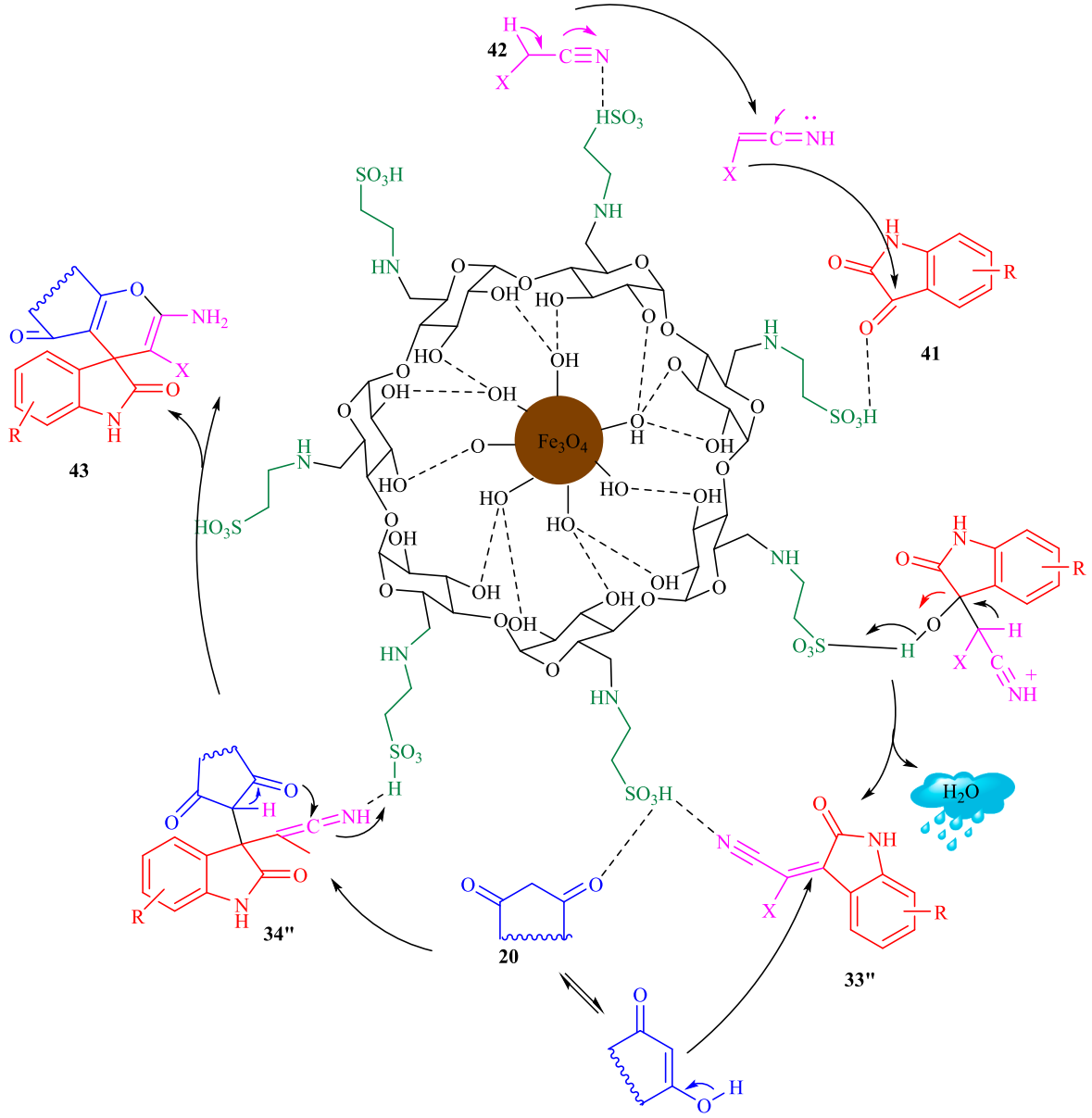
Avvadukkam *et al.* reported a solvent-free method for synthesizing pyrano[2,3-*d*:6,5-*d*′]dipyrimidines **61** catalyzed by β-CD as efficient catalysts with assist the microwave irradiations (Avvadukkam et al., 2021). The TCR of aryl aldehyde **1**, Meldrum's acid (**44**), and 6-amino-1,3-dimethyluracil (**60**) were developed, and pyrano[2,3-*d*:6,5-*d*′] dipyrimidine products afford good to excellent yields (85–96 %) (Scheme 51). The catalyst was recyclable for up to three runs without a notable change in performance. Some valuable features of this method are short reaction time, good to high yields, no tedious workup, eco-



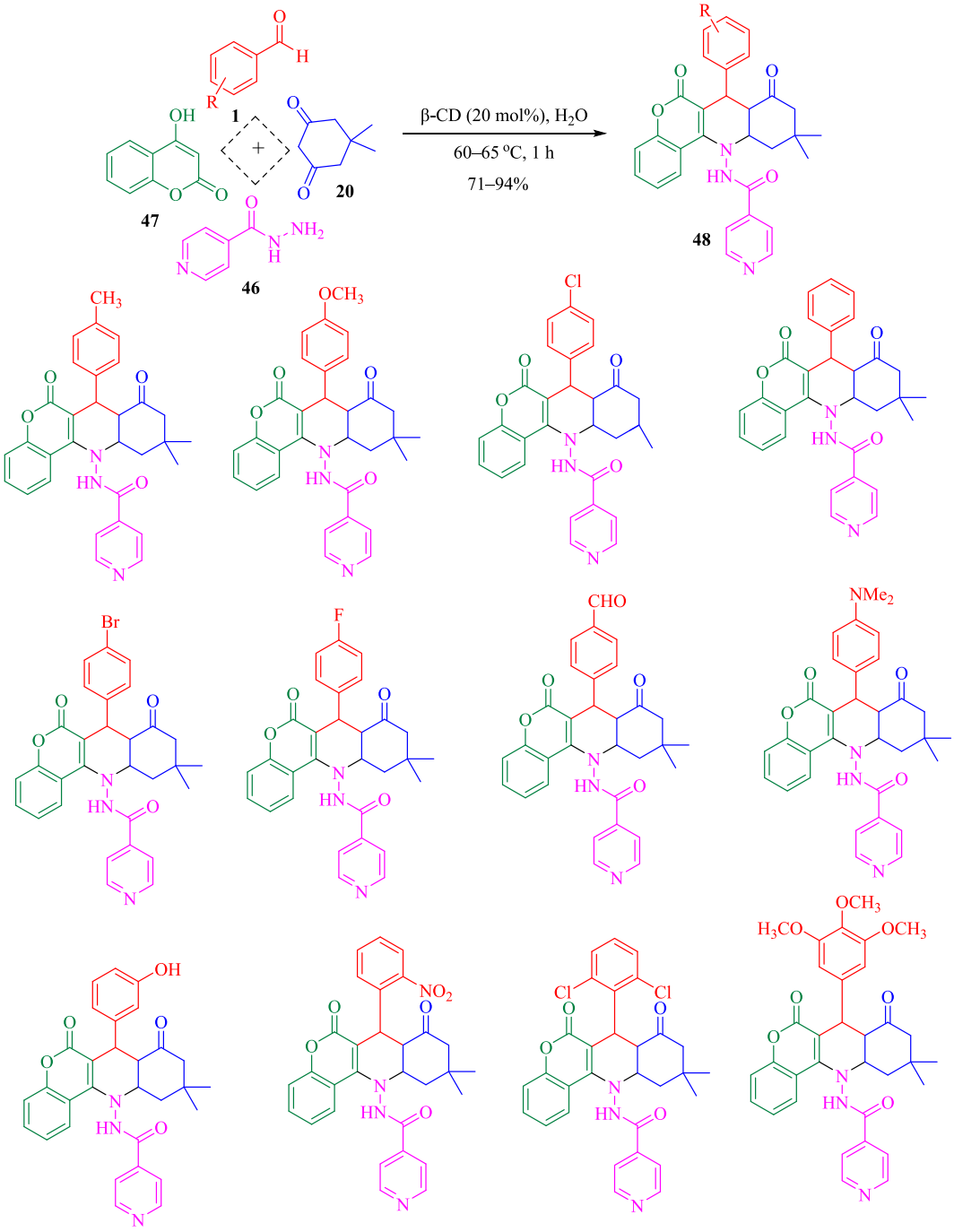
Scheme 35. Synthesis of different structurally spirooxindoles using Fe_3O_4/COS@β-CD-SO_3H NPs.

Table 6
Comparison between efficiency of Fe_3O_4/COS@β-CD-SO_3H NPs and other catalysts for the preparation of spirooxindoles **43**.

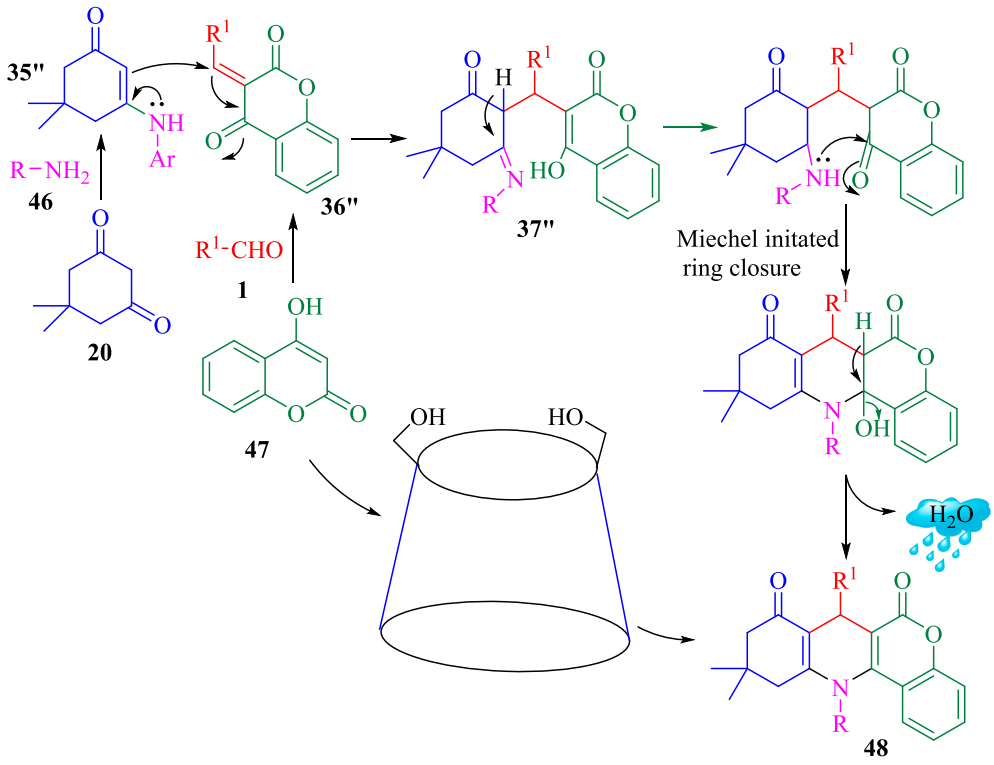
Entry	Catalyst	Time (min)	Solvent	Temperature (°C)	Yield (%)	Reference
1	I_2	60	H_2O	50	80	Kidwai et al., 2013
2	$Cu(OAc)_2{\cdot}H_2O$	240	–	80	86	Mohamadpour et al., 2016
3	Sodium stearate	180	H_2O	60	95	Wang et al., 2010
4	Fe_3O_4/COS@β-CD-SO_3H NPs	20	H_2O	50	98	Current discussed report



Scheme 36. Proposed mechanism for synthesis of spirooxindoles.



Scheme 37. The preparation of chromeno[4,3-*b*]quinolin-isonicotinamides by β-CD catalyst.



Scheme 38. Plausible reaction mechanism for this one-pot reaction.

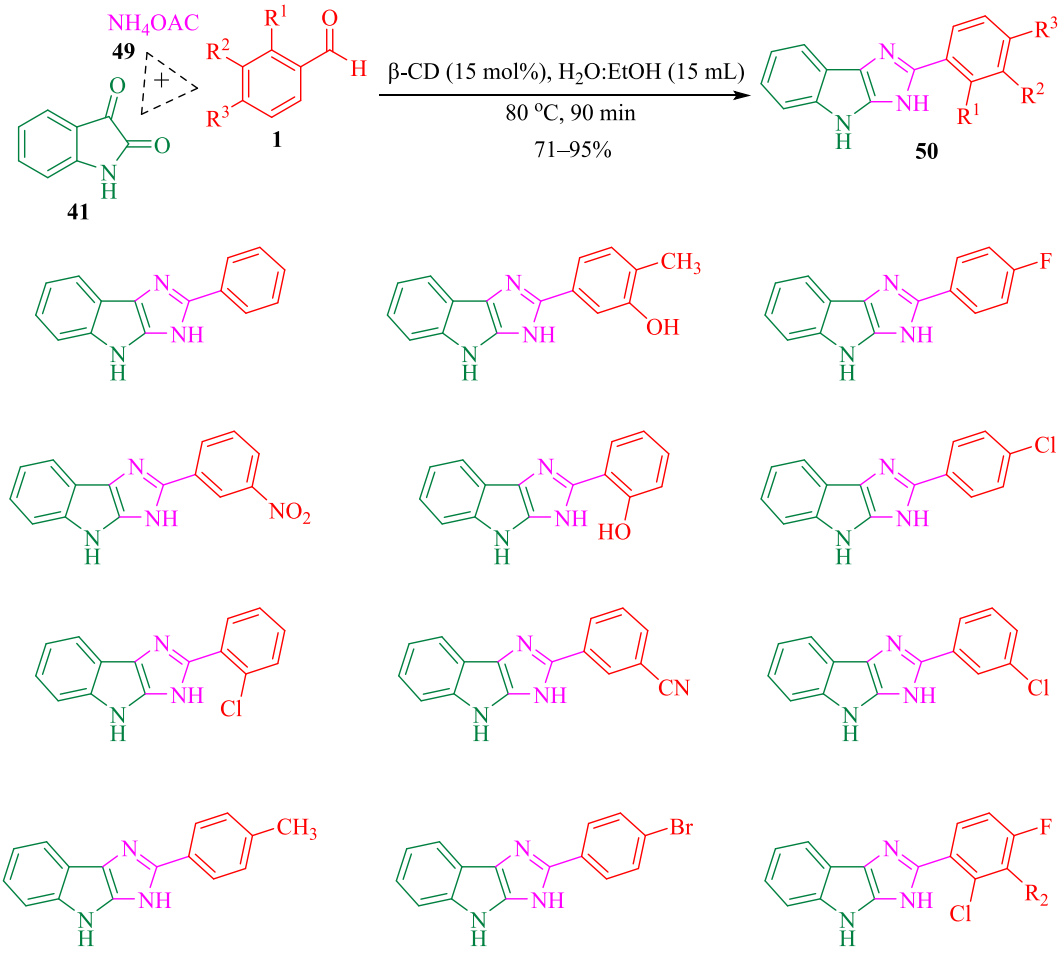
friendly strategy, no need for separation of products, and mild conditions.

In 2021, a simple, eco-friendly, and solvent-free approach for the Hantzsch reaction from aryl aldehyde **1**, dicarbonyl compounds (**20** and **9**), and ammonium acetate (**49**) was developed by Moheiseni *et al.* using [β-CD/Im](OTs)$_2$-silica as a catalyst (Moheiseni et al., 2021). This catalyst was prepared in the structure of a dicationic ionic liquid ([β-CD/Im](OTs)$_2$) and supported on the silica gel that the structure of [β-CD/Im](OTs)$_2$-Silica is displayed in Scheme 52. This new catalyst was characterized in various ways. SEM images of this nanocomposite catalyst revealed a layered structure with an enormous surface area. Three-component Hantzsch condensation of aryl aldehydes, ammonium acetate, and dimedone or ethyl acetoacetate was performed. Polyhydroquinolines **62** and 4-dihydropyridines **63** were obtained with excellent yield (84–98 %) (Scheme 53).

