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Nickel complexes as efficient catalysts in multicomponent synthesis of tetrahydropyridine derivatives

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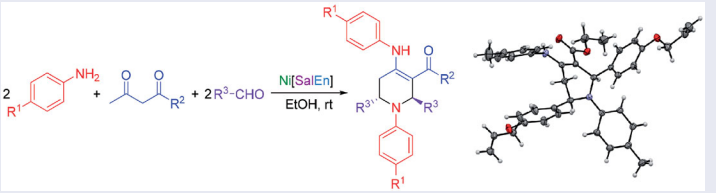
ABSTRACT

Ni(Salen) complexes as an efficient, homogeneous catalysts revealed a catalytic activity toward the one-pot synthesis of tetrahydropyridine derivatives through the pseudo five-component reactions of aromatic aldehydes, aromatic amines, and β-ketoesters in ethanol at room temperature. Mild reaction conditions, good yields, high diastereoseletivity, operational simplicity, and the absence of tedious separation procedures, clean reaction profiles, high atom economy, inexpensive starting materials, and environmentally benign catalyst are the key advantages of the present MCRs protocol.

GRAPHICAL ABSTRACT



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Heterocycles; Lewis acids; nickel complexes; one-pot synthesis; tetrahydropyridines

Introduction

The development of new metal catalysts has been of wide interest in synthetic methodology.[1] Schiff bases are widely used as ligands in coordination chemistry and catalysis,[2] and their complexes can be accessed by a variety of methodologies. As representative in the class of Schiff complexes, salen metal complexes have been explored in many applications, from material science and catalysis to coordination chemistry.[3] The easy to prepare salen metal complexes have been used in the development of catalysts for numerous reactions, which are widely used for the synthesis of practically significant compounds.[4–9] Salen derivatives have demonstrated their huge application as ligands for catalysis, since, associated to a large variety of metallic salts, they were able to promote formation of various C–C, C–heteroatom, or heteroatom–heteroatom bonds.[10]

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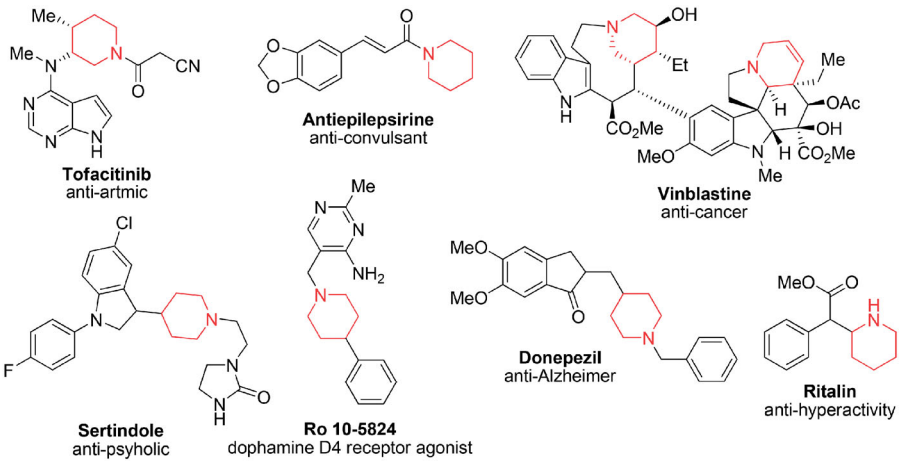


Figure 1. Representative compounds containing the tetrahydropyridine moiety.

