

Mg(ClO₄)₂ catalysed preparation of 1-amidoalkyl-2-naphthols under solvent-free conditions

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Abstract. A simple, efficient, and practical procedure for the synthesis of amidoalkyl naphthols via multi-component one-pot reaction of 2-naphthol, aldehydes and amides catalysed by Mg(ClO₄)₂ is described in high yields. The present work offers several advantages such as high yields, environmentally benign reaction condition, easy purification and economic availability of the catalyst.

Keywords. 1-Amidoalkyl-2-naphthols; Mg(ClO₄)₂; multi-component reaction; one-pot synthesis; solvent-free conditions.

1. Introduction

Multi-component reactions (MCRs) have been proven to be a very elegant and rapid way to access complex structures in a single synthetic operation from simple building blocks and show high atom economy and high selectivity.¹ Amidoalkyl naphthols are precursors for the synthesis of oxazines which are present in variety of biologically important natural products and potent drugs including a number of nucleoside antibiotics and HIV protease inhibitors, such as ritonavir and lipinavir.² Recently, some procedures for the synthesis of amidoalkyl naphthols are reported using I₂,³ nano-sulphated zirconia,⁴ montmorillonite K₁₀,⁵ bismuth (III) nitrate pentahydrate,⁶ HClO₄.SiO₂,⁷ potassium hydrogensulphate,⁸ silica chloride⁹ and dodecylphosphonic acid (DPA).¹⁰ These protocols, however, suffer from the use of toxic, highly acidic and expensive catalysts and require prolonged reaction times. Furthermore, the yields of the corresponding amidoalkyl naphthols are not always satisfactory. Therefore, the cleaning processes and utilizing eco-friendly and green catalysts, which can be simply recycled at the end of reactions are the main objectives.

Magnesium perchlorate (Mg(ClO₄)₂.xH₂O) as a white crystal, is moisture stable, non-toxic, cheap and commercially available material. Previously, much interest was in the synthesis of 1,5-benzodiazepines,¹¹ the Diels-Alder reaction,¹² the asymmetric reduction of carbonyl compounds,¹³ the Knoevenagel condensation,¹⁴ the synthesis of imines and phenyl hydrazines,¹⁵

and the synthesis of *t*-butyl ethers.¹⁶ In continuation of our research on the applications of solid acids in organic synthesis,¹⁷ we have investigated the one-pot three-component synthesis of amidoalkyl naphthol derivatives via a multi-component condensation reaction from various aryl aldehydes, 2-naphthol and different amides in the presence of Mg(ClO₄)₂.

2. Experimental

Products were characterized by FT-IR (ATR), ¹H-NMR and by comparison of their physical properties with those reported in the literature. FT-IR (ATR) spectra were run on a Bruker, Equinox 55 spectrometer. ¹H-NMR spectra were obtained using a Bruker Avance 400 MHz spectrometer (DRX). Melting points were determined by a Buchi melting point B-540 B.V.CHI apparatus.

3. General procedure

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

A mixture of an aldehyde (1 mmol), 2-naphthol (1 mmol, 0.144 g), amide (1.2 mmol), and Mg(ClO₄)₂.xH₂O (0.03 g) was heated under solvent-free condition in 100°C for appropriate time. After completion of the reaction, for isolation of catalyst the mixture was dissolved in hot chloroform and filtered. The solvent of resulted filtrate was evaporated and the pure product was obtained by recrystallization from ethanol.

*For correspondence

3.2 Selected spectroscopic data

3.2a *N*-[(2-hydroxynaphthalen-1-yl)-*p*-nitrophenyl-methyl]acetamide (table 2, entry 2): FT-IR (ATR, neat) = 3390, 2900–3400, 1638, 1618, 1521, 1438, 1350, 1280, 1245, 1169, 982, 825 cm⁻¹. ¹H NMR (400 MHz, DMSO-d₆): δ = 2.02 (s, 3H), 7.13 (d, *J* = 9 Hz, 1H), 7.17 (d, *J* = 9 Hz, 1H), 7.22 (t, *J* = 7.6 Hz, 1H), 7.34 (d, *J* = 8.6 Hz, 2H), 7.38 (br s, 1H), 7.64 (d, *J* = 8.8 Hz, 1H), 7.7 (d, *J* = 8 Hz, 1H), 7.91 (br s, 1H), 7.97 (d, *J* = 8.6 Hz, 2H), 8.03 (d, *J* = 8 Hz, 1H), 9.72 (s, 1H) ppm. ¹³C NMR (100 MHz, DMSO-d₆): δ = 22.98, 48.34, 118.32, 118.86, 123.05, 123.38, 123.70, 127.18, 127.59, 128.90, 129.14, 130.35, 132.65, 146.35, 151.69, 153.80, 170.23 ppm.