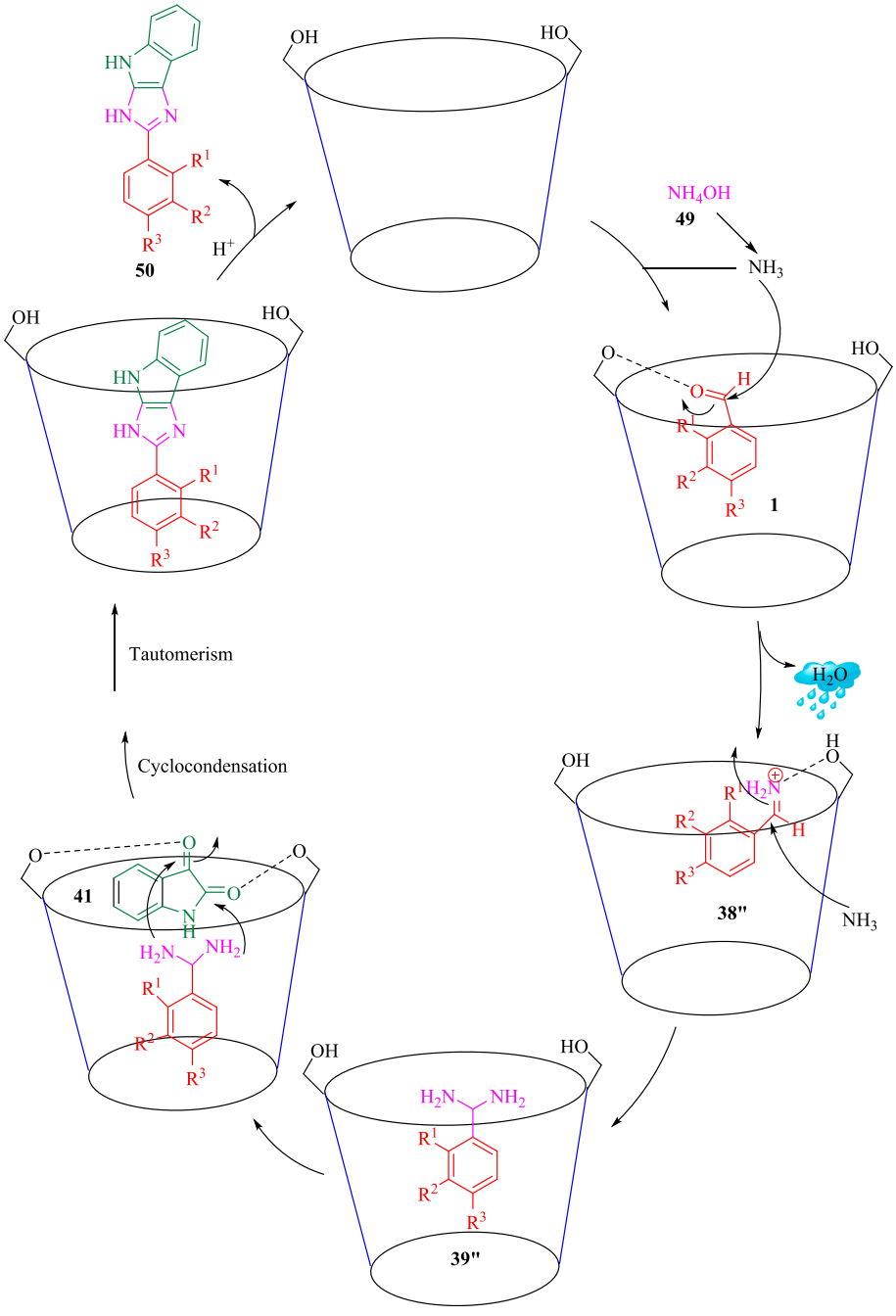
Catalyst reusability was conducted in the reaction of ammonium acetate, dimedone, and benzaldehyde, and the catalyst recovered four runs with a slight decline in performance. This protocol has advantages: short-time reaction, clean reactions, perfect yields, and green and reusable catalysts. The comparison of diverse catalysts for the Hantzsch coupling is shown in Table 8.

Ghalambaz *et al.* introduced MCM-41-β-CD/NH$_2$ (Scheme 54) as a heterogeneous catalyst for the high-efficiency synthesis of pyrans **64** through a one-pot, multicomponent process. The TCR of malononitrile (**42**), aryl aldehyde **1**, and 4-hydroxycoumarin (**47**) lead to the formation of pyrans in 76–88 % yield (Scheme 55) (Ghalambaz et al., 2021). This new catalyst has β-CD and amino basic units with pore channels that preparation through a surfactant-templated sol–gel approach. SEM, TEM, XRD, TGA, TGA, BET, and FT-IR analyzed this heterogeneous catalyst. The TEM image of MCM-41-β-CD/NH$_2$ displayed a particle size of around 200 nm. This nanocatalyst was recycled for five runs without loss of activity. The method has several advantages, including being environmentally friendly, reusability of catalyst, good yields of pyran heterocycle, and short reaction times.

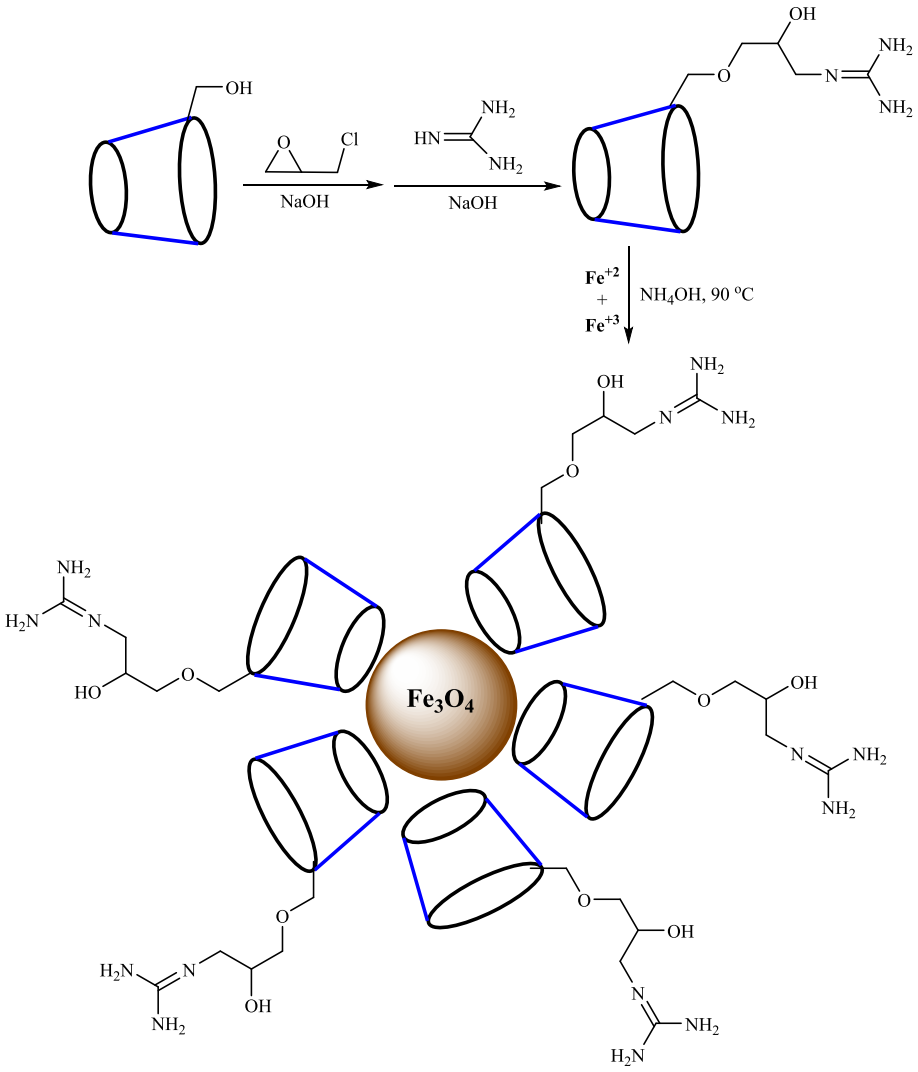
Mitra *et al.* explained a metal-free and green protocol for the preparation of 2-amino-4,6- diphenylnicotinonitriles **66** from aryl aldehydes **1**, acetophenone (**65**), malononitrile (**42**), and ammonium acetate (**49**) (Scheme 56) (Mitra et al., 2021) and 2,3-dihydroquinazolin-4(1*H*)-ones **68** from aryl aldehydes **1**, isatoic anhydride (**67**), and ammonium acetate (**49**) (Scheme 57) *via* one-pot, MCRs using β-CD as a recoverable catalyst in water and solvent-free conditions. The 2-amino-4,6-diphenylnicotinonitriles and 2,3-dihydroquinazolin-4(1*H*)-ones were attained in good to high yield (75–94 %). The critical features of this method are that it is an inexpensive, greener protocol without metal catalysts or toxic acid. The authors offered probable mechanisms of both reactions that are shown in Scheme 58 (formation of the pyridine motif) and Scheme 59 (construction of the 2,3-dihydroquinazolin-4(1*H*)-ones). β-CD as a catalyst activates the aryl aldehyde **1** and acetophenone compound **65** as the active electrophile species. The reaction of malononitrile **42** and ammonium acetate **49** with these two activated electrophiles provides the corresponding intermediates **48″** and **49″**, respectively. Then, the reaction between these **48″** and **49″** intermediates will create the corresponding intermediate **50″**. After the sequence of tautomerization, cyclization, and again tautomerization



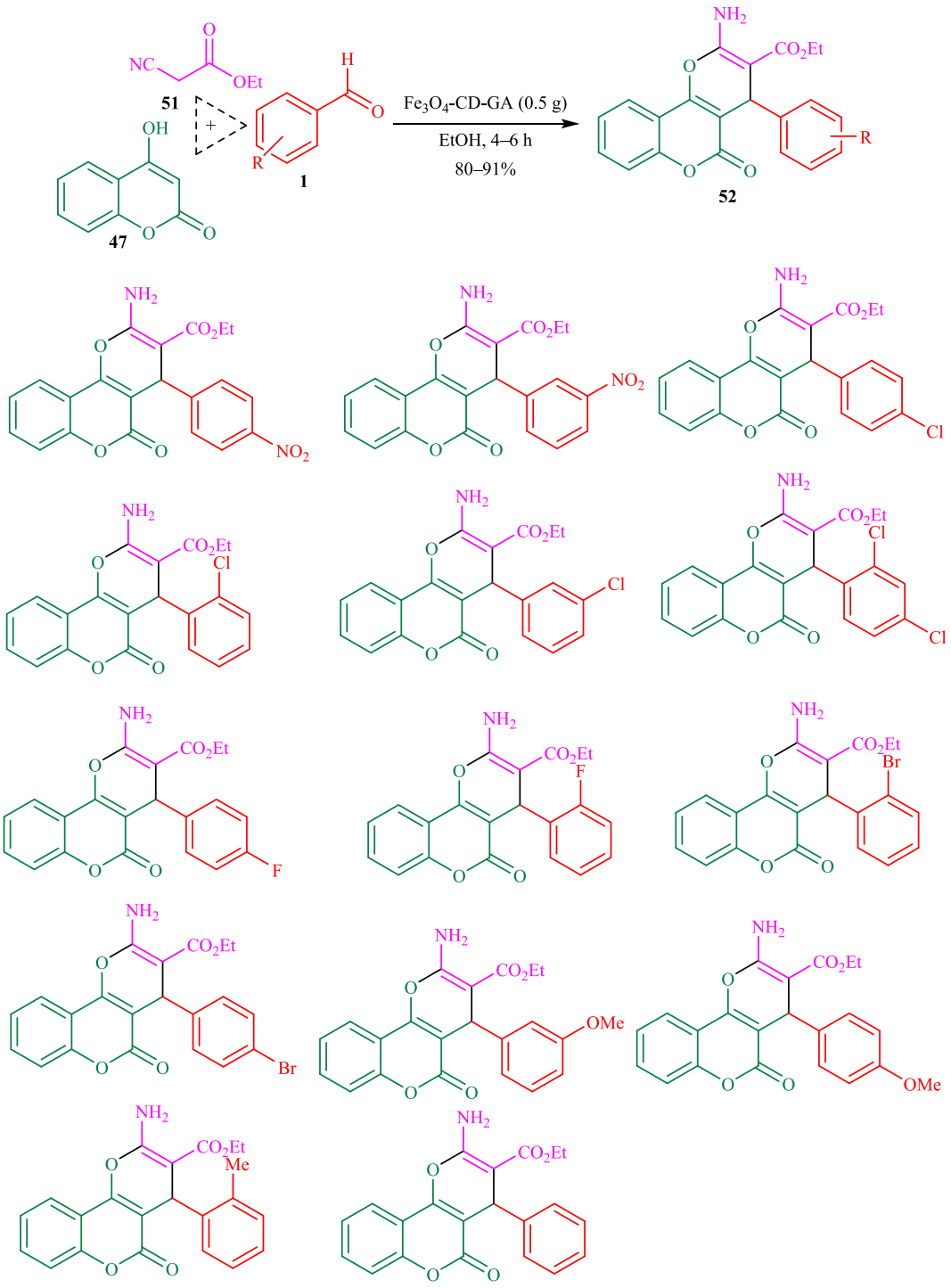
Scheme 39. Synthesis of imidazo[2,3-*b*]indoles.



Scheme 40. Possible reaction mechanism.



Scheme 41. Schematic representation of the synthesis of magnetic nanocatalyst.

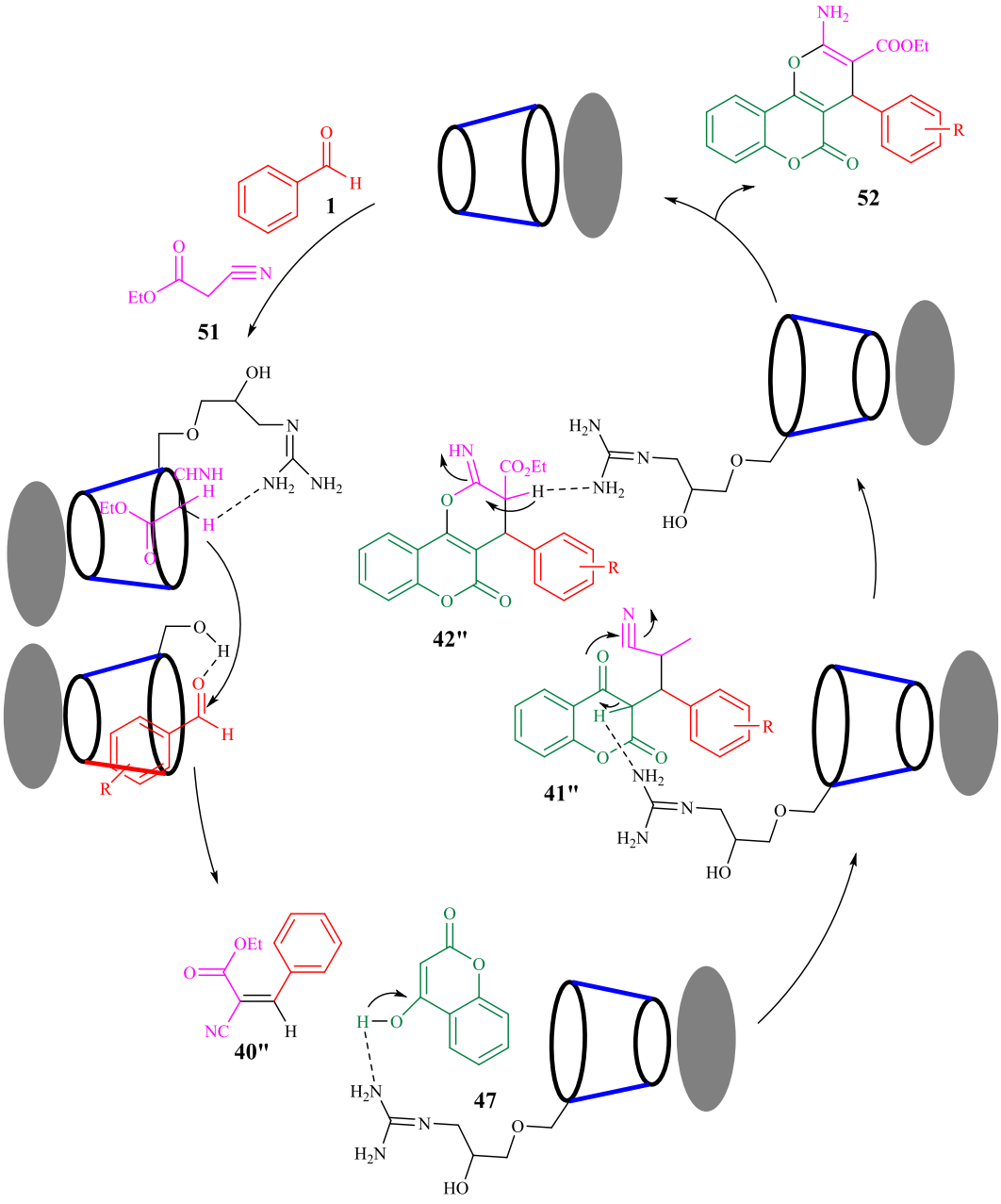


Scheme 42. Fabrication of dihydropyrano[3,2-*c*]chromenes using Fe_3O_4-β-CD-GA.