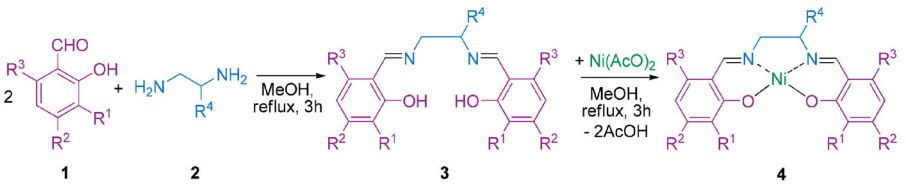
The synthesis of tetrahydropyridine derivatives is an important area of research, since these scaffolds are core motifs in numerous natural products[11,12] and find applications as dophamine D4-receptor agonist[13] and also exhibit antibacterial,[14,15] anti-inflammatory,[16] anticancer,[17] anticonvulsant,[16] antitubercular,[18] antiartmic,[19,20] antipsyholic,[21] etc. activities. Moreover, tetrahydropyridine derivatives possess enzyme inhibitory activity against acetylcholinesterase (AChE) which is believed to play an important role in the development of Alzheimer's disease.[22] Examples of well-known biologically active compounds containing a tetrahydropyridine moiety are shown in Figure 1. Hence, the synthesis of highly functionalized piperidines has evoked much attention as a result of which a number of methodologies have been developed involving a variety of cyclization techniques such as one-pot pseudo five-component reactions of aromatic aldehydes, anilines, and β-ketoesters under various reaction conditions.[15,23–25].

Herein, we described the synthesis of tetrahydropyridine derivatives, where we considered various Ni(Salen) complexes as catalysts, showing their effectiveness in comparison with Lewis acids.

Result and discussion

Initially, we obtained a series of nickel salen complexes through two stage synthesis (Scheme 1). At the first stage, ligand **3** was obtained via reaction salicylic aldehyde derivatives **1** with ethylene diamine **2**. Further, compound **3** was metalized via nickel(II) acetate ($Ni(AcO)_2$) with the formation of complexes **4**.[26–29]

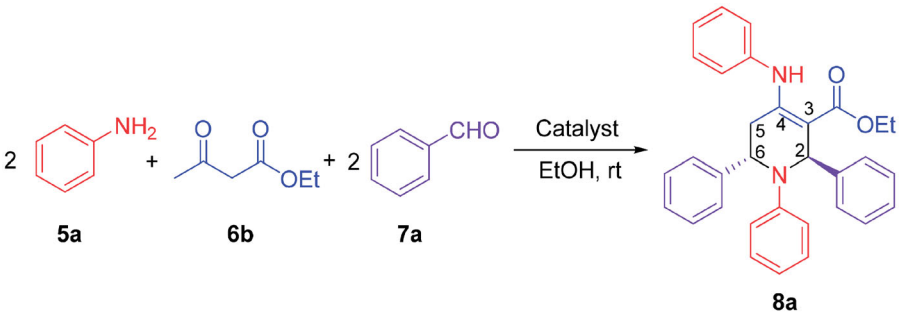
Standard reaction conditions were developed using the model reaction of ethylacetoacetate **6b** (10 mmol), aniline **5a** (20 mmol), and benzaldehyde **7a** (20 mmol) in ethanol (10 mL) at room temperature in the presence of various Ni(SalEn) complexes **4a–g** and Lewis acids such as iron(III) chloride ($FeCl_3$), bismuth(III) nitrate ($Bi(NO_3)_3$), and iodine (I_2), which were previously used as catalysts in the synthesizes various heterocyclic compounds,[30–34] including tetrahydropyridines[15] (Table 1). The best results were obtained in the presence of Ni(SalEn) **4a** (10 mol%) which gave tetrahydropyridine **8a** (Table 1, entry 5) in 79% yield. Ni(Salen) complexes **4b–g** with different substitutes in



Ni[SalEn] **4a**: R^1=H, R^2=H, R^3=H, R^4=H;
Ni[3-MeSalEn] **4b**: R^1=Me, R^2=H, R^3=H, R^4=H;
Ni[4-MeSalEn] **4c**: R^1=H, R^2=Me, R^3=H, R^4=H;
Ni[3-MeOSalEn] **4d**: R^1=MeO, R^2=H, R^3=H, R^4=H;
Ni[6-MeOSalEn] **4e**: R^1=H, R^2=H, R^3=MeO, R^4=H;
Ni[4-Et_2NSalEn] **4f**: R^1=H, R^2=Et_2N, R^3=H, R^4=H;
Ni[SalPn] **4g**: R^1=H, R^2=H, R^3=H, R^4=Me.

Scheme 1. Synthesis Ni(Salen) complexes.

Table 1. Catalyst screening for the synthesis of tetrahydropyridine **8a**[a].



