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Zirconocene dichloride catalyzed multi-component synthesis of 1-amidoalkyl-2-naphthols at ambient temperature

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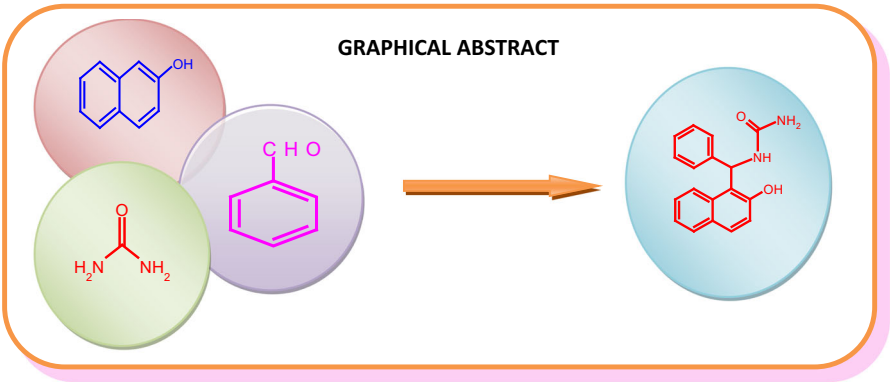
Abstract Zirconocene dichloride (Cp_2ZrCl_2) was found to be highly efficient catalyst for the synthesis of structurally diverse 1-amidoalkyl-2-naphthols by one-pot multi-component reaction (MCR) of 2-naphthol with a variety of aryl aldehydes and amides under ambient temperature. The activation of both carbonyl groups of aryl aldehydes and the *o*-quinine methide intermediate accounts for the high reactivity of Cp_2ZrCl_2.

Graphical Abstract



Keywords Multi-component reaction · Zirconocene dichloride · 1-Amidoalkyl-2-naphthols · *o*-Quinine methide

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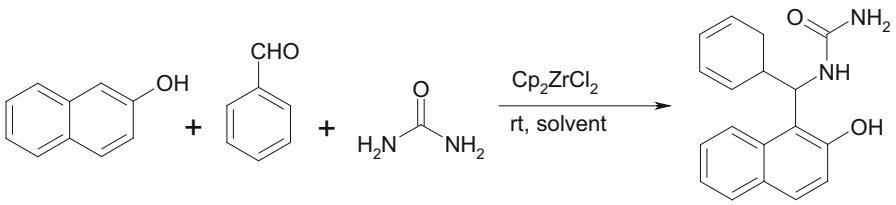
Introduction

Organozirconocenes represent a very powerful answer to the quest for new-generation, metallocene-based catalysts in organic synthesis. The synthetic applications of organozirconocenes have received a decisive impetus since the pioneering work of Schwartz [1–5] and Negeshi [6]. Among them, cationic zirconocene has been advanced in recent years as a versatile catalyst for mediating synthetically important transformations [7–9]. Zirconocene dichloride (Cp_2ZrCl_2) and its derivatives constitute an important class of cationic zirconocene complexes that are particularly promising for synthetic applications due to their stability, commercial availability and non-hazardous nature [10]. Cp_2ZrCl_2 has been extensively used as a catalyst in polymerization reactions [11–13]. The catalytic activity of Cp_2ZrCl_2 is attributed to its weak acidity. However, despite the impressive catalytic potential of Cp_2ZrCl_2, its utility in organic synthesis is only sporadically demonstrated. The catalytic processes initiated by Cp_2ZrCl_2 include synthesis of multi-substituted vinyl silanes [14], bis(indolyl) methanes [15], cyclobutenylphosphaonates [16] and quinozolin-4(3*H*)-ones [17]. Cp_2ZrCl_2 has also been used as a catalyst in acetylation of phenols/alcohols/amines [18], coupling of terminal alkynes as well as intramolecular coupling of amines [19, 20], and in the Refortmastky and Barbier reaction [21]. To tap the barely exploited potential of Cp_2ZrCl_2 in organic synthesis, we sought to explore its catalytic activity in synthesis of scaffolds with a high therapeutic value.