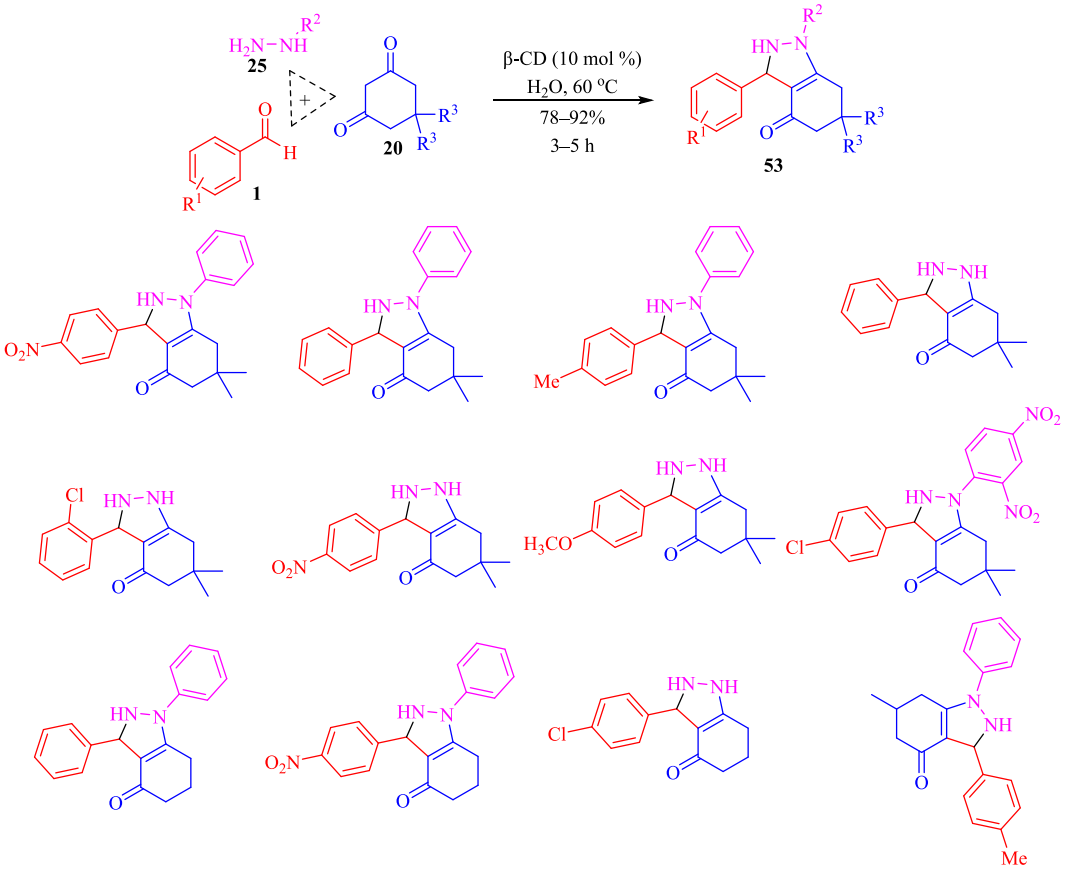
Table 7
Study of catalyst activity of various amounts of catalyst for the synthesis **52**.

Entry	Catalyst (mg)	Time (min)	Yield (%)
1	Fe_3O_4-β-CD-GA (50)	80	89
2	Nano Fe_3O_4 (40)	180	30
3	β-CD (190)	180	42
4	Guanidine (10)	180	58

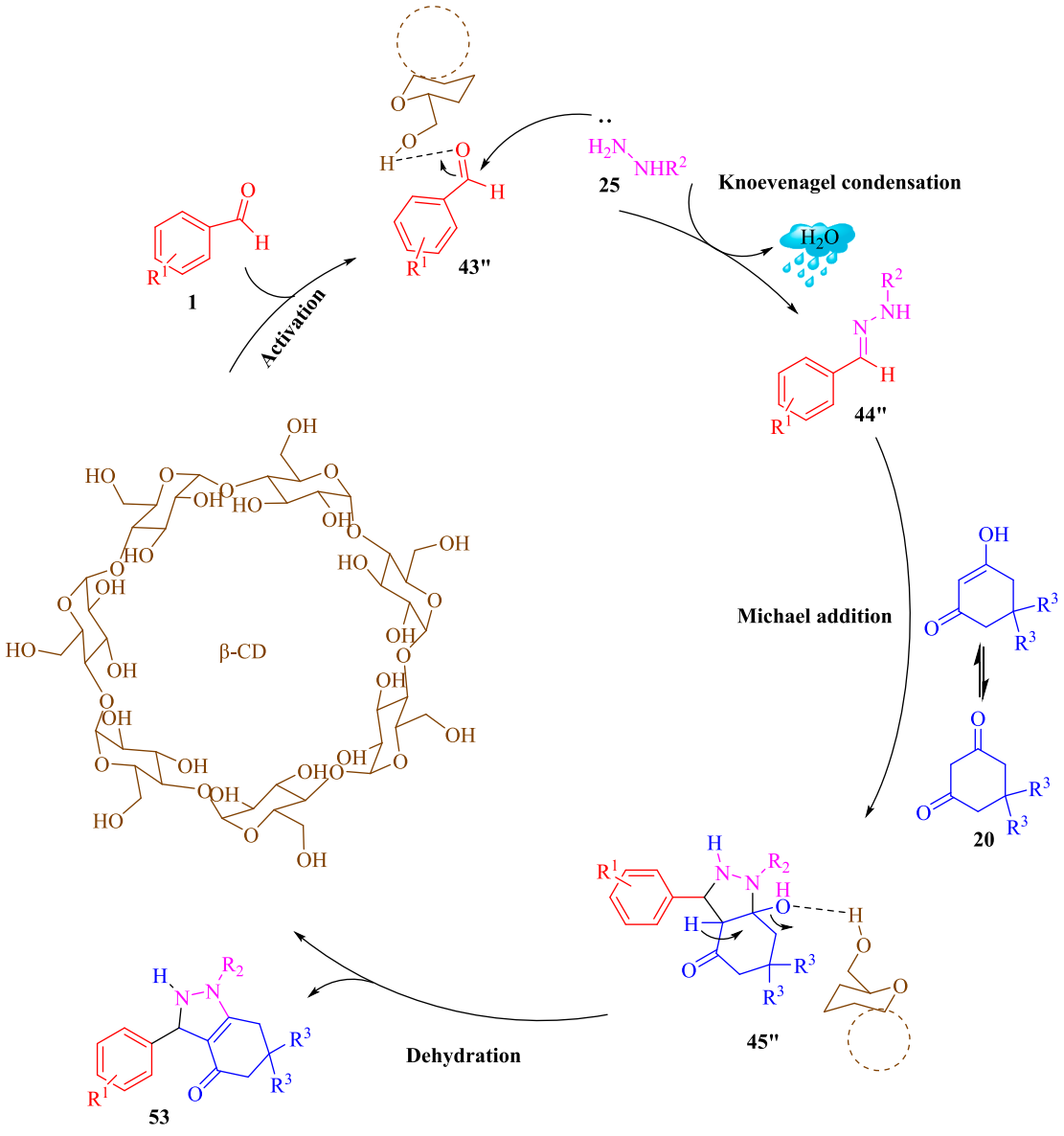
generates the product **66** (Scheme 58). In the first step, isotonic anhydride **67** coordinates with the β-CD cavity; the reaction of ammonium acetate (**49**) forms a **51**″ intermediate, which is then isolated. The following step facilitates the nucleophilic attack by the electron-rich nitrogen of the NH_2 group to the electrophilic carbonyl carbon center of aldehydes **1**, which β-CD activates. Then, the elimination of H_2O, followed by another nucleophilic attack by the NH_2 group of the amide to the carbon center on the substituted imine **52**″ leads to the expected product **68** (Scheme 59).



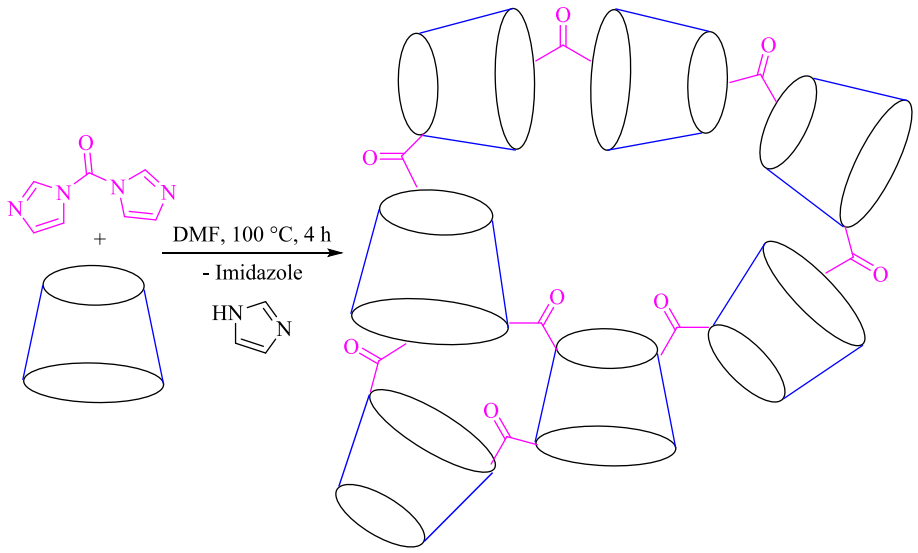
Scheme 43. Proposed reaction pathway.



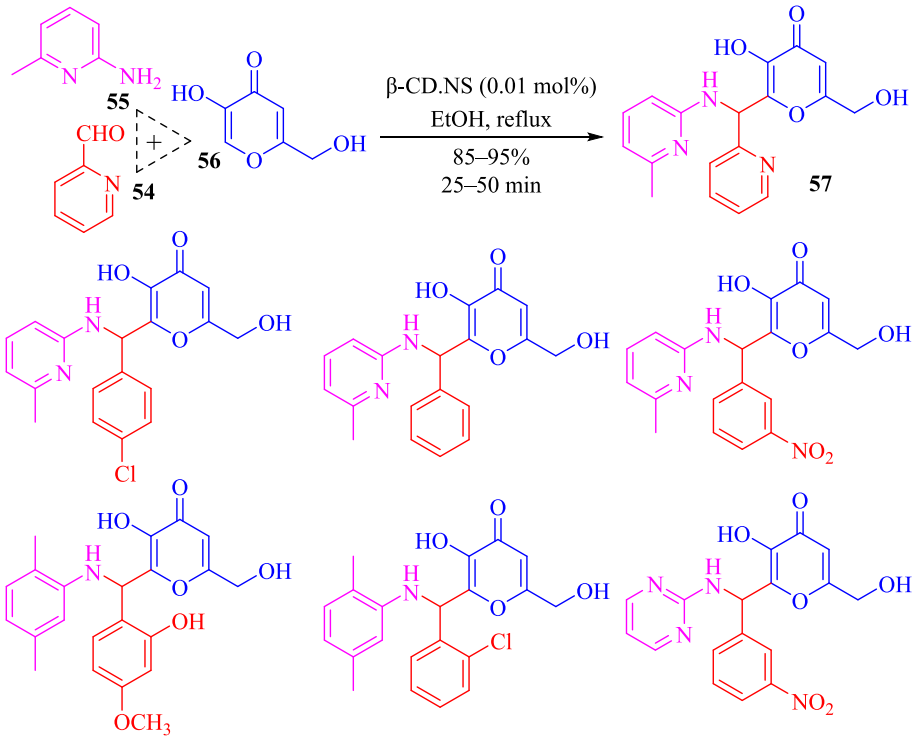
Scheme 44. Synthesis of hexahydro-4*H*-indazole-4-ones **53**.



Scheme 45. A convenient mechanism for synthesizing hexahydro-4*H*-indazol-4-ones.

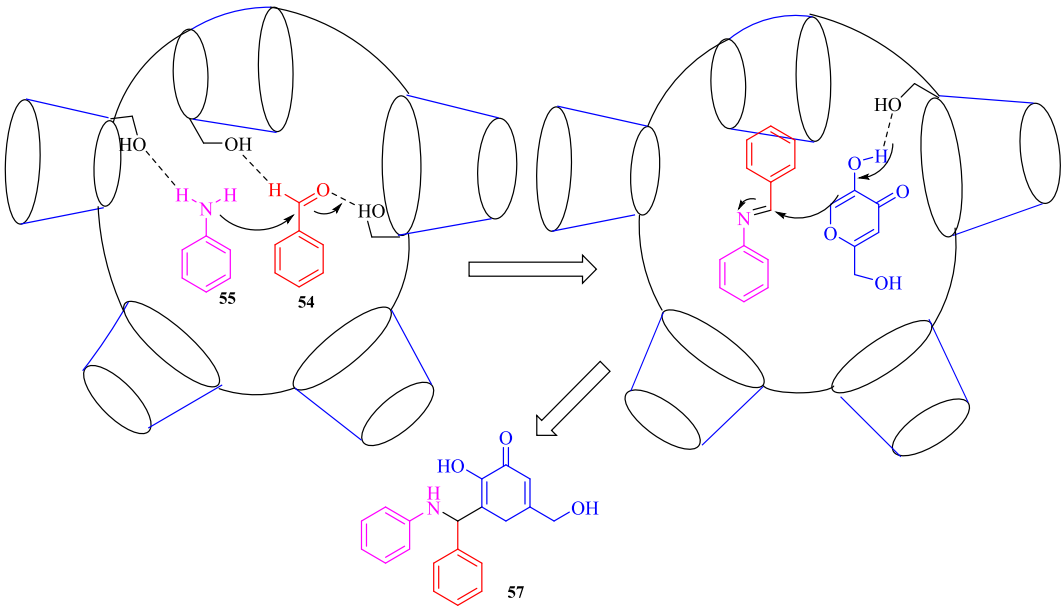


Scheme 46. Synthesis of β-CD-based nanosponges.



Scheme 47. Synthesis of aryl amino kojic acid by β-CD.NS as a catalyst.

In 2022, Jadhav and co-workers designed an efficient approach for synthesizing 2-amino-4*H*-pyranoquinolines **70** from TCR of aryl aldehydes **1**, 8-hydroxyquinoline (**69**) and malononitrile (**42**), or ethyl cyanoacetate by β-CD as a robust catalyst in water under ultrasound irradiation (Jadhav et al., 2022). The 2-amino-4*H*-pyranoquinolines were provided in 84–93 % yield (Scheme 60). They presented a gram-scale synthetic method for synthesizing 2-amino-4*H*-pyranoquinolines with perfect results. β-CD was recovered in four cycles with no notable loss in activity. Using water (a greener solvent), recycling the catalyst, and synthesizing pyranoquinolines, essential in pharmaceutical chemistry, are the main advantages of this work. The suggested pathway for the reaction is displayed in Scheme 61. Firstly, the Knoevenagel



Scheme 48. The mechanistic pathway represents the synthesizing of amino Kojic acid derivatives.

condensation of the aryl aldehyde **1** with the active methylene compound **42** generates the arylidene adduct **53**″. Then, Michael's addition of **69** to the Knoevenagel adduct **53**″ yields an intermediate **54**″, which undergoes aromatization followed by intramolecular cyclization facilitated by β-CD to the C–N triple bond to provide the cyclic intermediate **55**″ *via* intermediate **54**″. Ultimately, **70** products are created in situ by tautomerization of the imino group to an amino group.

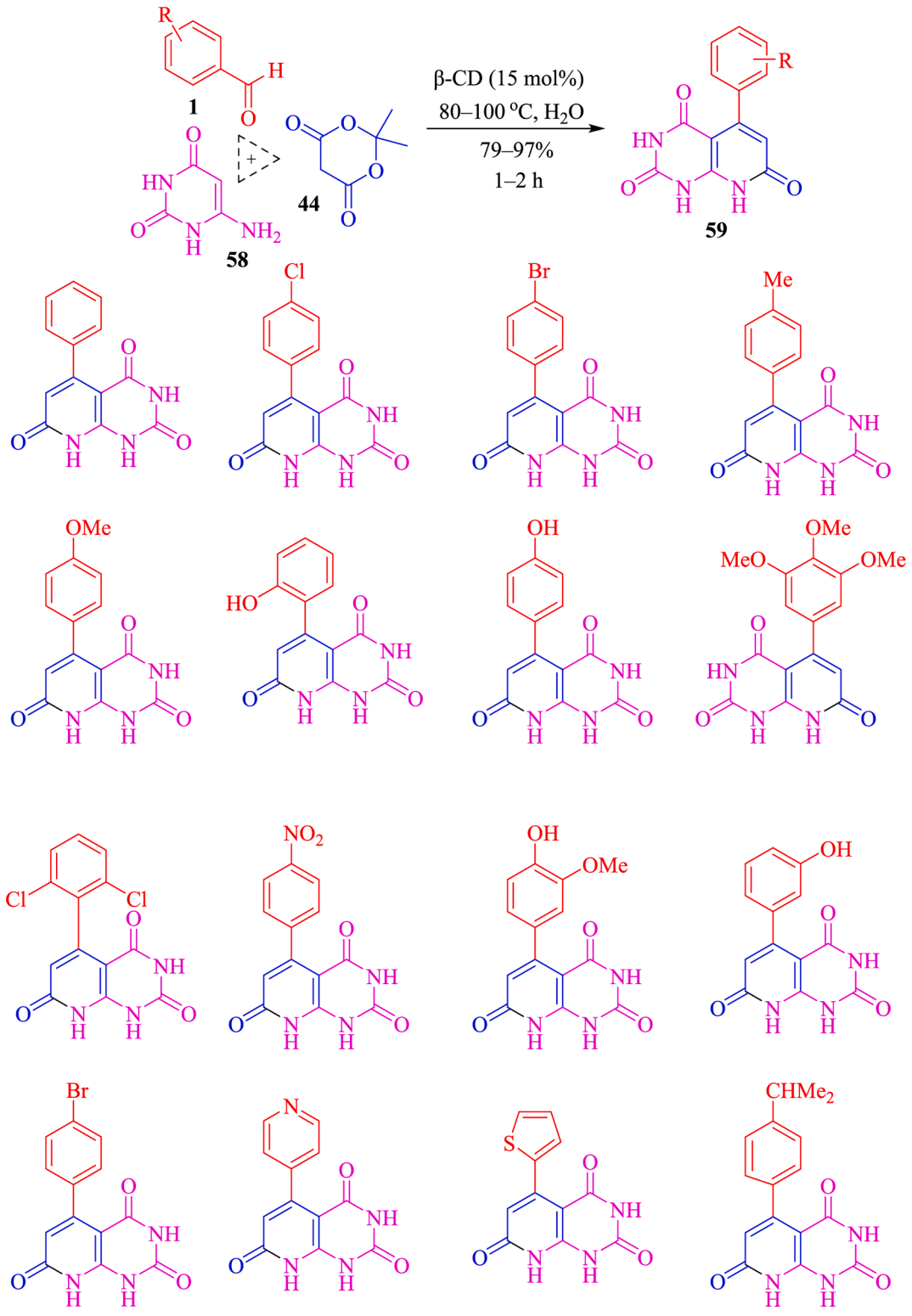
During the same years, Wang *et al.* used β-CD-SO_3H as a reusable catalyst for synthesizing tetrahydrobenzo [4,5] imidazo[2,1-*b*] quinazolin-1(2*H*)-ones in water (Wang et al., 2022). This approach is efficient and green for the preparation of tetrahydrobenzo[4,5]imidazo[2,1-*b*] quinazolin1(2*H*)-ones series in high yields through a MCR using β-CD-SO_3H under mild status. The best procedure for synthesizing these compounds is the high product yields, higher atom efficiency, and reusability of β-CD-SO_3H.