Entry	Catalyst		Time	Yield (%)[b]
1	None		192 h	35
2	$Bi(NO_3)_3{\cdot}5H_2O$ (10 mol%)		12 h	76
3	$FeCl_3{\cdot}6H_2O$ (10 mol%)		12 h	64
4	I_2 (10 mol%)		14 h	51
5	Ni(SalEn) (10 mol%)	**4a**	15 min	79
6	Ni(SalEn) (20 mol%)	**4a**	15 min	77
7	Ni(3-MeSalEn) (10 mol%)	**4b**	40 min	69
8	Ni(4-MeSalEn)	**4c**	6 h	74
9	Ni(3-MeOSalEn)	**4d**	2 h	71
10	Ni(6-MeOSalEn)	**4e**	3 h	67
11	Ni(4-Et_2NSalEn)	**4f**	7 h	56
12	Ni(SalPn)	**4g**	2 h	65

[a]Reagents and conditions: benzaldehyde (20 mmol), ethylacetoacetate (10 mmol), aniline (20 mmol), catalyst, ethanol (10 mL), room temperature.
[b]Isolated yield.

aromatic ring and diamine bridge showed a lower catalytic activity in comparison **4a**, apparently, probably due to the steric effect. Various solvents were tested for the synthesis of **8a** (Table 2) which revealed ethanol to be the best choice. The traces were obtained when dimethyl sulfoxide (DMSO) and water were employed as solvents (Table 2, entries 11 and 12).

To explore this reaction under the optimized conditions, a variety of aromatic aldehydes and amines containing electron donating or electron withdrawing substituents on the aromatic ring (Me, OMe, *tert*-Bu, allyloxy, F, and thiophenyl) were reacted with *β*-ketoesters (Table 3). A relationship between the nature of the substituents in the

Table 2. Solvent screening for the synthesis of tetrahydropyridine **8a**[a].

Entry	Solvent	Yield (%)
1	MeOH	75
2	EtOH	79
3	*i*-PrOH	70
4	THF	53
5	CH_2Cl_2	15
6	$CHCl_3$	12
7	MeCN	56
8	Me_2CO	50
9	1,4-Dioxane	44
10	DMF	5
11	DMSO	Traces
12	Water	Traces

[a]Reagents and conditions: benzaldehyde (20 mmol), ethylacetoacetate (10 mmol), aniline (20 mmol), Ni(SalEn) (10 mol%), solvent (10 mL), room temperature.
[b]Isolated yield.

Table 3. Synthesis of tetrahydropyridine **8a–h**[a].

Entry	R^3	R^4	R^5	Product	Yield (%)[b]
1	H	OEt	Ph	**8a**	79
2	H	OEt	4-*tert*-Bu(C_6H_4)	**8b**	82
3	H	OEt	Thiophen-2-yl	**8c**	75
4	OMe	OEt	Thiophen-2-yl	**8d**	81
5	H	O-*tert*-Bu	Thiophen-2-yl	**8e**	71
6	H	O-*i*-Pr	Thiophen-2-yl	**8f**	69
7	Me	OEt	4-$OCH_2CHCH_2(C_6H_4)$	**8g**	89
8	H	OEt	4-F(C_6H_4)	**8h**	67

[a]Reagents and conditions: aromatic aldehyde (20 mmol), *β*-ketoester (10 mmol), aromatic amine (20 mmol), Ni(SalEn) (10 mol%), ethanol (10 mL), room temperature.
[b]Isolated yield.

aromatic ring and such as -F (Table 3, entry 8). the product yield was not observed. However, the smallest product yield (67%) was obtained by electron-withdrawing substituents.