Multi-component reactions (MCRs) are arguably one of the most stimulating, dynamic and synthetically powerful areas in contemporary organic synthesis for generating high levels of molecular diversity from three or more reactants with a minimal number of operations [22–24]. The importance of molecular diversity has been clearly recognized to identify the lead compounds in drug discovery and development programs. Indeed, MCRs are intrinsically connected to sustainable chemistry as they comply with the principles of green chemistry in terms of economy of steps as well as many of the stringent criteria of an ideal organic synthesis. The MCRs are of particularly great utility when they lead to the formation of privileged medicinal scaffolds. 1-Amidoalkyl-2-naphthol scaffolds are of significant medicinal relevance since they constitute an important class of intermediates that can be converted into hypertensive and bradycardiac-active 1-aminoalkyl-2-naphthols by amine hydrolysis reactions [25]. Recently, Abou-Elmagd et al. [26] reported promising cytotoxicity and antiviral activity of 1-amidoalkyl-2-naphthols. The intriguing structural features and promising biological activities of 1-amidoalkyl-2-naphthols have rendered them the status of a novel pharmacophore in the context of drug design. Consequently, much effort has been made towards the development of efficient methods for their synthesis. 1-Amidoalkyl-2-naphthols are prepared by one-pot MCR of 2-naphthol with aryl aldehydes and amides. A large number catalysts such as piper-betle-shaped nano-S [27], $SiCl_4 + ZnCl_2$ [28], graphite-supported perchloric acid [29], $RuCl_2(PPh_3)_3$ [30], $[Cu(CH_3CN)_4PF_6]$ [31], silica gel-supported SO_3H [32], zinc benzene sulphonate [33], $[NMP]HSO_4$ [34], $[MeC(OH)_2]ClO_4$ [35], sulphonic acid-



Scheme 1 Cp_2ZrCl_2 catalyzed multi-component synthesis of 1-amidoalkyl-2-naphthols

functionalized imidazolium salts [36], silica gel-supported dual acidic ionic liquid [37], trityl chloride [38], $H_3Mo_{12}O_{40}P$ [39], montmorillonite K10 [40], SSA [41], P-TSA [42] etc. have been reported for the synthesis of 1-amidoalkyl-2-naphthols. However, despite significant progress, there is still room for improvement, especially towards developing a facile protocol working at ambient temperature.

In our continued interest in the development of highly expedient methodologies for the synthesis of bioactive scaffolds [43, 44], we report herein the Cp_2ZrCl_2-catalyzed synthesis of 1-amidoalkyl-2-naphthols by MCR of 2-naphthol with a variety of aryl aldehydes and amides at ambient temperature (Scheme 1).

Experimental

Melting points were determined in an open capillary and are uncorrected. All reactions were carried out under air atmosphere in dried glassware. Infrared spectra were measured with a Perkin-Elmer Spectrum One FTIR spectrometer. The samples were examined as KBr discs ~5 % w/w. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Brucker AC (300 MHz for ^{1}H NMR and 75 MHz for ^{13}C NMR) spectrometer using deuterated dimethylsulfoxide (DMSO-d_6) as the solvent and tetramethylsilane (TMS) as an internal standard. Chemical shifts (δ) are expressed in parts per million (ppm) values with TMS as the internal reference, and coupling constants are expressed in hertz (Hz). Mass spectra were recorded on a Shimadzu QP2010 gas chromatograph mass spectrometer. All the chemicals were obtained from local suppliers and used as received.

General procedure for the synthesis of 1-amidoalkyl-2-naphthols

A mixture of 2-naphthol (1 mmol), aldehyde (1 mmol), acetamide or benzamide or urea (1.2 mmol) and zirconocene dichloride (20 mol%) in ethylene dichloride (EDC; 5 mL) was stirred at room temperature for a specified time. After completion of the reaction, as indicated by thin layer chromatography (TLC), the reaction mixture was quenched in cold water. The obtained crude solid was filtered and purified by column chromatography on silica gel (Merck, 60–120 mesh, ethyl acetate: hexane) to afford the pure product which was then characterized by

spectroscopic methods such as infrared (IR), ^{1}H NMR, ^{13}C NMR and mass spectroscopy.