Liao *et al.* explained the preparation of indeno[1,2-*b*]quinoxalines **73** from *o*-phenylenediamine (OPD, **71**) and 2-indanones **72** using β-CD as an excellent catalytic (Scheme 62) (Liao et al., 2022). Optimization of the reaction status was studied, and the optimal catalytic consisted of β-CD (15 mol%) in H_2O at room temperature within 12 h. This approach is milder, easier, and slightly toxic than previous methods, leading to an eco-friendly option. The β-CD can be recycled and reused for four consecutive runs with a slight drop in activity. The superior advantages of the current method are excellent yields, environmental friendliness, reusable catalyst, low cost, and a broad substrate scope, making it a strong strategy for synthesizing indeno[1,2-*b*]quinoxalines. The mechanism proposed for the reaction is displayed in Scheme 63. First, OPD is included in the cavity of β-CD to create the OPD-β-CD inclusion complex. Then, the first Schiff base reaction happens, releasing a mole of H_2O to form an intermediate **56**″. The generated intermediate **56**″ is immediately oxidized by oxygen in the O_2 to form the intermediate **57**″, leading to the second Schiff base reaction. After cyclization and removal, a mole of H_2O product **73** was generated.

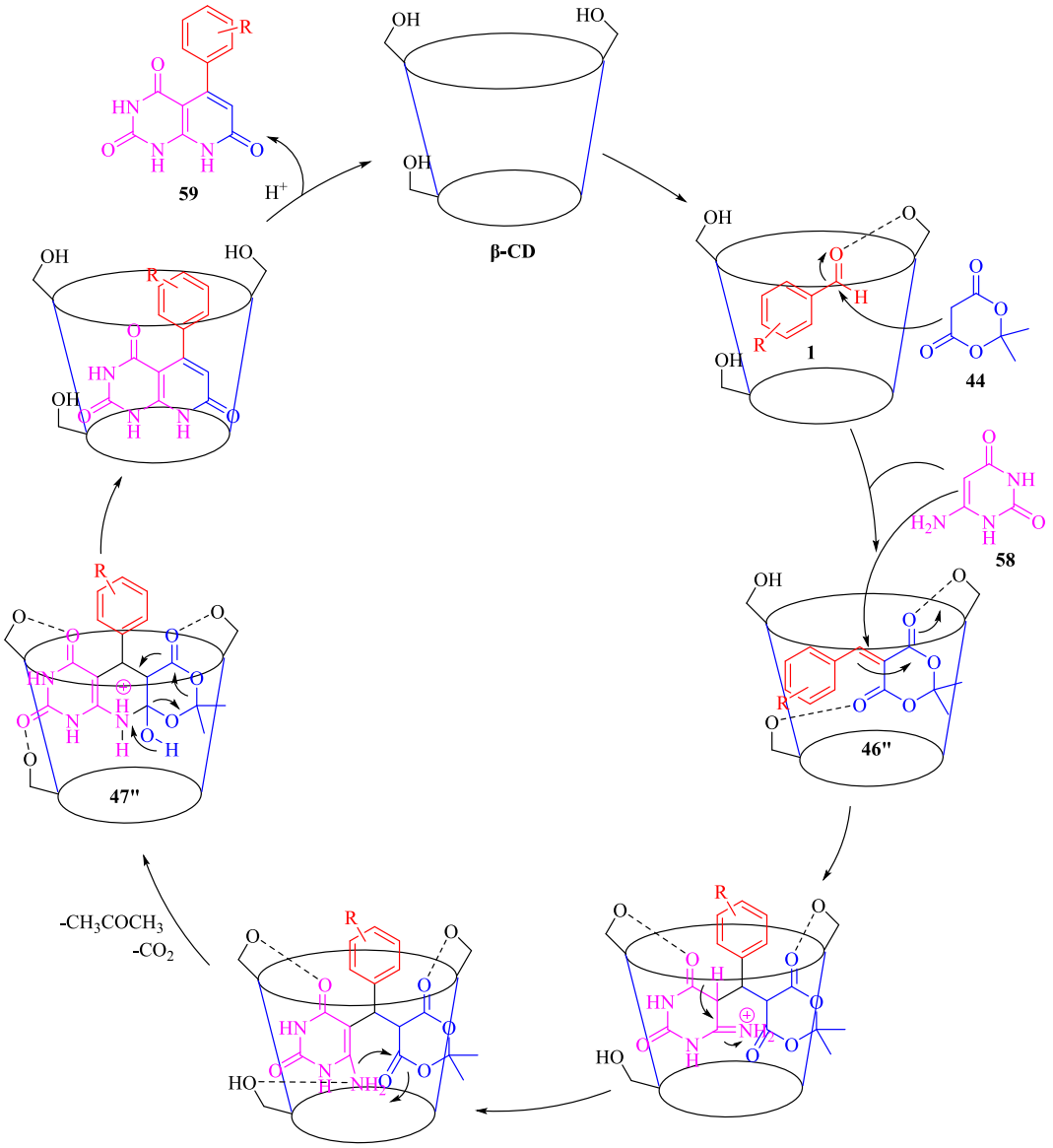
Xu's group illustrated a suitable procedure for forming spiroindolines by β-CD-SO_3H in water at 50 °C. Under mild conditions, a one-pot, TCR of various isatin **41**, diketones **74**, and malononitrile (**42**) was carried, and spiroindoline products **75** were obtained in 38–99 % yields (Scheme 64) (Xu et al., 2022). β-CD-SO_3H displayed high catalytic activity that was successfully recycled five times with no notable effect on the product outcomes. A comparison of catalytic performance β-CD–SO_3H with the formerly reported catalysts for the synthesis of spiroindoline **75** is shown in Table 9.

Mohamadpour designed a co-friendly three-compound method for synthesizing the 2-amino-4*H*-chromene frameworks **77** by *Knoevenagel-Michael* cyclocondensation under solvent-free in perfect yield (80–96 %) (Mohamadpour, 2022a, 2022b). The reaction of aryl aldehydes **1**, malononitrile (**42**), and resorcinol (**76**) was developed by per-6-amino-β-CD as a helpful catalyst at room temperature (Scheme 65). This catalyst is stable enough for six successive runs without a remarkable decline in structure and activity. This approach has the advantages of green and solvent-free reaction conditions, easy workup, superior yields, and recoverable catalysts. The suggested path for synthesizing 2-amino-4*H*-chromene scaffolds is indicated in Scheme 66. Knoevenagel condensation occurs between active methylene **42** compound and aryl aldehyde 1leading to intermediate **58**″. Per-6-NH_2-β-CD also catalyzed the resorcinol **76** attack on intermediate **58**″ as Michael acceptor to provide **59**″, which, after cyclizing and tautomerizing, aims the objective products **77**.

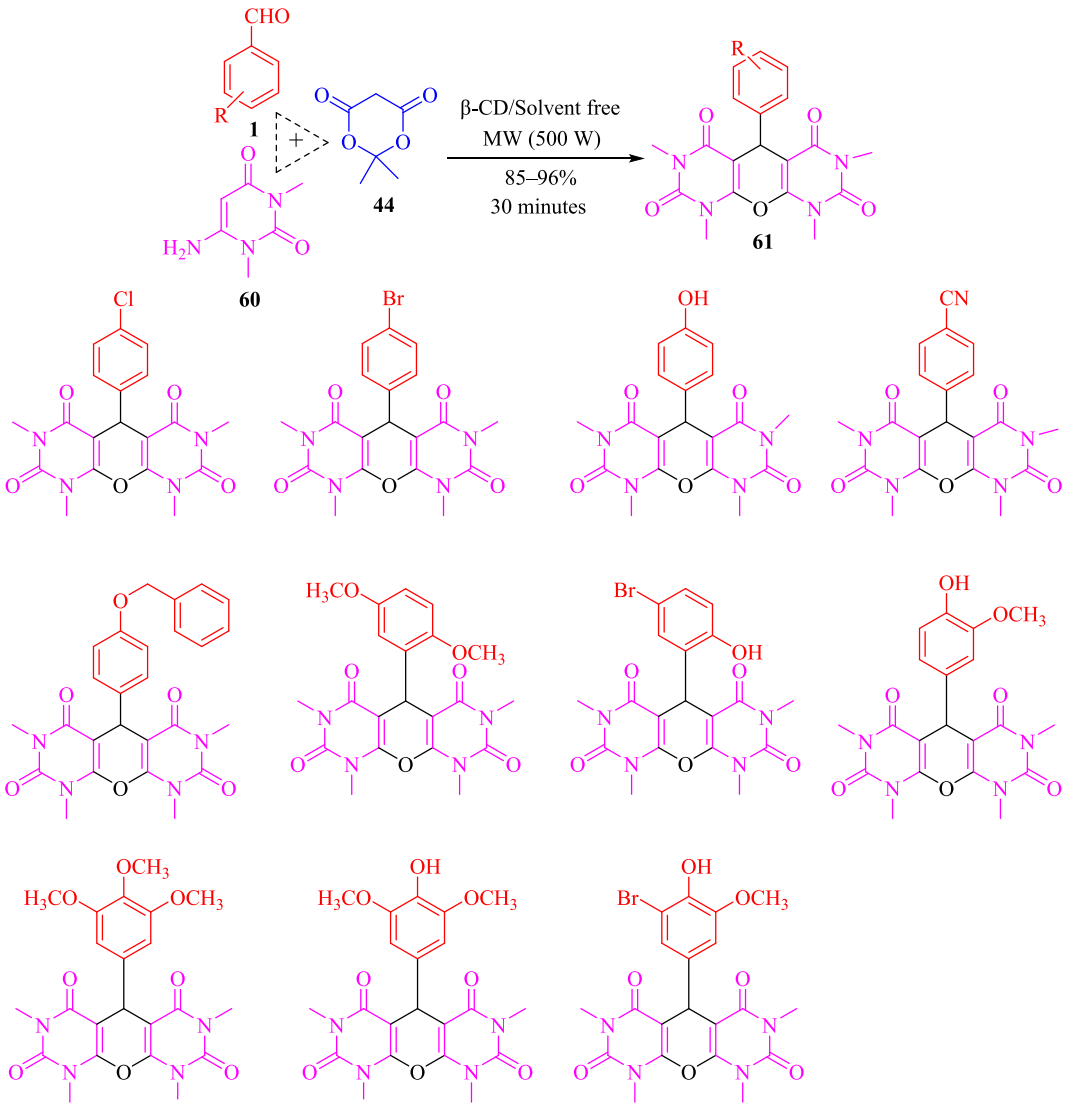
A highly effective, suitable approach has been designed for synthesizing pyrano[2,3-*d*]pyrimidines **78** by β-CD as a significant catalyst in water media published by the same researcher (Mohamadpour, 2022a, 2022b). Three-component Knoevenagel-Michael addition cyclo condensation reaction of malononitrile (**42**), aryl aldehydes **1**, and BA/1,3-dimethylbarbituric acid **16** for the preparation of pyrano[2,3-*d*]



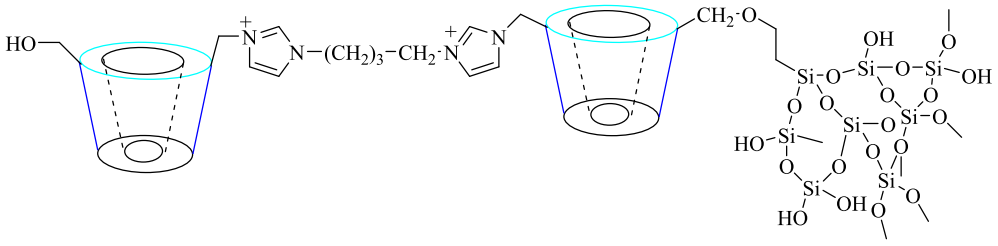
Scheme 49. The formation of 5-phenyl-5,6-dihydropyrido[2,3-*d*]pyrimidine-2,4,7(1*H*,3*H*,8*H*)-triones **59** using β-CD.



Scheme 50. The possible way for the compounds **59** using β-CD.

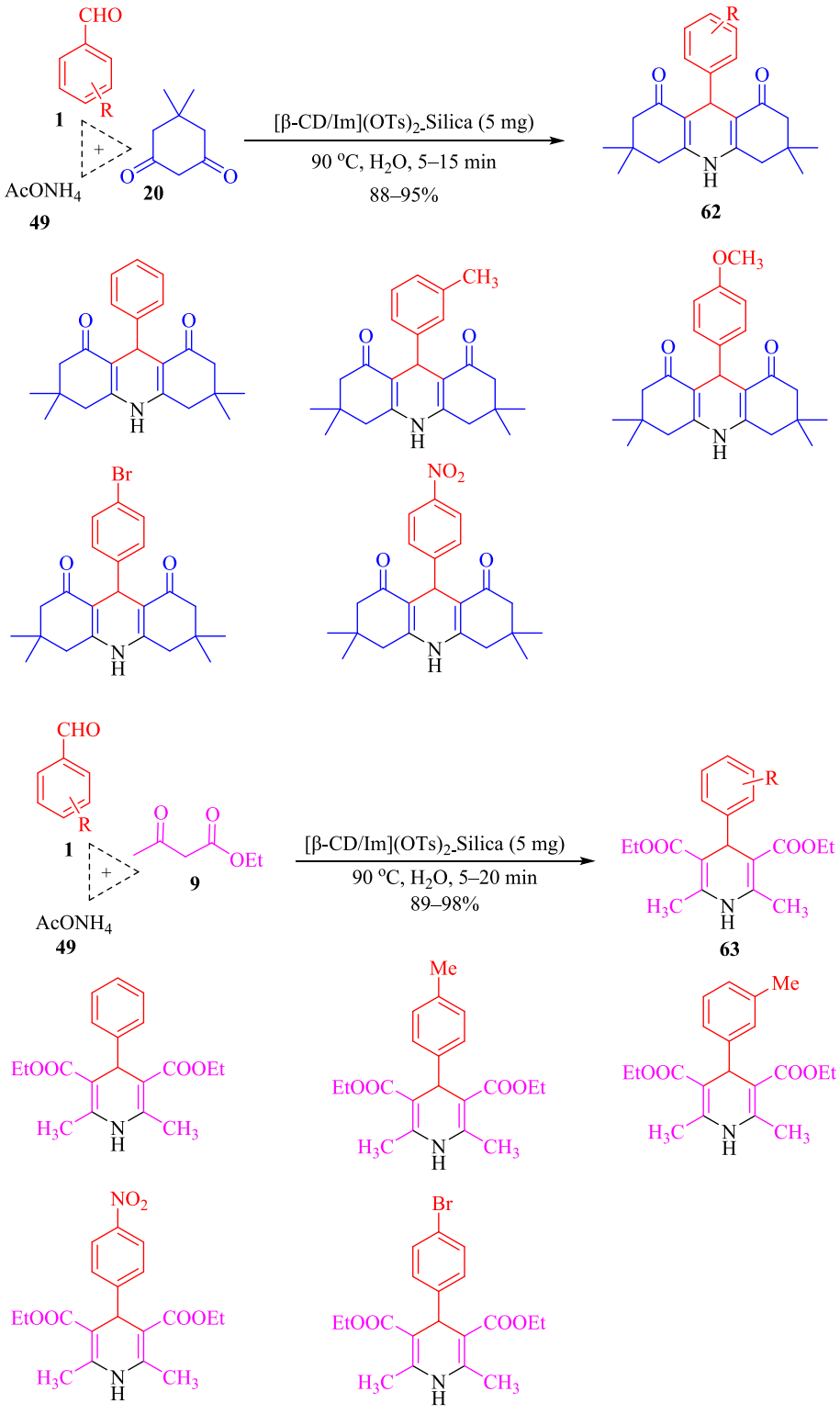


Scheme 51. The provision of pyrano[2,3-*d*:6,5-*d*′]pyrimidines.