3.2b *N*-[(2-hydroxynaphthalen-1-yl)-3-phenylpropyl]acetamide (table 2, entry 4): FT-IR (ATR, neat) = 3421, 3350–3050, 1636, 1582, 1495, 1516, 740, 703.

¹H NMR (400 MHz, DMSO-d₆): δ = 1.99 (s, 3H), 2.3–2.8 (m, 4H), 7.12 (d, *J* = 7.6 Hz, 1H), 7.16 (d, *J* = 7.2 Hz, 1H), 7.23 (m, 3H), 7.32 (t, *J* = 7.6 Hz, 2H), 7.43 (t, *J* = 7.6 Hz, 2H), 7.66 (d, *J* = 8.8 Hz, 1H), 7.77 (d, *J* = 8 Hz, 1H), 8.01 (brs, 1H) ppm. ¹³C NMR (100 MHz, DMSO-d₆): δ = 23.25, 33.03, 36.01, 46.22, 119.11, 120.08, 122.73, 123.02, 126.12, 126.56, 128.68, 128.71, 128.95, 132.75, 142.30, 153.60, 169.09 ppm.

3.2c *N*-[(2-hydroxynaphthalen-1-yl)-2-naphthyl-methyl]acetamide (table 2, entry 9): FT-IR (ATR, neat) = 3443, 3058, 1648, 1582, 1538, 1512, 1437, 1335, 845, 778. ¹H NMR (400 MHz, DMSO-d₆): δ = 1.99 (s, 3H), 7.22 (d, *J* = 6.4 Hz, 1H), 7.5 (d, *J* = 8.4 Hz, 1H), 7.38–7.5 (m, 4H), 7.62 (d, *J* = 8 Hz, 1H), 7.73–7.85 (m, 3H), 7.91 (d, *J* = 7.2 Hz, 1H), 7.97 (d, *J* = 8.4 Hz, 1H), 8.02 (d, *J* = 8.4 Hz, 1H), 8.72 (d, *J* = 8 Hz, 1H), 10 (s, 1H).

3.2d *N*-[(2-hydroxynaphthalen-1-yl)-*p*-dimethylamino-phenyl-methyl]acetamide (table 2, entry 13): FT-IR (ATR, neat) = 3394, 3175, 1648, 1600, 1583, 1515, 1435, 1339, 1276, 808. ¹H NMR (400 MHz, DMSO-d₆): δ = 1.93 (s, 3H), 2.78 (s, 6H), 6.59 (d, *J* = 8 Hz, 2H), 6.98 (m, 3H), 7.19 (d, *J* = 8.8 Hz, 1H), 7.24 (t, *J* = 8 Hz, 1H), 7.33 (sbr, 1H), 7.72 (d, *J* = 8.8 Hz, 1H), 7.77 (d, *J* = 8 Hz, 1H), 7.86 (sbr, 1H), 8.39 (d, *J* = 8.4 Hz, 1H), 9.94 (s, 1H).

3.2e *N*-[(2-hydroxynaphthalen-1-yl)-3-pyridyl-methyl]acetamide (table 2, entry 16): FT-IR (ATR, neat) = 3413, 3300–3000, 1646, 1577, 1539, 1512, 1437, 1370,

1333, 1274, 778. ¹H NMR (400 MHz, DMSO-d₆): δ = 1.98 (s, 3H), 7.14 (d, *J* = 7.6 Hz, 1H), 7.21 (d, *J* = 8.8 Hz, 1H), 7.26 (brs, 2H), 7.39 (brs, 1H), 7.51 (d, *J* = 6.8 Hz, 1H), 7.81 (m, 3H), 8.36 (brs, 2H), 8.55 (d, *J* = 7.2 Hz, 1H), 10.11 (s, 1H) ppm.