All products were characterized by IR, ^{1}H NMR, ^{13}C NMR, elemental analysis, and mass spectroscopic studies (see ESI). The relative anti-configuration of the product was confirmed by comparing the physical and spectroscopic data of the known compounds with those in the literature,[24] and also by single crystal X-ray analysis of the model compound **8g** (Fig. 2, S27).[35] This is also important to mention here that the presence of the corresponding syn-diastereomer in the products could not be identified from the

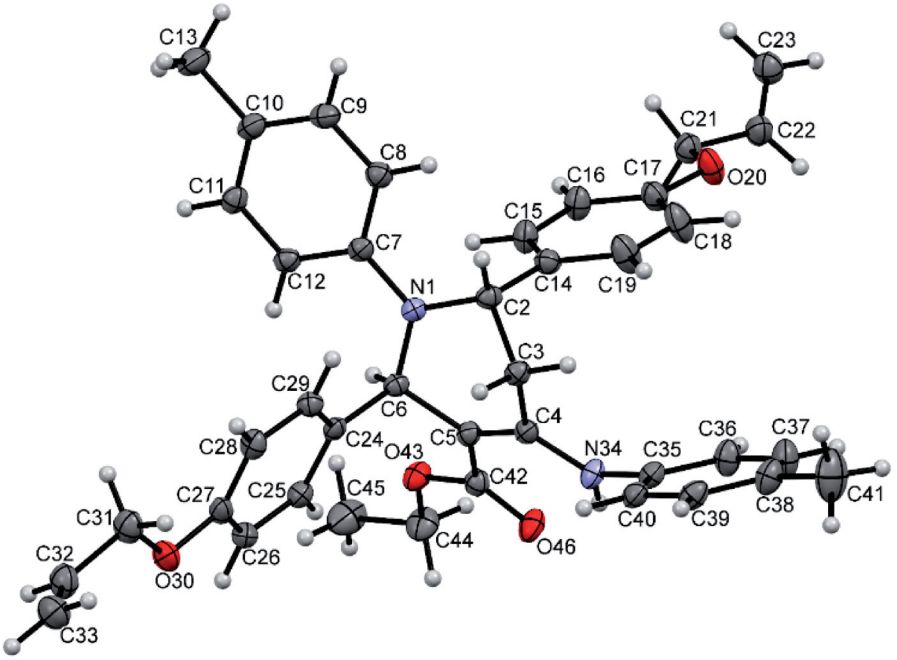


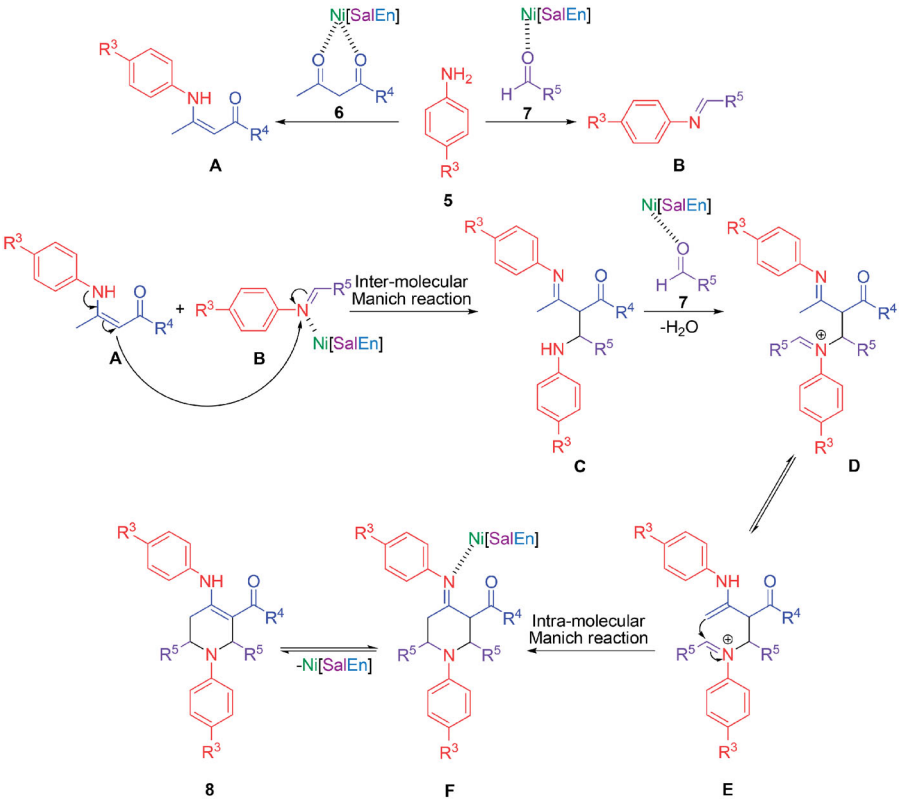
Figure 2. Molecular structure of compound **8g** showing 50% probability displacement ellipsoids (CCDC 1919473).