Scheme 52. The structure of [β-CD/Im](OTs)$_2$-Silica.

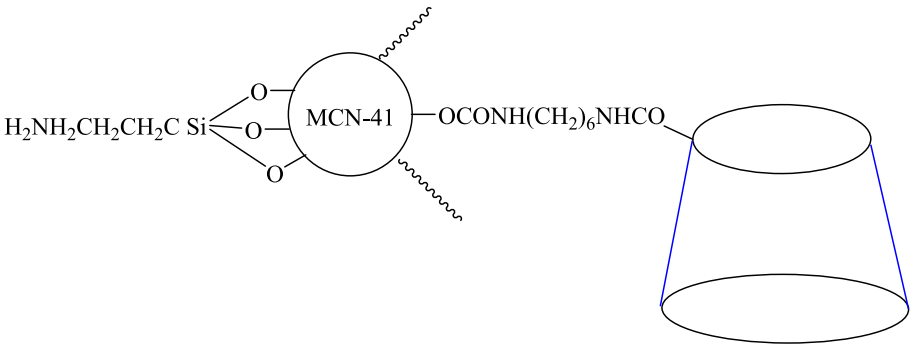
pyrimidines were carried out and expected products obtained in 76–95 % yields (Scheme 67). The catalytic recyclability of β-CD was studied so that the catalyst could be recovered three times without a considerable drop in activity. β-CD is a highly stable catalyst that, after reaction, the structure of it doesn't change. The use of a biodegradable catalyst, high catalyst stability, easy workup, avoidance of toxic solvents, and catalyst recyclability are several of the principal features of this work. The proposed mechanism for the Knoevenagel–Michael–cyclo condensation is indicated in Scheme 68. The intermediate cyano olefin **61**″ was readily created in situ from Knoevenagel condensation between β-CD



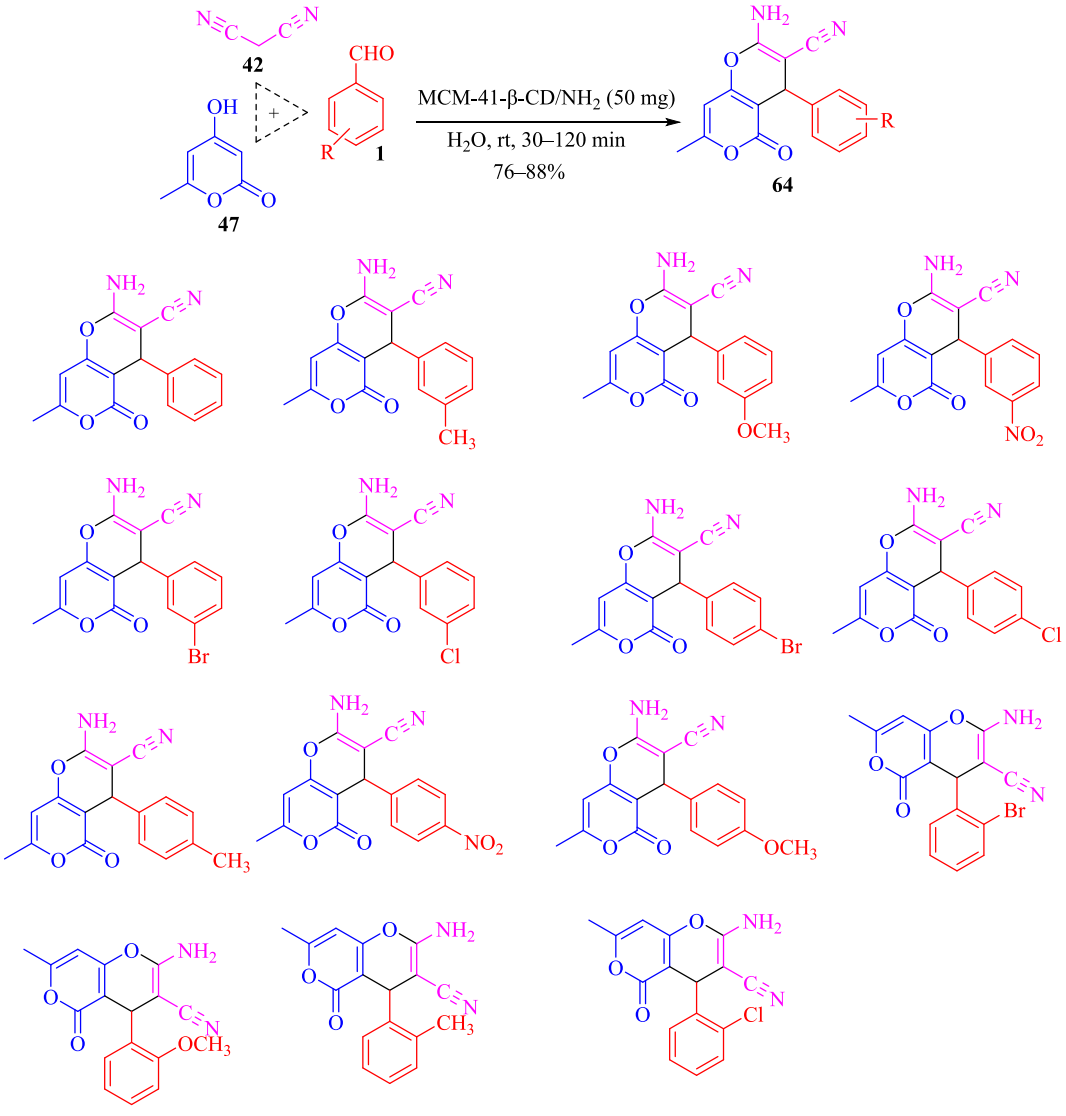
Scheme 53. Synthesis of 1,4-dihydropyridines **62** and **63**.

Table 8
Comparison of different catalysts for the Hantzsch coupling.

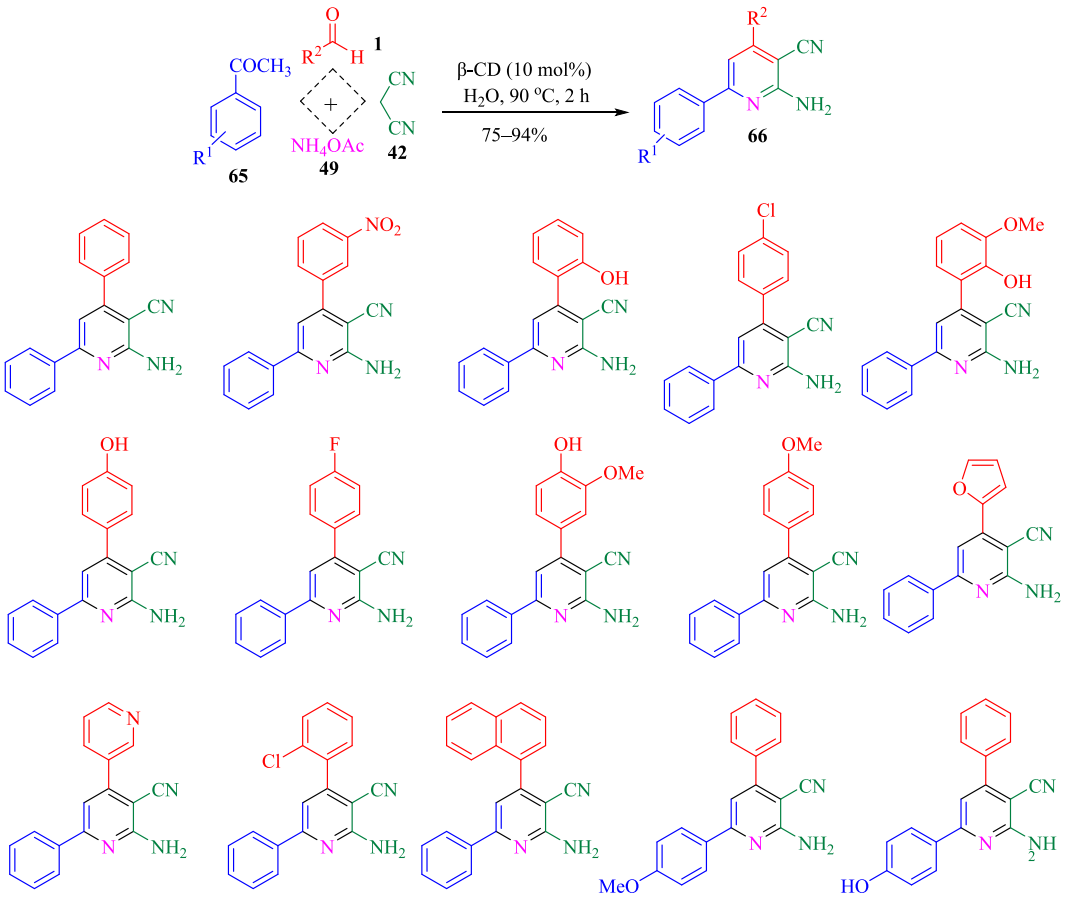
Entry	Catalyst/condition	Catalyst loading (mg)	Time (min)	Yield (%)	Reference
1	Silica gel/$NaHSO_4$	60	6 h	85	Igder et al., 2015
2	$HClO_4$-SiO_2/80 °C	50	20	95	Maheswara et al., 2006
3	CAN	28	60	92	Ko and Yao, 2006
4	L-Proline	115	30	95	Kumar and Maurya, 2007
5	[β-CD/Im](OTs)$_2$-Silica	5	15	98	Current discussed report



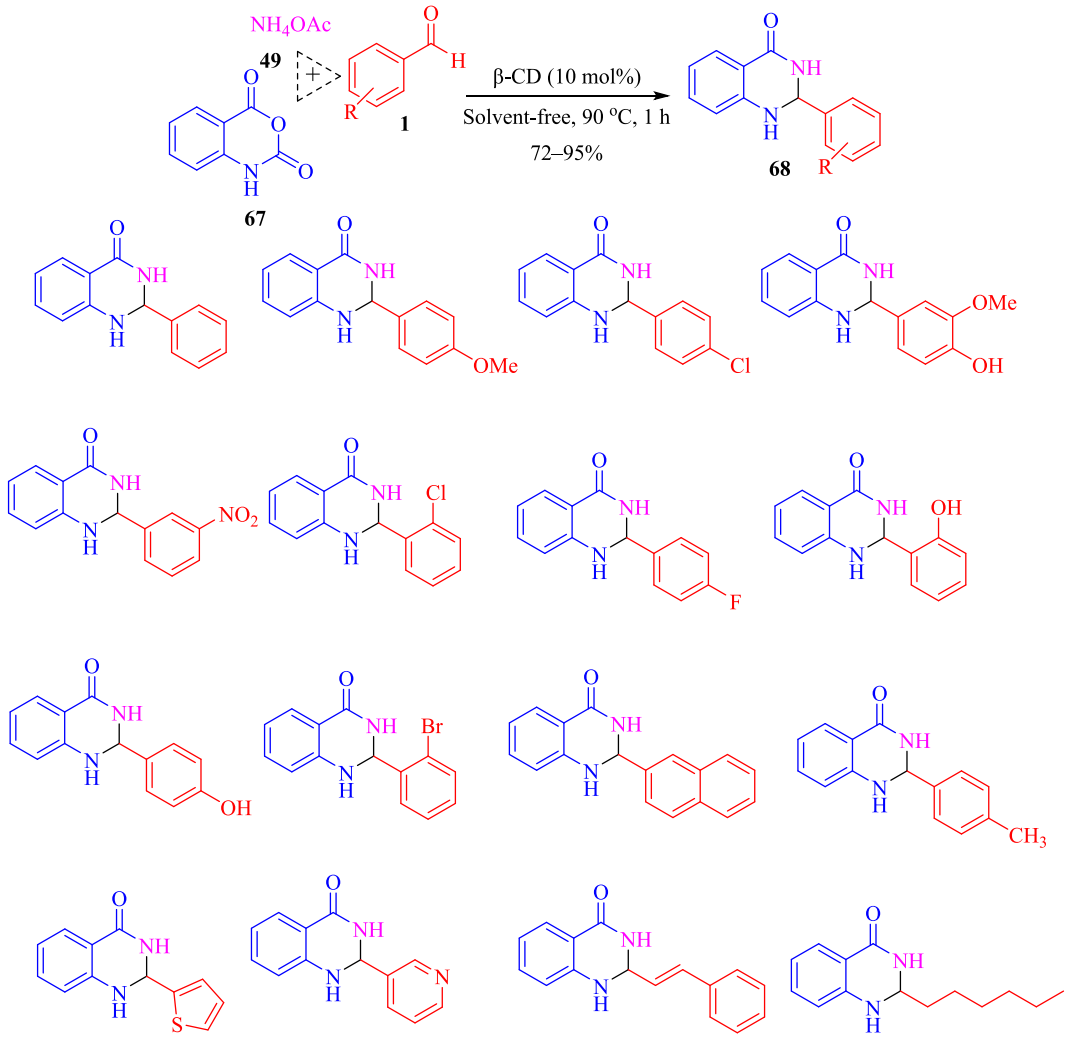
Scheme 54. The structure of MCM-41-β-CD.NH_2.



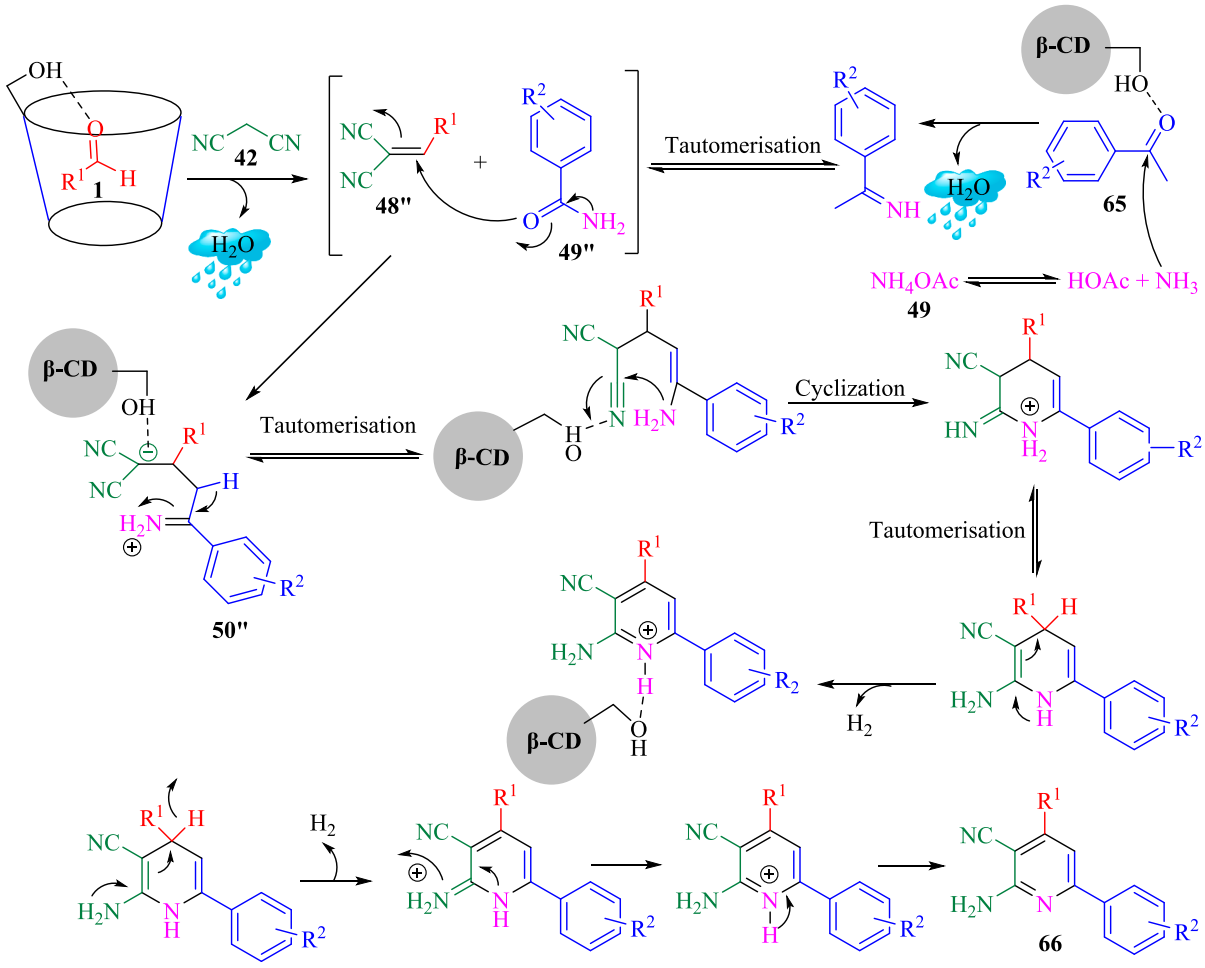
Scheme 55. Synthesis of various pyran derivatives by MCM-41-β-CD/NH_2.