3.2f *N*-[(2-hydroxynaphthalen-1-yl)-*p*-nitrophenyl-methyl]benzamide (table 2, entry 18): FT-IR (ATR, neat) = 3420, 3062, 1627, 1572, 1534, 1489, 1347, 1271, 1026, 942, 822, 750 cm⁻¹. ¹H NMR (400 MHz, DMSO-d₆): δ = 7.25 (m, 8H), 7.47 (t, *J* = 6.4 Hz, 3H), 7.53 (d, *J* = 6.8 Hz, 1H), 7.82 (m, 4H), 8.07 (d, *J* = 8 Hz, 1H), 9.03 (d, *J* = 8 Hz, 1H), 10.34 (br s, 1H) ppm. ¹³C NMR (100 MHz, DMSO-d₆): δ = 49.51, 117.86, 119.03, 123.11, 123.25, 123.86, 127.46, 127.91, 128.03, 128.90, 129.19, 130.45, 132.05, 132.75, 134.44, 146.61, 150.76, 153.94, 166.77 ppm.

3.2g *N*-[(2-hydroxynaphthalen-1-yl)-*p*-nitrophenyl-methyl]carbamate (table 2, entry 19): FT-IR (ATR, neat) = 3390, 3000–3250, 1680, 1625, 1602, 1438, 1342, 1247, 1046, 945, 851. ¹H NMR (400 MHz, DMSO-d₆): δ = 3.7 (s, 3H), 6.93 (d, *J* = 7.6 Hz, 1H), 7.2 (d, *J* = 8.8, 1H), 7.3 (t, *J* = 7.2 Hz, 1H), 7.4 (t, *J* = 7.3 Hz, 1H), 7.5 (d, *J* = 8 Hz, 2H), 7.8 (d, *J* = 9.6 Hz, 2H), 7.85 (s, 1H), 7.9 (brs, 1H), 8.15 (d, *J* = 8 Hz, 1H), 10.2 (s, 1H) ppm. ¹³C NMR (100 MHz, DMSO-d₆): 50.71, 52.27, 118.43, 118.84, 123.13, 123.80, 127.29, 127.61, 128.84, 129.13, 130.37, 132.41, 146.53, 151.17, 153.60, 157.23 ppm.

3.2h *N*-[(2-hydroxynaphthalen-1-yl)-*p*-nitrophenyl-methyl]acrylamide (table 2, entry 21): FT-IR (ATR, neat) = 3411, 3000–3200, 1650, 1624, 1602, 1439, 1514, 1506, 1340, 822, 747. ¹H NMR (400 MHz, DMSO-d₆): δ = 5.6 (d, *J* = 9.6 Hz, 1H), 6.1 (d, *J* = 16 Hz, 1H), 6.6 (dd, *J* = 16 and 9.6 Hz, 1H), 7.2 (m, 3H), 7.37 (m, 3H), 7.82 (m, 2H), 8.1 (d, *J* = 8.4 Hz, 2H), 8.85 (brs, 1H), 10.16 (s, 1H) ppm.

3.2i *N*-[(2-hydroxyphenol-1-yl)-*p*-nitrophenyl-methyl]benzamide (scheme 4): FT-IR (ATR, neat) = 3383, 3300–3200, 1649, 1579, 1486, 1548, 1505, 1342, 852, 717. ¹H NMR (400 MHz, DMSO-d₆): δ = 7.06 (brs, 1H), 7.49 (brs, 3H), 7.55 (brs, 2H), 7.73 (brs, 2H), 7.92 (brs, 4H), 8.25 (brs, 2H), 9.21 (brs, 2H) ppm. ¹³C NMR (100 MHz, DMSO-d₆): 49.51, 117.86, 119.03, 123.11, 123.25, 123.86, 127.46, 127.91, 128.03, 128.90, 129.19, 130.45, 132.05, 132.75, 134.44, 146.61, 150.76, 153.94, 166.77 ppm

3.2j *N, N'-(4-nitrophenylmethylene)diacetamide* (compound 2, scheme 5): FT-IR (ATR, neat) = 3262, 3110, 1667, 1605, 1372, 1564, 1511, 1351, 825. ^1H NMR (400 MHz, DMSO- d_6): δ = 1.86 (s, 3H), 6.53 (brs, 1H), 7.55 (d, J = 8 Hz, 2H), 8.21 (d, J = 8 Hz, 2H), 8.71 (d, J = 6.8 Hz, 2H) ppm.