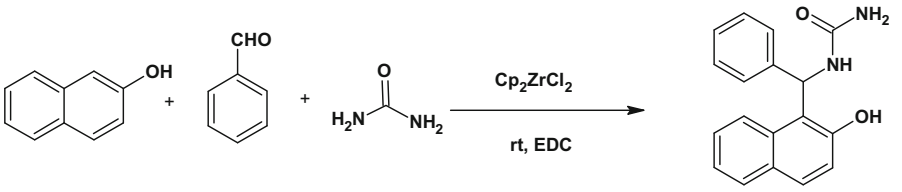
Spectral data of representative compound

N-[(4-Methylphenyl)-(2-hydroxynapthalen-1-yl)-methyl]acetamide (Table 2, entry 4p) Mp: 233 °C, ^{1}H NMR (300 MHz, DMSO-d_6): δ 1.92 (s, 3H), 2.23(s, 3H), 7.04–7.01 (m, 5H), 7.21 (d, J = 8.8 Hz, 1H), 7.20 (t, J = 7.1 Hz, 1H), 7.31 (m, 1H), 7.71 (d, J = 8.8 Hz, 1H), 7.75 (d, J = 7.9 Hz, 1H) 7.81(brd, 1H), 8.31 (d, J = 8.1 Hz, 1H), 9.89 (s, 1H) ppm; ^{13}C NMR (75 MHz, DMSO-d_6): 20.1, 22.4, 47.3, 118.1, 118.7, 121.9, 122.4, 125.1, 126.4, 128.8, 128.1, 128.6, 132.5, 134.4, 139.2, 143.1, 152.5, 169.2 ppm; IR (KBr): υ = 3417, 3317, 3074, 1625, 1599, 1567, 1517, 1464, 1395, 12,838, 1209, 1148, 1059, 941, 889, 788, 741, 718 cm^{-1}; MS (EI): *m*/*z* 305 (M^{+}).

Results and discussion

We initiated our studies by examining the effectiveness of different solvents on the model reaction of 2-naphthol, benzaldehyde and urea using a catalytic amount of the Cp_2ZrCl_2 (20 mol%). The polar protic solvents, such as methanol and ethanol, gave a poor yield of corresponding product (Table 1, entry 2 and 3). No improvement was detected when the same reaction was carried out using polar aprotic solvents such as DMF and acetonitrile (Table 1, entries 1, 6). To our delight, the desired

Table 1 Optimization of the reaction conditions using different solvents

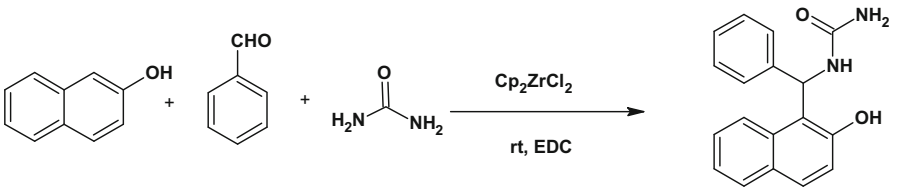


Sr. no.	Solvent	Reaction time (h)	Yield (%)[a]
1.	DMF	12	65
2.	Methanol	15	40
3.	Ethanol	18	45
4.	**Ethylene dichloride**	**6**	**91**
5.	Dichloromethane	24	40
6.	Acetonitrile	24	40

Reaction conditions: 2-Naphthol (1 mmol), benzaldehyde (1 mmol), urea (1.2 mmol) and Cp_2ZrCl_2(20 mol %) in solvent (5 mL) were stirred at room temperature

[a] Isolated yields after chromatography

Table 2 Optimization study for the amount of Cp_2ZrCl_2



Sr. no.	Amount of catalyst (mol%)	Reaction time (h)	Yield (%)[a]
1.	00	24	No reaction
2.	05	24	45
3.	10	19	54
4.	15	14	58
5.	**20**	**6**	**91**
6.	25	8	91
7.	30	8	92