^{1}H NMR of the product(s), indicating the reactions to be exclusively anti-diastereoselective.

A probable mechanistic pathway for this five-component reaction is outlined in Scheme 2, which is in analogy to the established mechanism as reported in the literature.[15,23–25] Based on the presented mechanism, initially, the carbonyl groups of *β*-ketoester **6** and aromatic aldehyde **7** were activated by Ni(Salen) as catalyst, which made them susceptible to the nucleophilic attack of aromatic amine **5**. Thereafter, the activated **6** and **7** were separately condensed with **5** to give *β*-enaminone **A** and imine **B**, respectively. Subsequently, an intermolecular Mannich reaction occurred between **A** and **B** to generate intermediate **C**. Intermediate **C** was reacted with another molecule of aldehyde **7** to give intermediate **D** through the elimination of one H_2O molecule. Finally, tautomerization of **D** led to the formation of intermediate **E**, which instantaneously underwent an intramolecular Mannich reaction to offer the product **F**. Next, upon tautomerization, **F** was converted to the more stable form of the desired tetrahydropyridine **8**.

Conclusion

In conclusion, a straightforward and efficient protocol for one-pot synthesis of functionalized piperidine scaffolds has been developed in the presence of Ni(Salen) complexes as catalyst via five-component tandem reaction of aromatic aldehydes, *β*-ketoesters, and amines at room temperature. Mild reaction conditions, good yields, high diastereoselectivity, operational simplicity and the absence of tedious separation procedures, clean



Scheme 2. Plausible mechanism for the formation of tetrahydropyridine derivatives **8**.

reaction profiles, high atomeconomy, inexpensive starting materials, and environmentally benign catalyst are the key advantages of the present MCRs protocol.

Experimental

General

IR spectra were recorded on FTIR-spectrometer «IRAffinity-1» (Shimadzu, Kyoto, Japan). NMR spectra were recorded on Bruker AVANCE 400SX using DMSO-d_6 and $CDCl_3$ as solvents, TMS as internal standard substance, with proton and carbon resonances at 400 and 100 MHz, respectively. HR-ESI-MS spectra were recorded on «MaXis» mass spectrometer with accessories, Bruker Daltonik GmbH.

X-ray crystallographic analysis

The unit cell parameters and the X-ray diffraction intensities were measured on a SuperNova diffractometer. The empirical absorption correction was introduced by multi-scan method using SCALE3 ABSPACK algorithm.[36] Using the Olex2,[37] the structure was solved with the SHELXT program[38] and refined by the full-matrix least-squares method in the anisotropic approximation for all non-hydrogen atoms with the

SHELXL program.[39] Hydrogen atoms were located from the Fourier synthesis of the electron density and refined using a riding model.

General procedure for the preparation of complexes 4a–g

Corresponding salicylic aldehyde **1** (10 mmol) and 1,2-diaminoethane **2** (5 mmol) were mixed in MeOH (10 mL). Reaction mixture was refluxed for 3 h, cooled down and filtered. The ligand **3** obtained was redissolved in MeOH, and $Ni(OAc)_2{\cdot}4H_2O$ (0.88 g, 5 mmol) was added to the solution. The reaction mixture was stirring and refluxed for 3 h, cooled down, filtered off, recrystallized from MeCN:EtOH (1:1) and dried *in vacuo* to obtain the desired complex **4**.

General procedure for the preparation of tetrahydropiridines 8a–h

A mixture of corresponding aromatic amine **5** (20 mmol), β-ketoester **2** (10 mmol) and Ni(SalEn) **4a** (10 mol%) in EtOH (10 mL) were stirred for 30 min at room temperature. Then aromatic aldehyde **7** (20 mmol) was added to the solution. The reaction mixture was stirring for 15 min, filtered off, recrystallized from EtOH:AcOEt (2:1) and dried *in vacuo* to obtain the desired tetrahydropiridine **8**.