3.2k *N-[(2-hydroxynaphthalene-1-yl)-p-nitrophenyl-methyl]aniline* (scheme 2): FT-IR (ATR, neat) = 3400-3000, 1601, 1456, 1341, 1510, 846, 743. ^1H NMR (400 MHz, DMSO- d_6): δ = 6.57 (d, J = 8 Hz, 2 H), 6.76 (d, J = 8 Hz, 2 H), 7.01 (d, J = 8 Hz, 1H), 7.08 (d, J = 8.4 Hz, 1H), 7.24 (t, J = 8 Hz, 1H), 7.3 (d, J = 8 Hz, 1H), 7.38 (t, J = 8.4 Hz, 1H), 7.56 (d, J = 8 Hz, 1H), 7.65 (d, J = 8 Hz, 1H), 7.75 (t, J = 8 Hz, 2H), 8.1 (t, J = 8 Hz, 1H), 8.15 (d, J = 8.4 Hz, 2H), 9.74 (s, N-H), 10.3 (s, OH).

4. Results and discussion

In order to optimize the reaction conditions, we studied the reaction of 2-naphthol (1 mmol), 4-nitrobenzaldehyde (1 mmol) and acetamide (1.2 mmol) as a model reaction in various reaction conditions (table 1).

The model reaction was done at different temperatures and solvents in the presence of $\text{Mg}(\text{ClO}_4)_2 \cdot x\text{H}_2\text{O}$. The best condition was solvent-free at 100 °C. In order to evaluate the appropriate catalyst loading, the model reaction was carried out using 0.01 g, 0.02 g, 0.03 g and

0.04 g of $(\text{Mg}(\text{ClO}_4)_2 \cdot x\text{H}_2\text{O})$ at 100 °C without solvent. It was found that the reaction in the absence of the catalyst failed to give the product and 0.03 g was the best quantity of the catalyst. Thus, we have prepared a range of amidoalkyl naphthols under the optimized reaction conditions (scheme 1, table 2).

The scope of the reaction was also investigated with aliphatic aldehydes and α , β -unsaturated aldehydes. The reaction with aliphatic aldehydes such as 3-phenyl propionaldehyde and α , β -unsaturated aldehydes such as cinnamaldehyde gave the corresponding amidoalkyl naphthols in lower yield (table 2, entries 4 and 7). The aromatic aldehydes with electron-withdrawing groups have reacted well in faster rate and shorter time than aromatic aldehydes with electron-donating groups (table 2, entries 2 and 10).

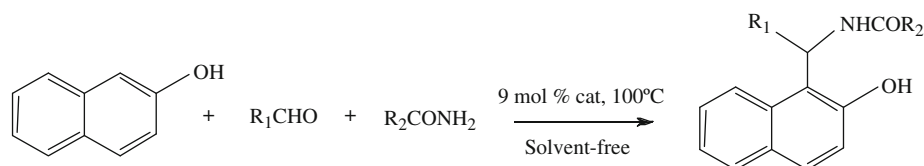
The reactions of methyl carbamate, *N*-methylurea or acrylamide with 2-naphthol and 4-nitro benzaldehyde were examined and the corresponding products were produced in high yields (table 2, entries 19, 20 and 21). Also aniline was utilized as substrate and *N-[(2-hydroxynaphthalen-1-yl)-p-nitrophenyl-methyl]aniline* was produced as a green solid with high yield (scheme 2). Also, this product was formed by the reaction of 2-naphthol with compound (1) (scheme 3).

The reaction between phenol, 4-nitrobenzaldehyde and benzamide or acetamide have produced *N-[(2-hydroxybenzen-1-yl)-p-nitrophenyl-methyl]benzamide* or *N-[(2-hydroxybenzen-1-yl)-p-nitrophenyl-methyl]acetamide* with good yield (80% and 85%, respectively) (scheme 4).