Reaction conditions: 2-Naphthol (1 mmol), benzaldehyde (1 mmol), urea (1.2 mmol) and Cp_2ZrCl_2 in EDC (5 mL) were stirred at room temperature

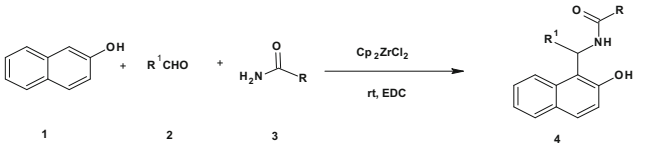
[a] Isolated yields of purified products

product was obtained in good to excellent yield in a polar, aprotic solvent such as ethylene dichloride (Table 1, entry 4).

The optimum quantity of catalyst was assessed by performing the model reaction in the presence of various amounts of Cp_2ZrCl_2 (Table 2). The best results were obtained when 20 mol% of catalyst was used (Table 2, entry 5). The use of higher catalyst loading (>20 mol%) did not improve the yield significantly (Table 2, entry 6 and 7). On the other hand, for loading of catalyst below 20 mol %, Cp_2ZrCl_2 did not promote the reaction to a synthetically useful degree (Table 2, entries 2–4). It is worthy of note that when the model reaction was carried out in the absence of Cp_2ZrCl_2, no product was formed either at ambient temperature or under reflux conditions for 24 h (Table 2, entry 1).

With optimized conditions in hand, we evaluated the scope of the protocol by reacting 2-naphthol and different amides such as urea/acetamide/benzamide with electronically and structurally diverse aryl aldehydes. The results are summarized in Table 3. The nature of substituents on the aromatic ring had a profound effect on the yield of the product. In all cases, the corresponding 1-amidoalkyl-2-naphthols were the sole products and no anomalies were observed. It was observed that substituents in the aromatic ring of aldehydes have a delicate effect on the reaction process. Aromatic aldehydes with electron-withdrawing groups reacted faster than those with electron-donating groups. To further expand the scope of the present method, the replacement of aryl aldehydes with heterocyclic and aliphatic aldehydes was examined. To our delight, furfuraldehyde, thiophene-2-aldehyde (Table 3, entries f and g) as well as formaldehyde (Table 3, entry h) reacted, giving satisfactory

Table 3 Cp_2ZrCl_2 -catalyzed reaction of 2-naphthol, aldehydes, and urea or acetamide or benzamide



1 2 3 4

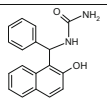
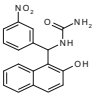
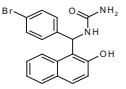
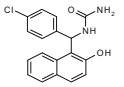
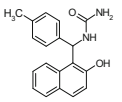
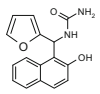
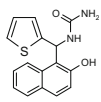
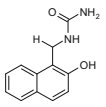
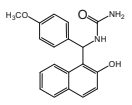
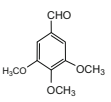
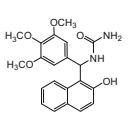
Entry	Aryl Aldehyde (2)	Amide (3)	Product (4)	Time (h)	Yield[b] (%)	Physical Const.[c] (°C)
a				7	91	170 (168-170)[55]
b				5	94	179 (179-180)[56]
c				7	89	171 (170-172)[56]
d				5	93	167 (168)[32]
e				8	62	117 (118-120)[53]
f				7	45	163 (162.3-163.7)[27]
g				7	42	159 (159-161)[27]
h				5	51	175 (174)[32]
i				9	63	186 (182-184)[27]
J				10	Trace	--------