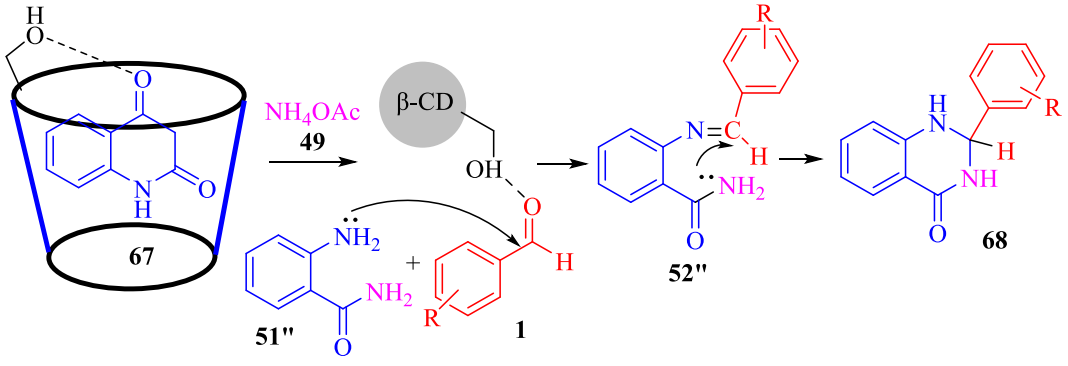
Scheme 56. Preparation of diversified 2-amino-4,6-diphenylnicotinonitriles.



Scheme 57. Fabrication of the 2,3-dihydroquinazolin-4(1*H*)-ones.



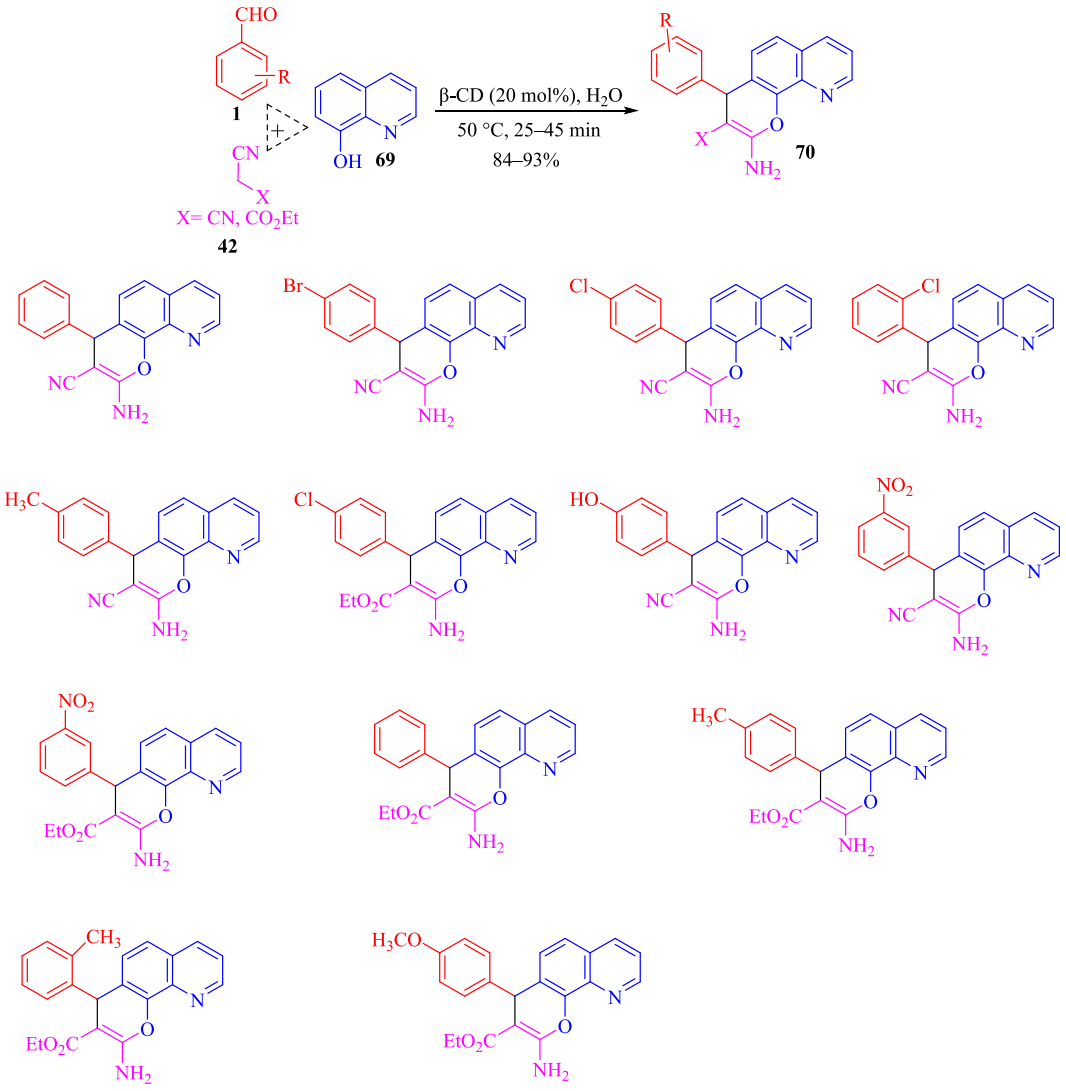
Scheme 58. The suitable pathway for synthesizing pyridine motif using β-CD catalyst.



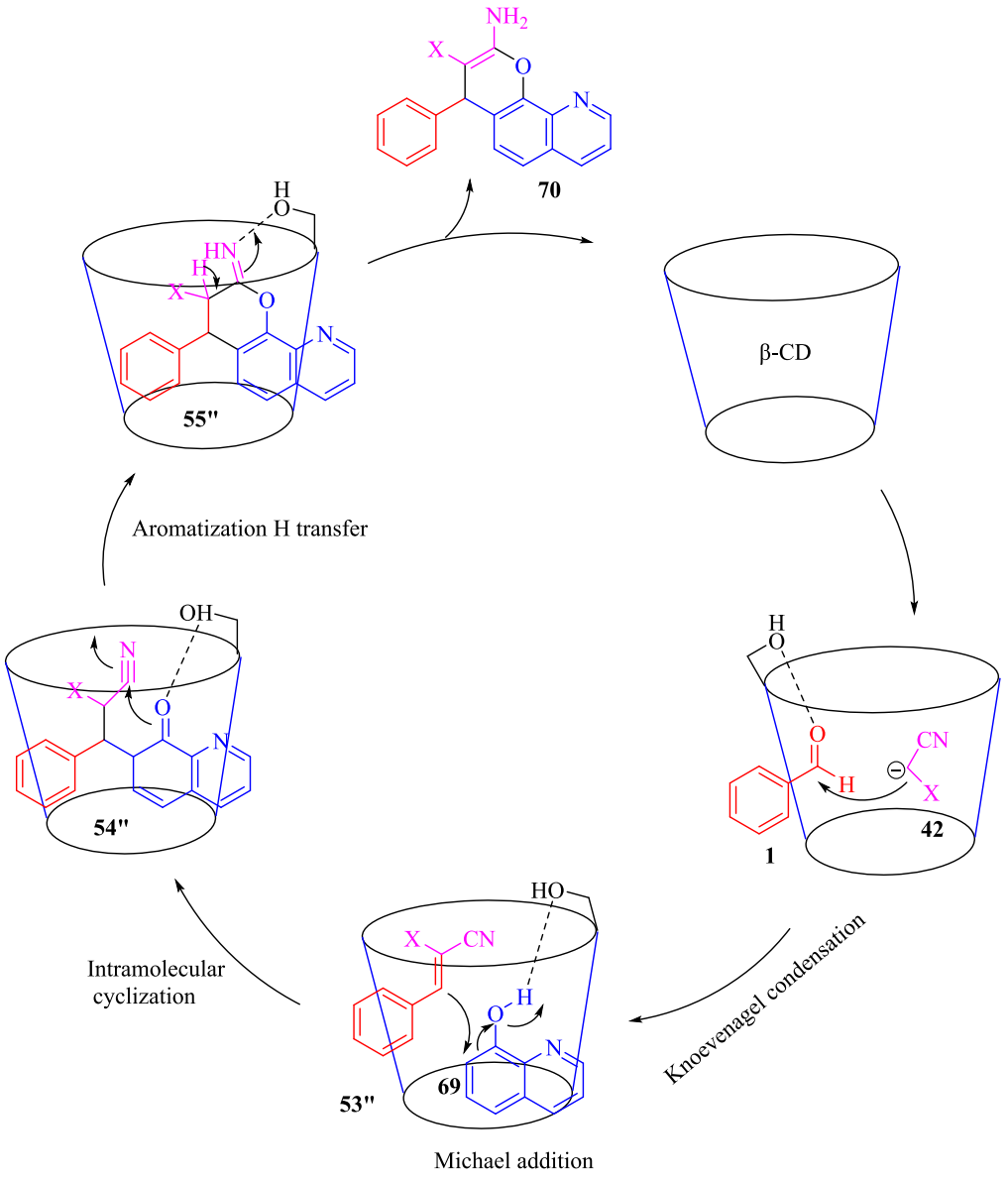
Scheme 59. The stepwise synthesis pathway of 2,3-dihydroquinazolin-4(1*H*)-ones.

solubilized aryl aldehyde **1** and active methylene compound **42** in H_2O. β-CD also catalyzed the construction of the enolic form of barbituric acid **16,** which could readily react with cyano olefin **61**″ and provide intermediate **62**″, followed by cyclization and tautomerization of **63**″ and **64**″ afford the products **78.**

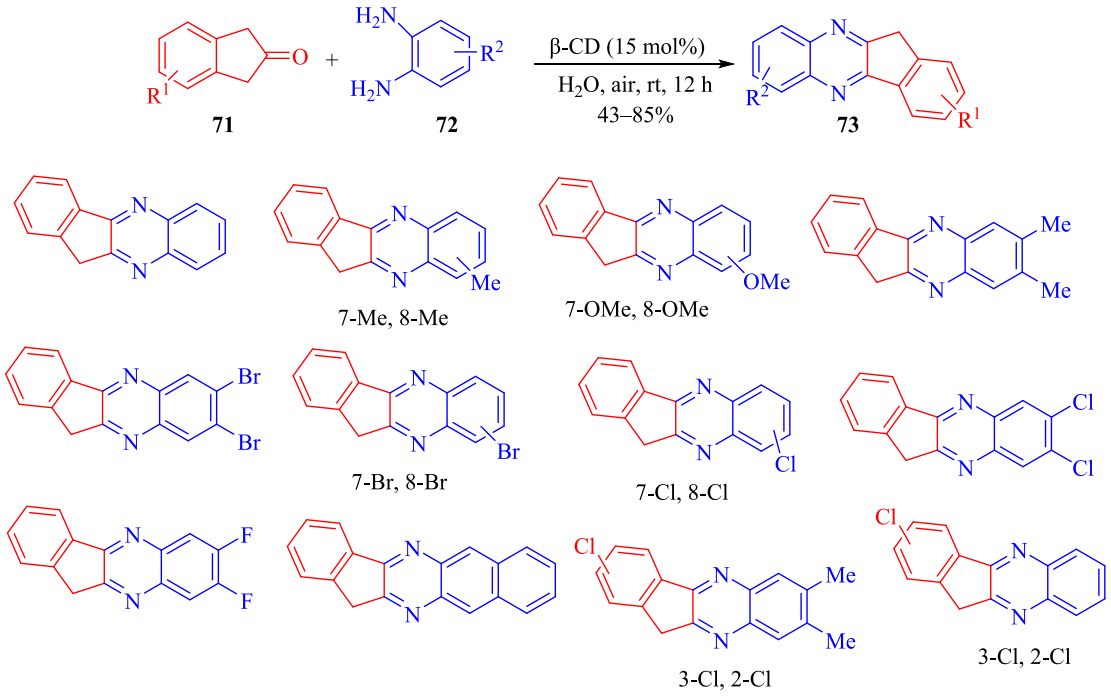
In 2023, this researcher illustrated the preparation of xanthene derivatives **80** from β-naphthol (**79**), aryl aldehyde **1**, and dimedone (**20**) by β-CD as reusable in the water media (Mohamadpour, 2023). In general, the reaction was performed by β-CD (15 mol%) in H_2O solvent at 80 °C, and xanthene products were given in good yield (91–94 %)



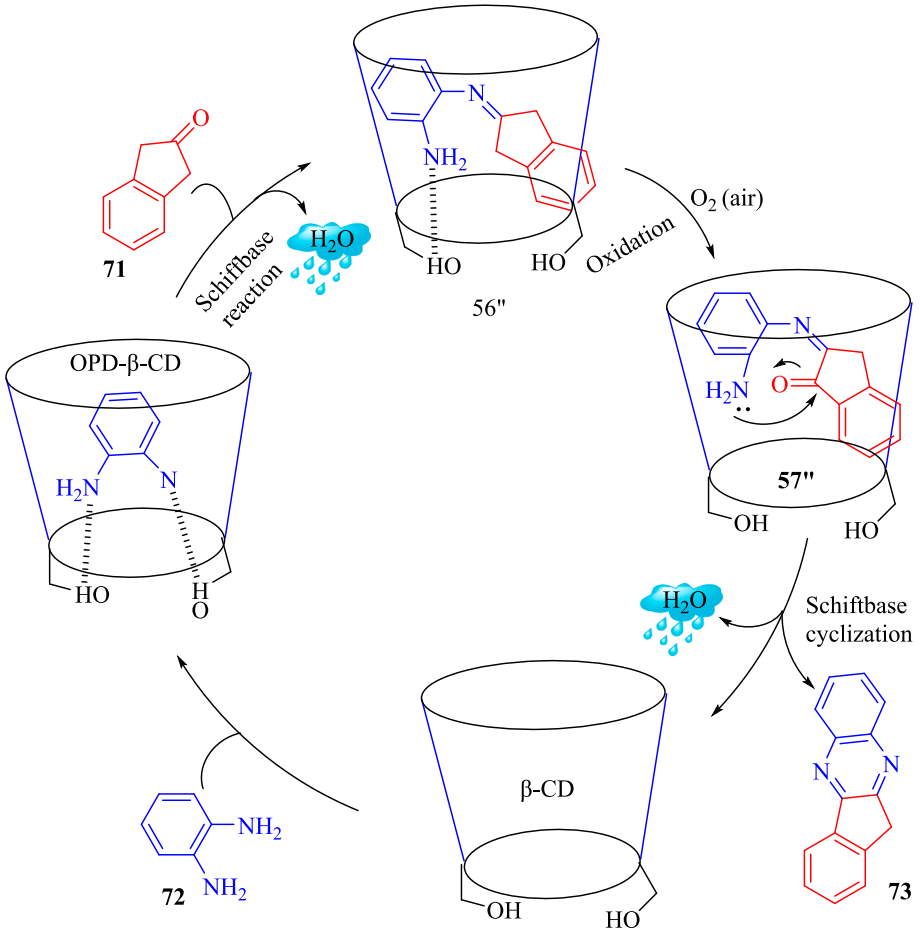
Scheme 60. The synthesis of pyranoquinolines by β-CD catalyst.



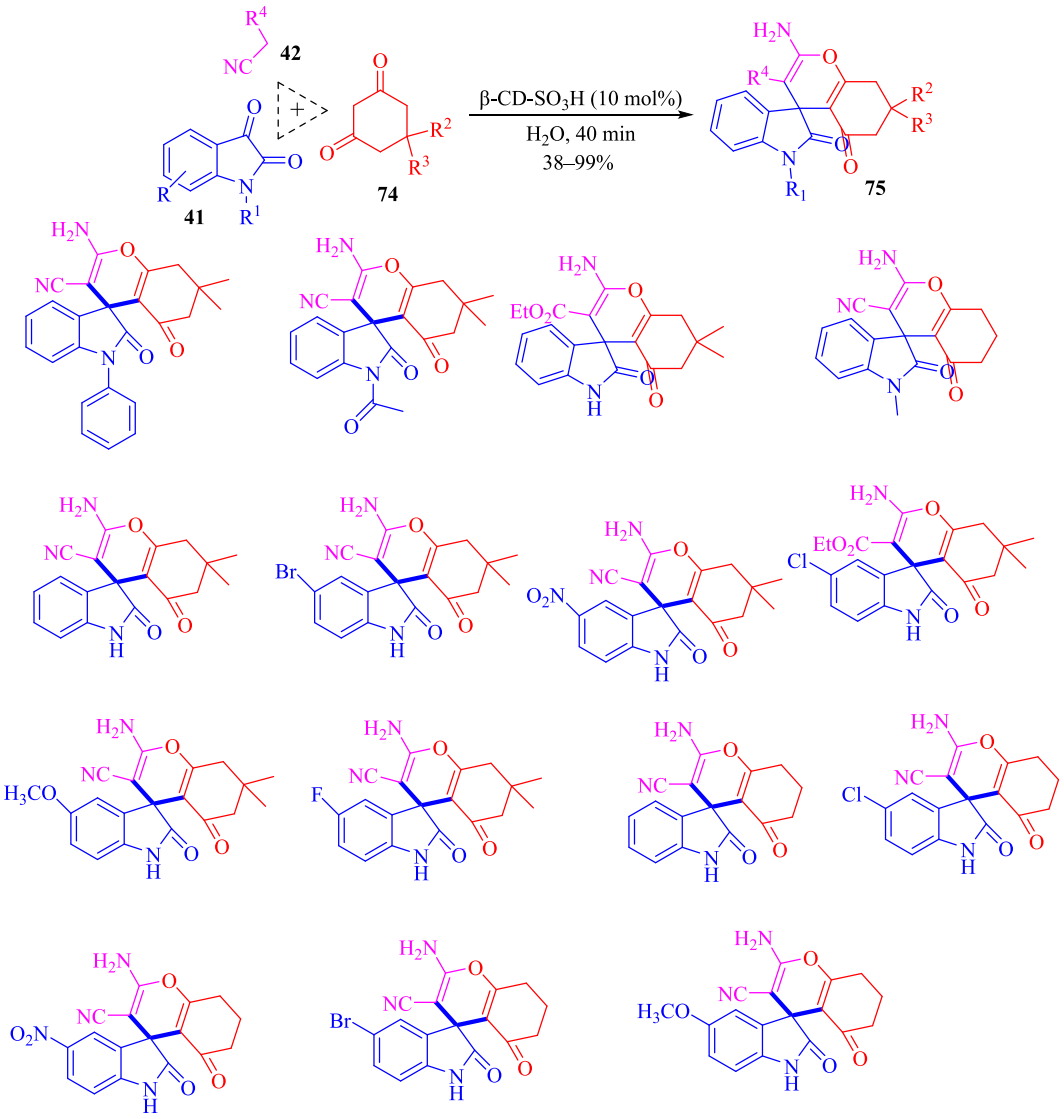
Scheme 61. The suitable mechanism for the reaction.