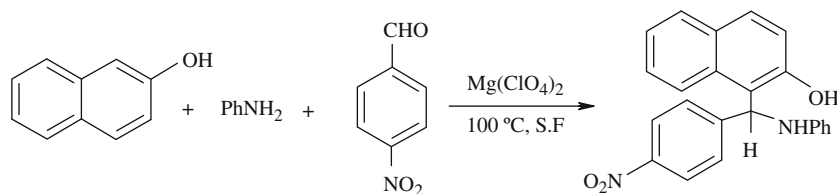
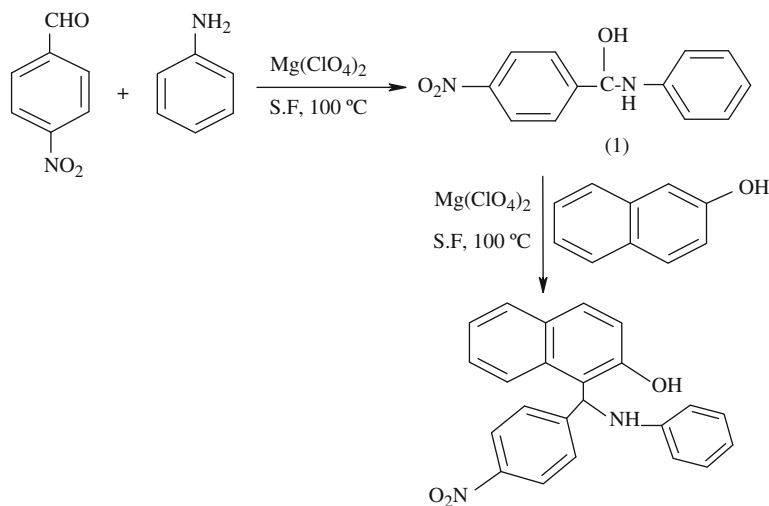
Table 1. Optimization of reaction conditions on the reaction of 2-naphthol, 4-nitrobenzaldehyde, and acetamide under thermal solvent-free conditions in 100 °C.

Entry	Catalyst (gr)	Conditions	Solvent	Time (min/h)	Yield (%)	Ref.
1	–	80 °C	–	45 min	–	–
2	$\text{Mg}(\text{ClO}_4)_2$ (0.02)	60 °C	–	45 min	65	–
3	$\text{Mg}(\text{ClO}_4)_2$ (0.02)	80 °C	–	45 min	73	–
4	$\text{Mg}(\text{ClO}_4)_2$ (0.02)	100 °C	–	45 min	82	–
5	$\text{Mg}(\text{ClO}_4)_2$ (0.02)	120 °C	–	45 min	84	–
6	$\text{Mg}(\text{ClO}_4)_2$ (0.01)	100 °C	–	45 min	60	–
7	$\text{Mg}(\text{ClO}_4)_2$ (0.03)	100 °C	–	45 min	92	–
8	$\text{Mg}(\text{ClO}_4)_2$ (0.04)	100 °C	–	45 min	94	–
9	$\text{Mg}(\text{ClO}_4)_2$ (0.03)	reflux	EtOAc	3 h	70	–
10	$\text{Mg}(\text{ClO}_4)_2$ (0.03)	reflux	CH_2Cl_2	3 h	60	–
11	$\text{Mg}(\text{ClO}_4)_2$ (0.03)	reflux	CHCl_3	3 h	50	–
12	$\text{Mg}(\text{ClO}_4)_2$ (0.03)	reflux	EtOH	3 h	30	–
13	Montmorillonite K10 (0.1 g) ^a	125 °C	–	30 min	96	5
14	Thiamine hydrochloride ^a	80 °C	EtOH	4 h	88	19
15	Iodine (5) ^a	125 °C	–	5 h	89	3
16	Sulphamic acid (0.05 g)	30 °C	CH_2Cl_2	90 min	93	18

^aSynthesis of 1-amidoalkyl-2-naphthols via multi-component reaction of 2-naphthol, 3-nitro benzaldehyde, and acetamide

**Scheme 1.** One-pot synthesis of 1-amidoalkyl-2-naphthols.**Table 2.** Synthesis of 1-amidoalkyl-2-naphthols in the presence of $\text{Mg}(\text{ClO}_4)_2$ at 100 °C.