Table 3 continued

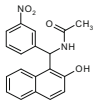
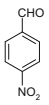
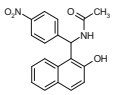
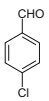
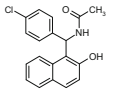
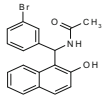
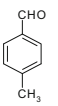
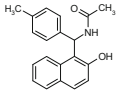
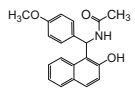
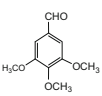
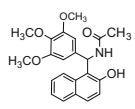
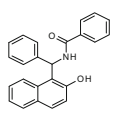
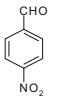
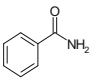
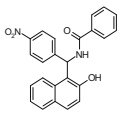
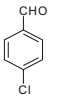
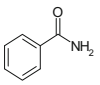
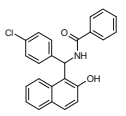
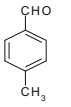
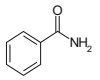
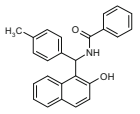
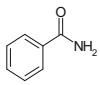
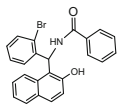
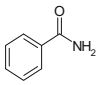
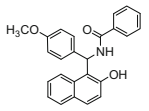
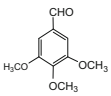
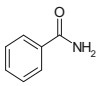
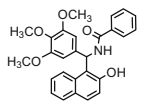
Entry	Aryl Aldehyde (2)	Amide (3)	Product (4)	Time (h)	Yield[b] (%)	Physical Const.[c] (°C)
k				8	89	245 (245-246)[55]
l				8	90	213 (212-215)[40]
m				7	91	247 (249)[32]
n				7	87	225 (226)[32]
o				8	85	251 (250-252)[56]
p				9	66	223 (222-223)[55]
q	H—CHO			7	52	244 (242)[32]
r				8	79	187 (182-183) [48]
s				9	70	191 (190-193) [53]
t				9	86	235 (234-236)[51]

Table 3 continued

Entry	Aryl Aldehyde (2)	Amide (3)	Product (4)	Time (h)	Yield[b] (%)	Physical Const.[c] (°C)
u				6	90	227 (228)[32]
v				7	88	179 (177-178)[51]
w				10	68	175 (175-177)[53]
x				7	86	228 (228-230)[51]
y				8	76	196 (197-199)[48]
z				9	67	233 (234-236)[53]

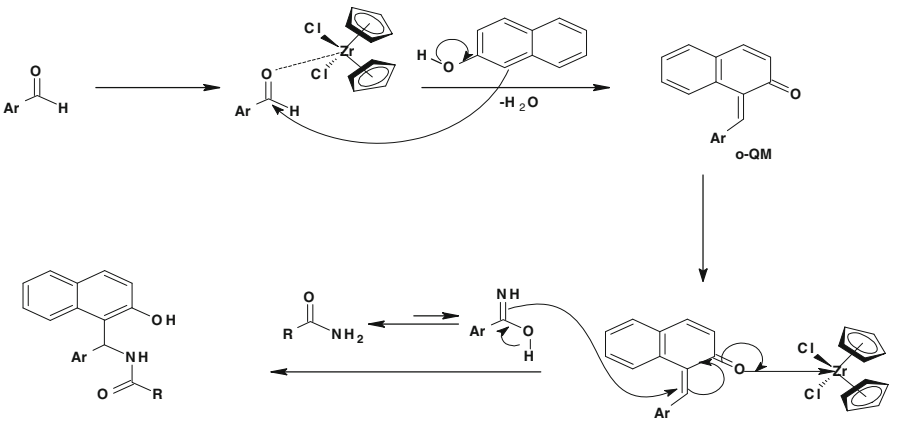
Reaction conditions: 2-Naphthol (1 mmol), aldehyde (1 mmol), Urea/benzamide/acetamide (1.2 mmol) and Cp_2ZrCl_2 (20 mol%,) in EDC (5 mL) were stirred at rt