Ethyl 2,6-bis(4-(allyloxy)phenyl)-1-p-tolyl-4-(p-tolylamino)-1,2,5,6-tetrahydropyridine-3-carboxylate 8g

White crystalline solid; 89%. FTIR (KBr) ν, cm^{-1}: 3236 (NH), 1651 (C = O). ^1H NMR (400 MHz, $CDCl_3$, 25 °C): δ_H = 1.47 (t, J(H,H) = 7.1 Hz, 3H; $OCH_2\underline{CH_3}$), 2.19 (s, 3H; Me), 2.30 (s, 3H; Me), 2.70–2.78 (m, 1H; $C^5\underline{H}_A H_B$), 2.83 (dd, J(H,H) = 15.0, 5.5 Hz, 1H; $C^5H_A\underline{H}_B$), 4.34 (dq, J(H,H) = 10.9, 7.1 Hz, 1H; $OC\underline{H}_A H_B CH_3$), 4.45 (ddd, J(H,H) = 14.3, 9.0, 5.4 Hz, 1H; $OCH_A\underline{H}_B CH_3$), 4.55 (dd, J(H,H) = 8.9, 3.4 Hz, 4H; OCH_2), 5.07 (d, J(H,H) = 2.7 Hz, 1H; $C^6\underline{H}$), 5.30 (dd, J(H,H) = 5.3, 1.3 Hz, 1H; $=C\underline{H}_A H_B$), 5.32 (dd, J(H,H) = 5.3, 1.3 Hz, 1H; $=C\underline{H}_A H_B$), 5.40–5.43 (m, 1H; $=CH_A\underline{H}_B$), 5.45–5.48 (m, 1H; $=CH_A\underline{H}_B$), 6.03–6.15 (m, 2H; =CH), 6.27 (d, J(H,H) = 8.1 Hz, 2H; Ar-2C$\underline{H}$), 6.35 (s, 1H; $C^2\underline{H}$), 6.47 (d, J(H,H) = 8.6 Hz, 2H; Ar-2C$\underline{H}$), 6.85 (dd, J(H,H) = 8.6, 1.9 Hz, 4H; Ar-4C$\underline{H}$), 6.92 (dd, J(H,H) = 14.9, 8.2 Hz, 4H; Ar-4C$\underline{H}$), 7.09 (d, J(H,H) = 8.6 Hz, 2H; Ar-2C$\underline{H}$), 7.25 (d, J(H,H) = 8.6 Hz, 2H; Ar-2C$\underline{H}$), 10.25 (s, 1H; NH). ^{13}C NMR (100 MHz, $CDCl_3$, 25 °C): δ_H = 14.8 ($OCH_2\underline{C}H_3$), 20.1 (Me), 20.9 (Me), 33.7 (C^5), 54.7 (C^6), 57.5 (C^2), 59.5 ($O\underline{C}H_2CH_3$), 68.9 ($O\underline{C}H_2$), 68.9 ($O\underline{C}H_2$), 97.9 (C^3), 113.0, 114.3, 114.8, 117.6, 117.6, 125.0, 125.9, 127.5, 127.7, 129.4, 129.4, 133.4, 133.5, 135.2, 135.4, 135.5, 136.5, 145.0, 156.5, 157.0, 157.7 (C^4, –$\underline{C}H$=$\underline{C}H_2$, C-Ar), 168.3 (CO). HRMS (ESI): calcd. for $C_{40}H_{43}N_2O_4Na$ $[M+H]^+$ 615.3217, found 615.3192; calcd. for $C_{40}H_{42}N_2O_4Na$ $[M+Na]^+$ 637.3037, found 637.3012.

Full experimental details, elemental analysis, IR, HR-ESI-MS, ^1H, and ^{13}C NMR spectra for all new compounds, crystal data and structure refinement for **8g**. This material can be found through the "Supplementary Content" section of this article's webpage.

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[35] CCDC 1919473 (for **8g**) contain the supplementary crystallographic data for this paper. The data can be obtained free of charge from The Cambridge Crystallographic Data Centre via https://www.ccdc.cam.ac.uk/
[36] CrysAlisPro, Version 1.171.40.50a, Rigaku Oxford Diffraction.
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