Scheme 62. Synthesis of indeno[1,2-*b*]quinoxalines by β-CD catalyst.



Scheme 63. A possible mechanism for the reaction.



Scheme 64. Synthesis of drivers spiro indole derivatives by β-CD-SO_3H catalyst.

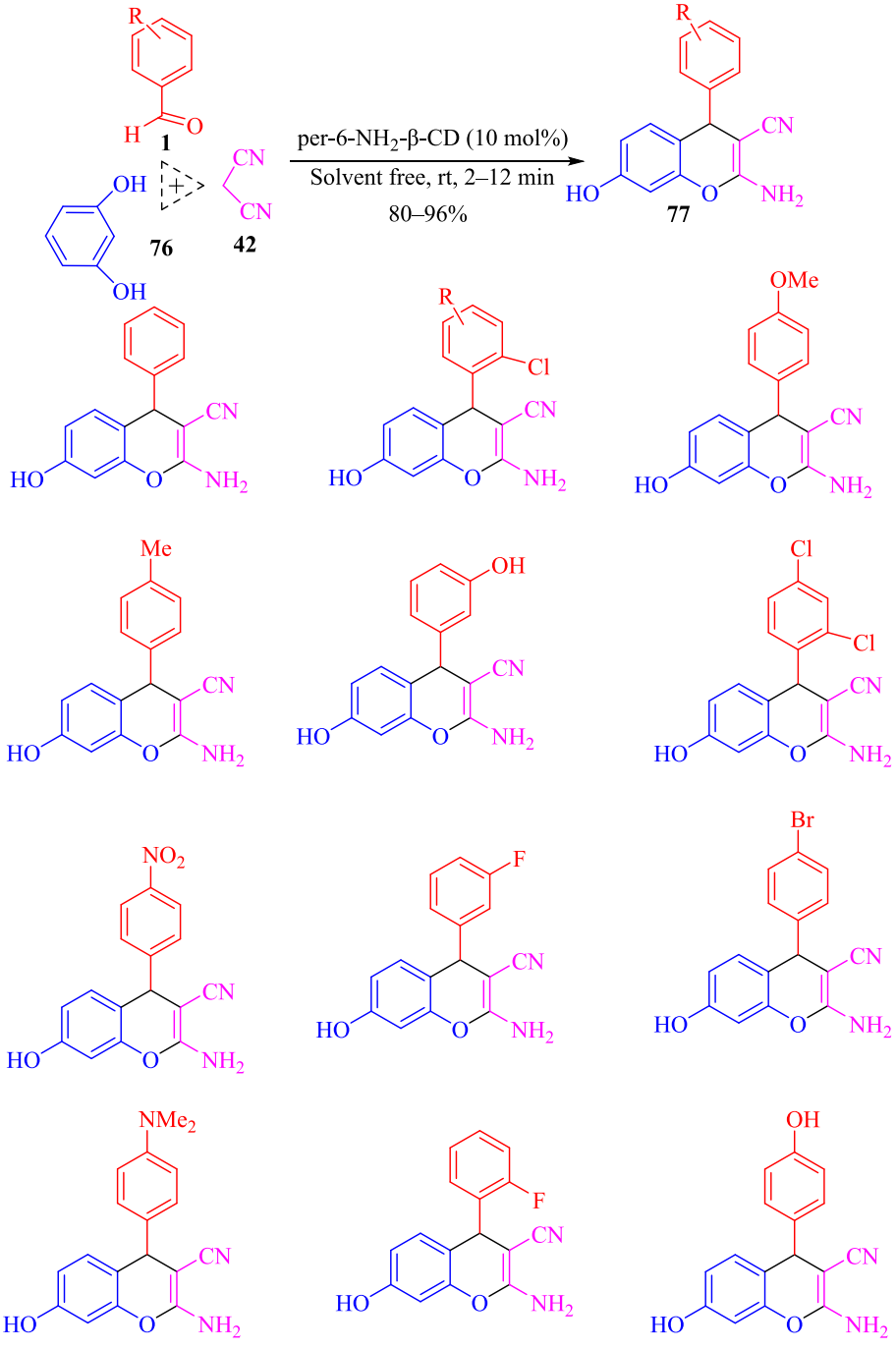
Table 9
A comparison of catalytic performance β-CD–SO_3H with the various catalysts for the synthesis of spiroindoline **75**.

Entry	Catalyst	Temperature (℃)	Time (min)	Yield (%)	Reference
1	Hexamethylenetetramine	60	20–150	60–93	Wang et al., 2013
2	Nanocrystalline MgO	80	90–180	80–95	Karmakar et al., 2012
3	Silica sulfuric acid magnetic NPs	60	80–120	90–95	Karimi et al., 2015
4	β-CD–SO_3H	50	40	38–99	Current discussed report

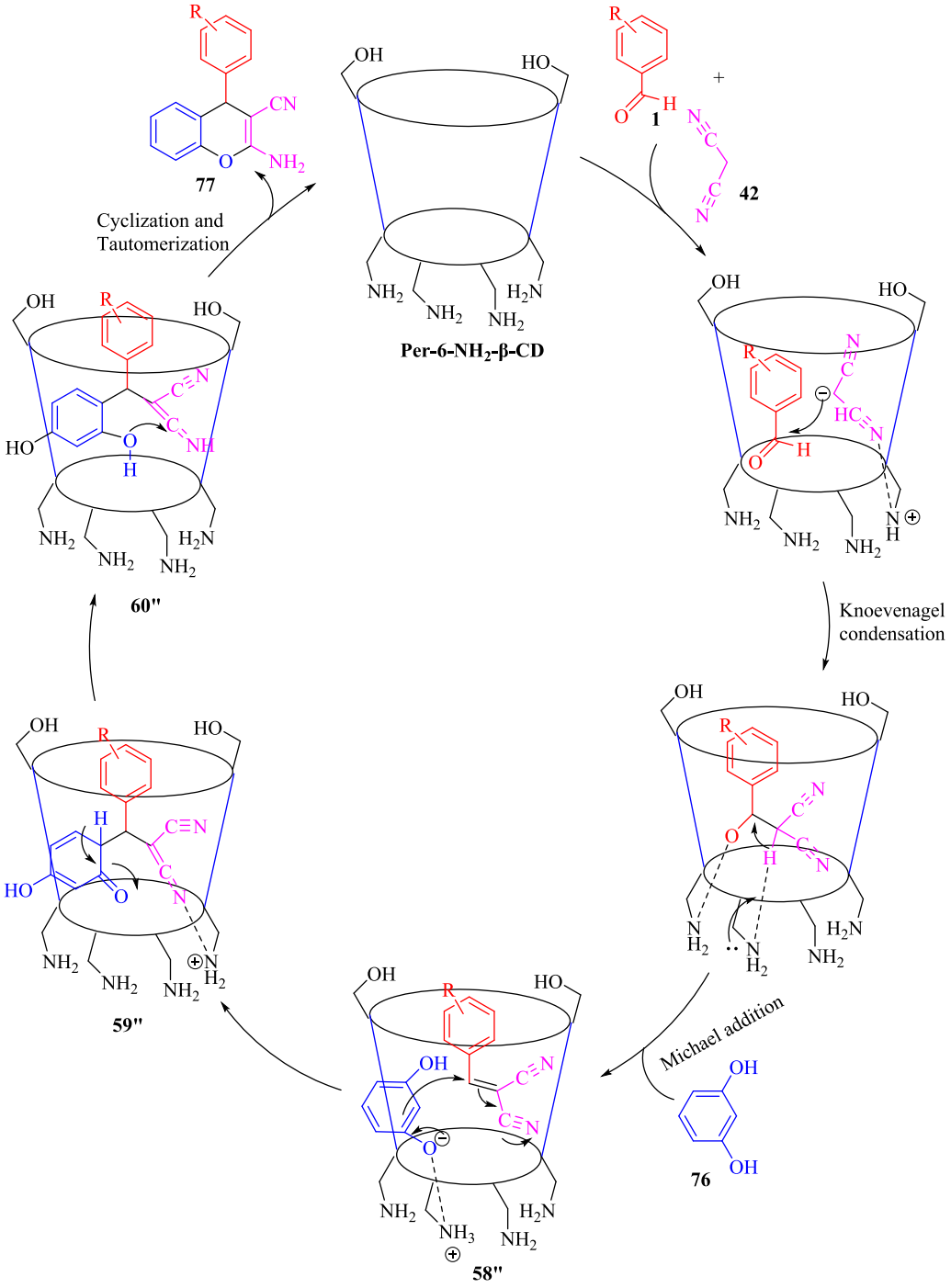
(Scheme 69). β-CD could be recovered four times without a crucial reduction in catalytic performance. This approach had excellent yields with short-period reactions. The plausible mechanism is shown in Scheme 70. The enol tautomer of β-naphthol **79** reacted with the activated aldehydes **65″**. After creating intermediates (**66″**and **67″**), it generated the products **80**.

2.3. Synthesis of seven-membered heterocycles

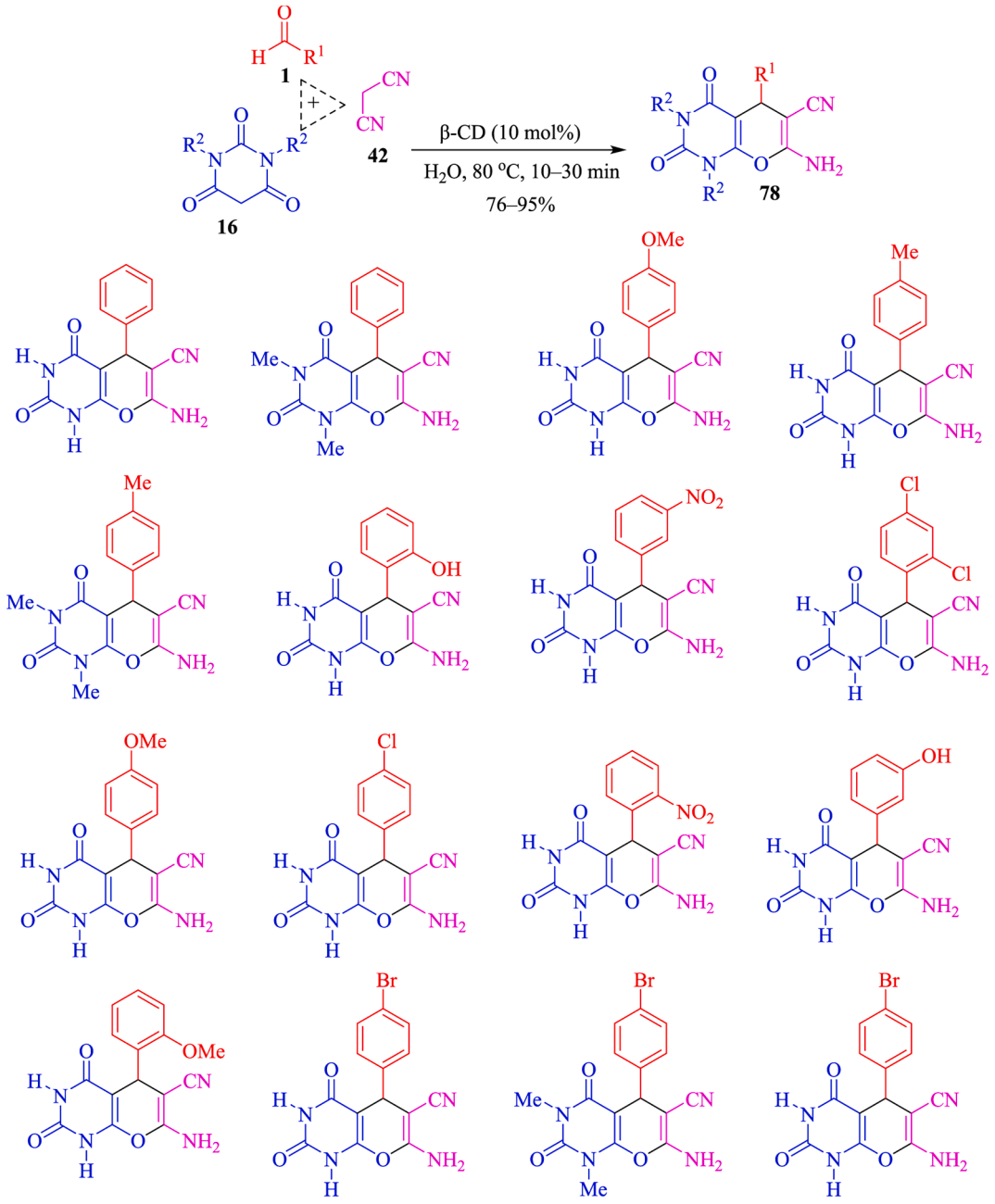
Masram *et al.* used a green protocol to synthesize four 1,5-benzodiazepines **81** under mild status involving β-CD as a recyclable catalyst in H_2O at reflux conditions (Scheme 71) (Masram et al., 2022). The TCR of OPD (**71**), dimedone (**20**), and aryl aldehydes **1** is developed to provide



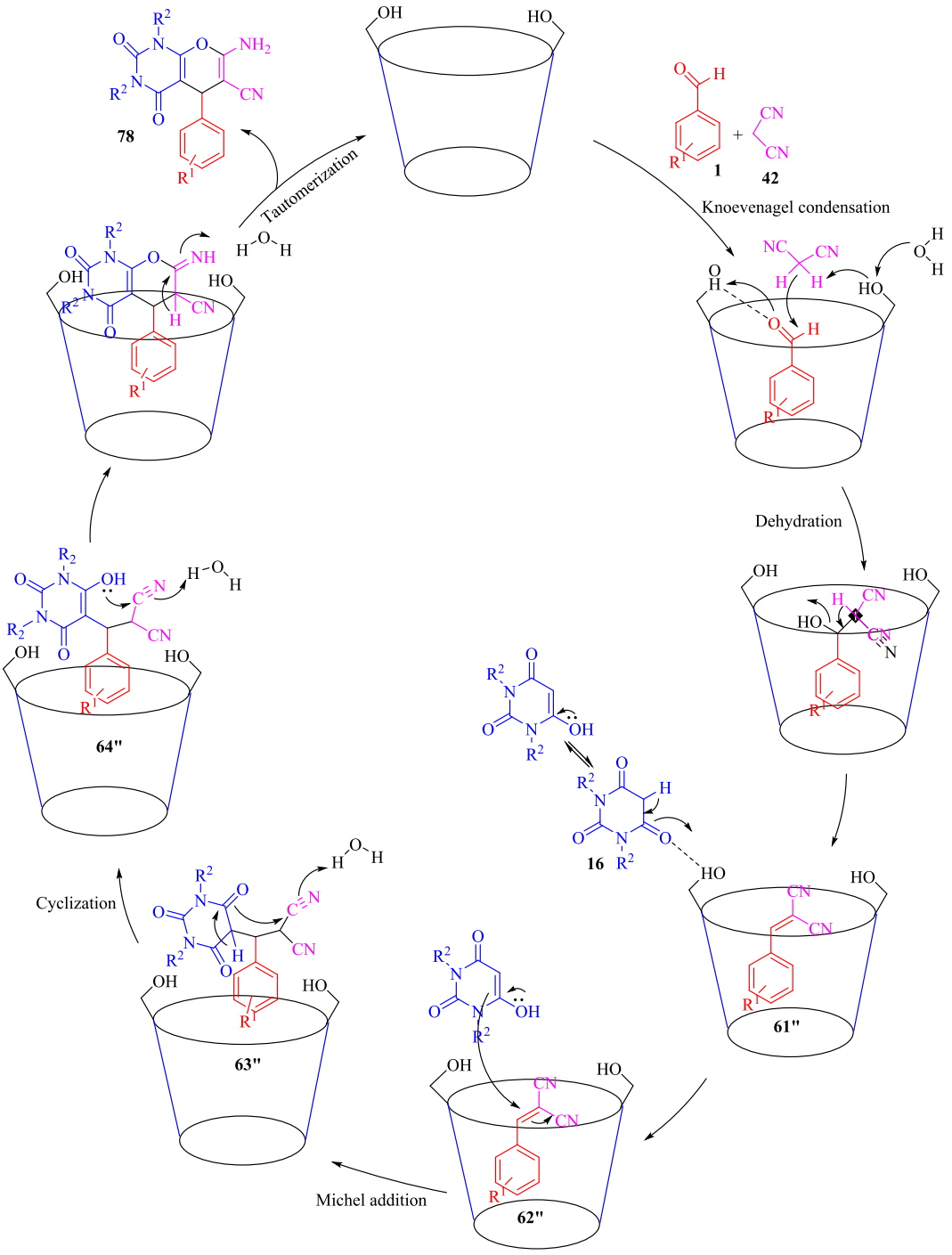
Scheme 65. Preparation of 2-amino-4*H*-chromenes scaffolding by per-6-NH_2-β-CD.