[a] Isolated yields of purified products

product yields. The molecular structure of products **4a−z** were elucidated from their IR and ^{1}H and ^{13}C NMR spectra, as described for **4p**. The mass spectrum of **4p** displayed the molecular ion peak at $m/z = 305$ (M^+), which is in agreement with the proposed structure. The IR spectrum of **4p** shows broad absorption bands at 3417 for O–H stretching and a sharp band at 3317 for N–H stretching of amide. Also, three absorption bands at 1625, 1599 and 1567 cm^{-1}, which are related to C=O, C=N and C=C stretching, respectively, clearly indicated the most significant functional groups of the product. The ^{1}H NMR spectra of **4p** exhibited two sharp singlet signals at δ 1.92 and 2.23 ppm, readily recognized as a methyl group, as well as broad singlets at 8.31 and 9.89 ppm, indicating N–H and O–H, respectively. Eleven signals in the aromatic region gave rise to characteristic peaks for an

aromatic, and one signal for active methylene proton. Observation of 18 distinct signals in the ^{1}H-decoupled ^{13}C NMR spectrum of **4p** is in agreement with the proposed structure. In all other cases, spectral and physical data are in harmony with the proposed structures and are in good agreement with the reported values [55].

A mechanistic account simulating the probable sequence of events in the Cp_2ZrCl_2 -catalyzed synthesis of 1-amidoalkyl-2-naphthols is given in Scheme 2. Formation of amidoalkylnaphthols in the reaction of 2-naphthol, aldehyde, and amides has been reported, possibly through *ortho*-quinonemethides (*o*-QMs) [56]. Initially, Cp_2ZrCl_2 coordinates with aryl aldehyde causing electrophilic activation of a carbonyl moiety which triggered a nucleophilic attack upon 2-naphthol to



Scheme 2 Proposed mechanism for the synthesis of 1-amidoalkyl-2-naphthols using Cp_2ZrCl_2

Table 4 Comparison of different catalysts for the reaction of benzaldehyde, acetamide and 2-naphthol

Sr. no.	Catalyst used	Amount of catalyst	Temp (°C)	Time (h)	Yield (%)	References
1.	$Fe(HSO_4)_3$/solvent-free	5 mol%	85	1.08	83	[45]
2.	Montmorillonite K10	0.1 g/mol	125	1.5	89	[40]
3.	$K_5CoW_{12}O_40_3H_2O$/solvent-free	1 mol%	125	2	90	[46]
4.	$HClO_4$_SiO_2/solvent-free	100 mg	125	6.50	82	[47]
5.	p-TSA/solvent-free	10 mol%	125	6	89	[42]
6.	Polymer-supported sulphonic acid	170 mg	65	5	96	[57]
7.	Iodine	5 mol%	Room temp.	10	85	[49]
8.	$Sr(OTf)_2/CHCl_3$	10 mol%	65	10	90	[50]
9.	Indion	0.25 g/mol	110	10	94	[51]
10.	Sulphamic acid	10 mol%	Room temp.	12	81	[52]
11.	$Al(H_2PO_4)$	0.75 g/mol	125	21	93	[53]
12.	$Ce(SO_4)_2$	–	Under reflux	36	72	[54]
13.	Cp_2ZrCl_2	20 mol%	Room temp.	8	89	This work

generate *ortho*-quinonemethide (*o*-QM). Further, Cp_2ZrCl_2 facilitates 1,4-nucleophilic addition of amides on *o*-QMs, affording the desired 1-amidoalkyl-2-naphthol.

In order to show the merit of Cp_2ZrCl_2 in comparison with the other catalysts used for the similar reaction, we have summarized several results for the preparation of *N*-[(phenyl)-(2-hydroxy-naphthalen-1-yl)-methyl] acetamide from benzaldehyde, 2-naphthol and acetamide in Table 4. It is evident from these results that $Cp_2ZrCl_2$is an highly effective catalyst for the synthesis of 1-amidoalkyl-2-naphthols at ambient temperature.

Conclusion

In conclusion, we have developed an efficient methodology for multi-component synthesis of 1-amidoalkyl-2-naphthols at ambient temperature by using zirconocene dichloride as a congruous catalyst. In addition, this method offers several significant advantages such as a mild reaction condition, a clean reaction profile, an easy work up and a high yield.

Compliance with Ethical Standards

Ethical standards The manuscript represents valid work; neither this manuscript nor one with substantially similar content under my authorship has been published or is being considered for publication elsewhere (except as described in the manuscript submission); and copies of any closely related manuscripts are enclosed in the manuscript submission. I confirm that this work is an accurate representation of the trial results.

Conflict of interest The authors declare that they have no conflicts of interest.

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