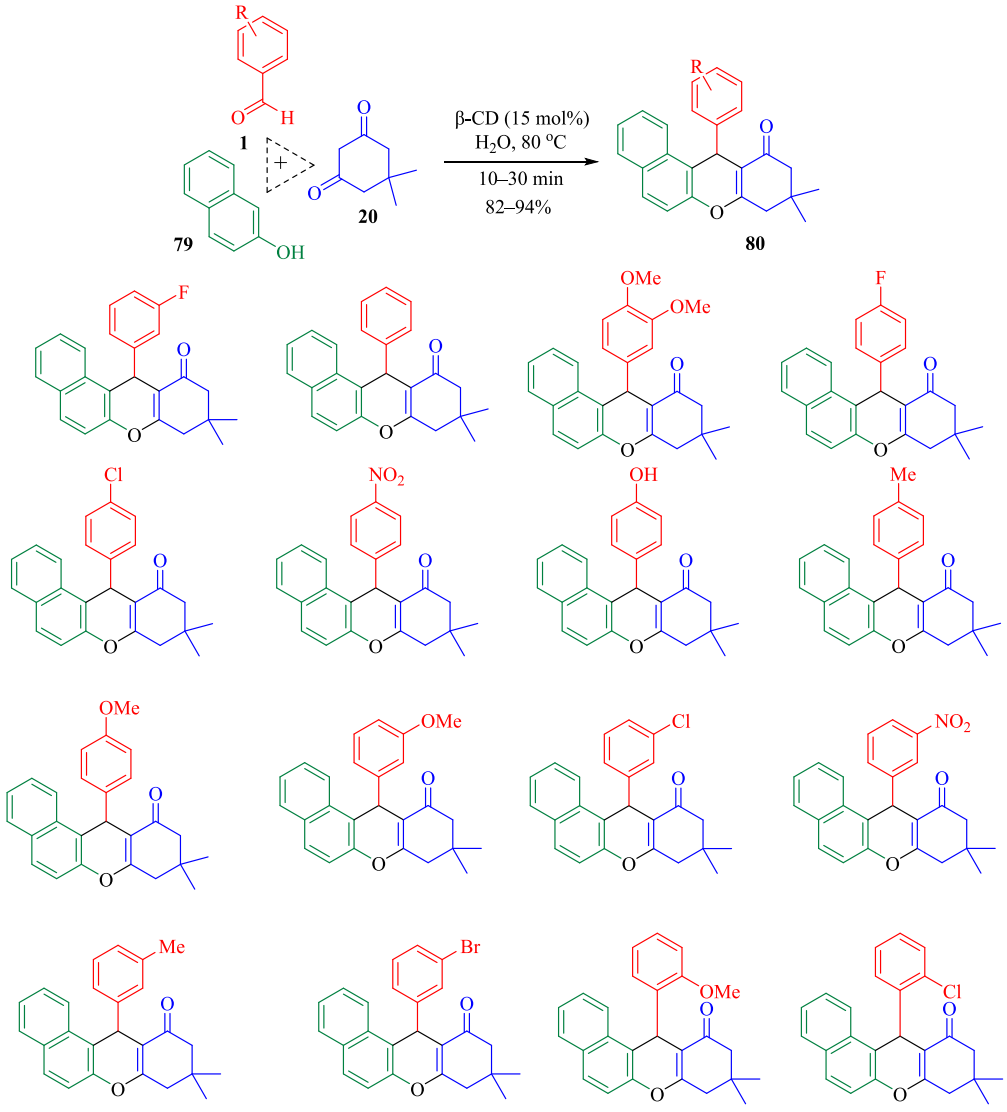
Scheme 66. Suggested path for the reaction.



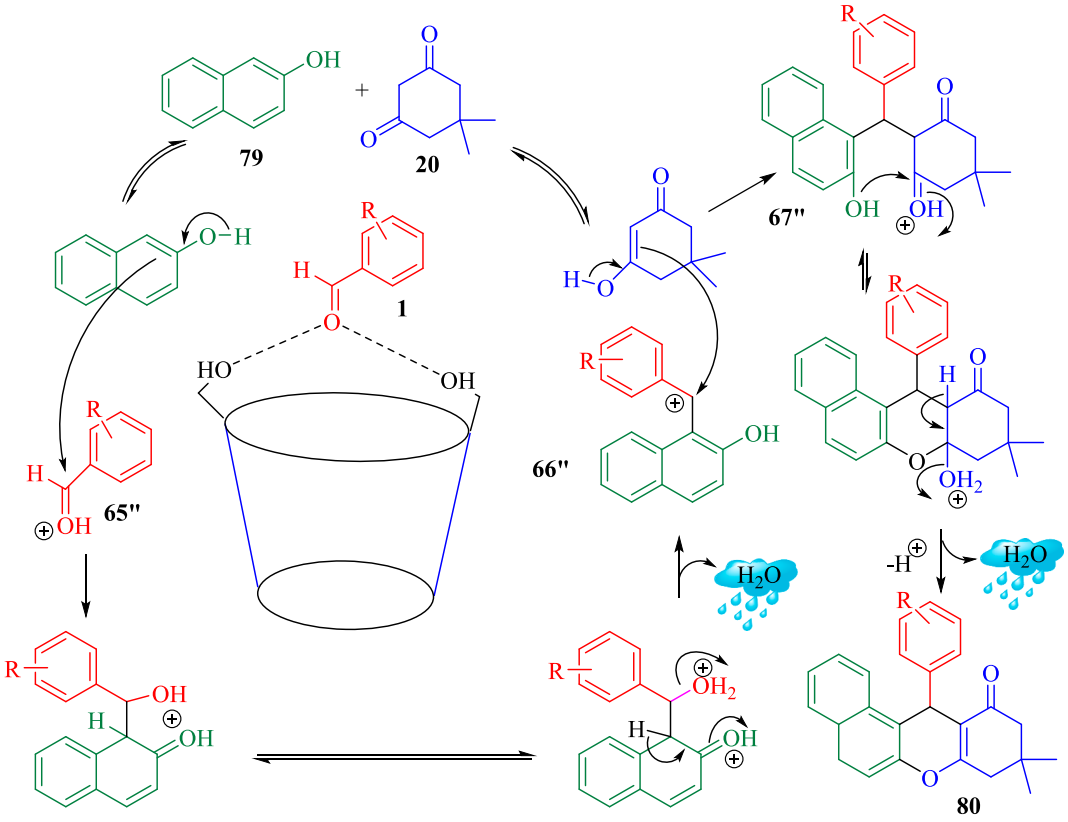
Scheme 67. The preparation of pyrano[2,3-*d*]pyrimidines by β-CD catalyst.



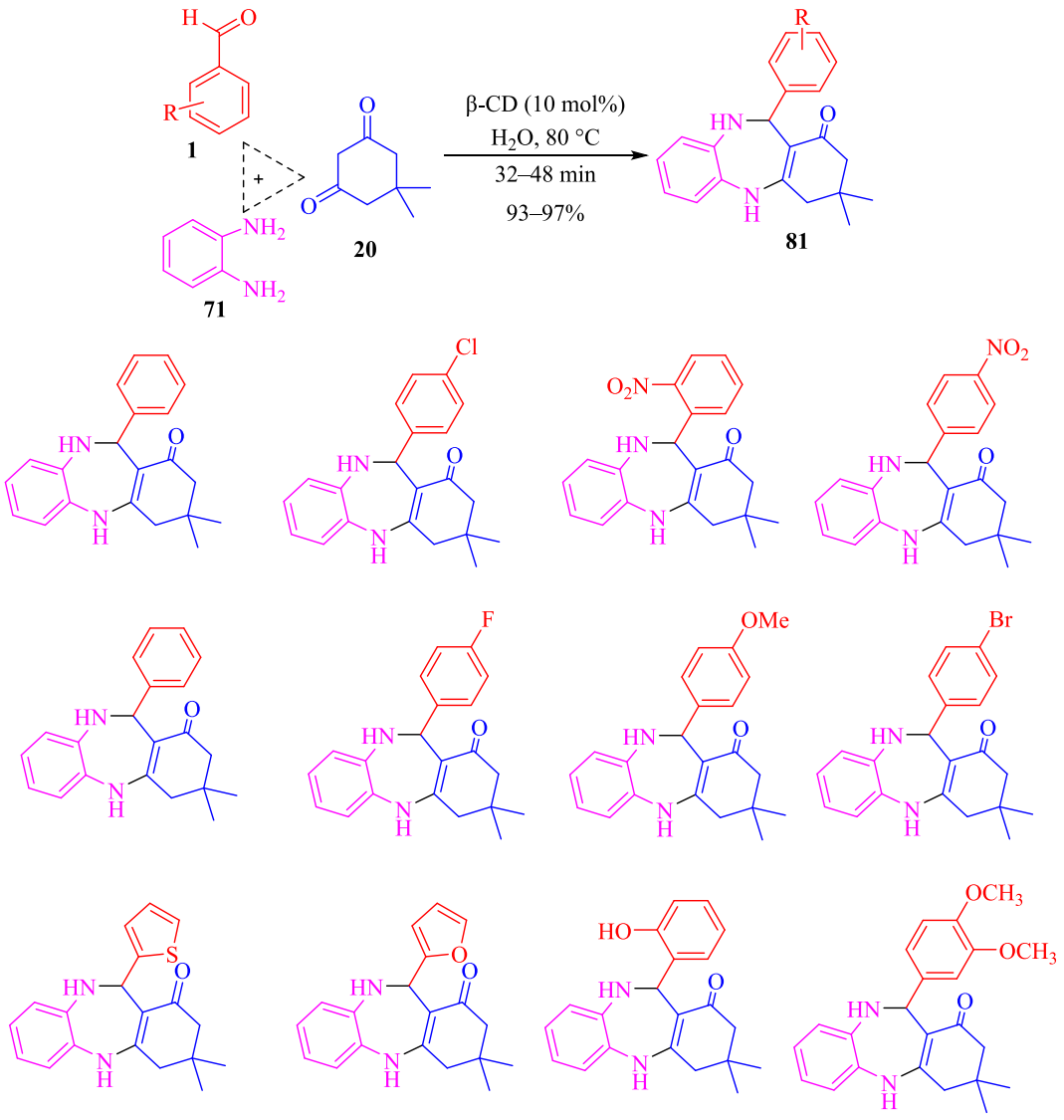
Scheme 68. A suggested mechanistic path for the reaction.



Scheme 69. Synthesis of xanthene products using β-CD catalyst.



Scheme 70. The proposed way for the preparation of xanthenes by β-CD.

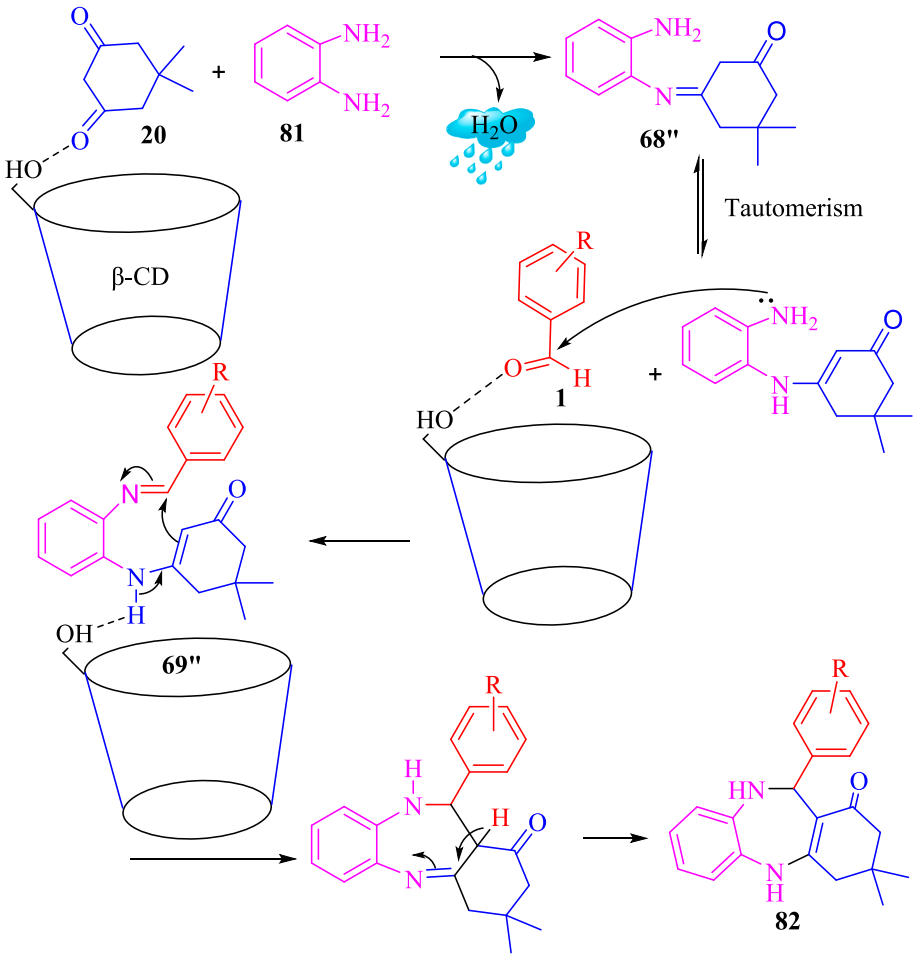


Scheme 71. Preparation of 3,4-dihydropyrimidine-2(1*H*)-ones catalyzed by β-CD.

good to excellent yields (93–98 %). Other CDs were also tested as catalysts. α- and γ- CD showed a lower result of the product than the β-CD. The catalyst was recovered in four runs without lowering the yield of the product. The excellent yields, easy workup, shorter reaction time, green catalyst, and lack of use of dangerous material are some advantages of this work. The plausible mechanism is displayed in Scheme 72. At first, dimedone **20** attaches to the OH group of β-CD, increasing the electrophilicity of the C═O group reaction. Then it reacts with **81,** which subsequently provides an intermediate of imine **68″**. In the next step, aldehyde carbonyl is attacked by the o-phenyl diamine amine group, which further undergoes tautomerism to create the intermediate **69″**. After further cyclization, the final product **82** is generated.

3. Conclusions

In summary, we have reported an overview of the usage of β-CD as a helpful catalyst in synthesizing diverse heterocyclic structures and printed in the last five years. It is significant to mention that a broad spectrum of the reactions catalyzed by β-CD can be performed in the H_2O media, making those methods green and eco-friendly. One of the most significant advantages of β-CD catalyst is that it can be recycled and reused many times with no drop in efficiency. This kind of organic reaction has recently found more and more applications in synthesizing pharmacological and natural compounds. There is a rising attraction to using β-CD catalysts, as they play a significant role in preparing medical and organic heterocyclic material. MCR has received much interest in preparing various biological compounds for developing new drugs. Heterocyclic compounds have been a fundamental structure in medical chemistry for many years. They indicate various biological activities comprising anti-bacterial, anti-cancer activity, anti-diabetic, and anti-inflammatory. They are plentiful in many biomolecules, such as natural products, enzymes, and vitamins. Heterocyclic compound chemistry has become a critical field because of the connection between their chemistry and medical issues. This review will investigate the recent



Scheme 72. The putative mechanism for the preparation of compounds **82**.

developments, catalysts based on β-CD, and their applications in heterocyclic structures. This review will benefit and be invaluable for pharmaceutical chemistry and drug design development researchers. One of the most notable attainments in this study has been helping scientists develop green and low-price catalysts based on green chemistry aims in organic reactions.

CRediT authorship contribution statement

Sara Payamifar: Writing – original draft, Investigation. **Majid Abdouss:** Supervision. **Ahmad Poursattar Marjani:** Writing – review & editing, Supervision, Investigation.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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