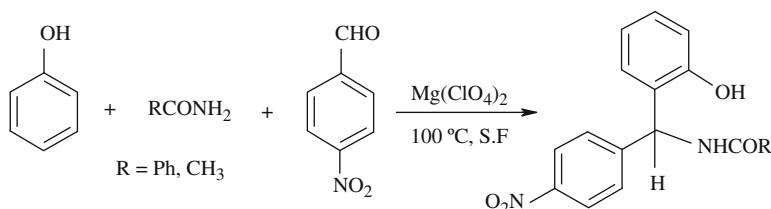
Entry	R ¹	R ²	M.P.	Time (min)	Yield (%)	Ref.
1	C ₆ H ₅	CH ₃	242–243	45	91	3
2	4-NO ₂ C ₆ H ₄	CH ₃	230–232	40	92	7
3	4-ClC ₆ H ₄	CH ₃	231–233	45	88	7
4	C ₆ H ₄ -CH ₂ CH ₂	CH ₃	184–186	60	75	10
5	2, 4-Cl ₂ C ₆ H ₃	CH ₃	201–202	55	86	9
6	C ₆ H ₄ -CH=CH	CH ₃	186–188	60	78	10
7	4-F C ₆ H ₄	CH ₃	230–232	43	90	7
8	C ₁₀ H ₇	CH ₃	238–240	50	89	4
9	2-OHC ₆ H ₄	CH ₃	215–216	70	79	—
10	4-OCH ₃ C ₆ H ₄	CH ₃	191–193	45	83	9
11	3-BrC ₆ H ₄	CH ₃	212–214	50	89	18
12	N(CH ₃) ₂ C ₆ H ₄	CH ₃	191–192	75	82	9
13	2, 5-(OCH ₃) ₂ C ₆ H ₃	CH ₃	245–247	55	87	7
14	4-CH ₃ C ₆ H ₄	CH ₃	218–219	53	87	7
15	C ₅ H ₄ N	CH ₃	194–195	48	89	—
16	C ₆ H ₅	Ph	230–232	38	90	9
17	4-NO ₃ C ₆ H ₄	Ph	218–219	45	89	3
18	4-NO ₂ C ₆ H ₄	Ph	233–235	35	95	9
19	4-NO ₂ C ₆ H ₄	OCH ₃	194–196	35	80	20
20	4-NO ₂ C ₆ H ₄	NHCH ₃	193–194	25	87	—
21	4-NO ₂ C ₆ H ₄	CH=CH ₂	234–235	35	84	—

**Scheme 2.** One-pot synthesis of 1-aminoalkyl-2-naphthols.**Scheme 3.** Suggested mechanism for preparation of 1-aminoalkyl-2-naphthols.

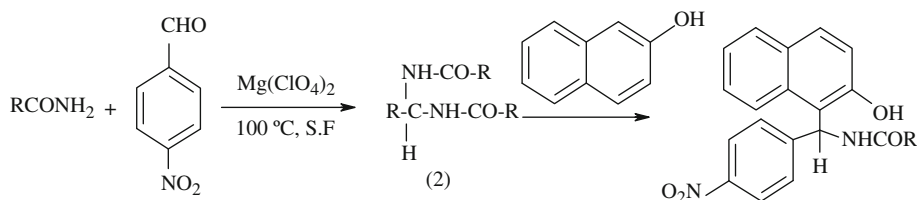
Previously, two types of mechanisms were suggested for 1-amidoalkyl-2-naphthols formation. In the first one, the ortho-quinone methides (O-QMs)³⁻⁹ and in the second one an amidimine¹⁹ were introduced as intermediate. For investigation about mechanism of 1-amidoalkyl-2-naphthols formation, when we have treated 1 mmol of acetamide with 1 mmol of 4-nitrobenzaldehyde in the presence of $\text{Mg}(\text{ClO}_4)_2$, the product (2) was formed, isolated and identified. By reaction of (2) with 2-naphthol in the presence of $\text{Mg}(\text{ClO}_4)_2$, the final product *N*-[(2-hydroxynaphthalen-1-yl)-*p*-nitro phenyl-methyl] acetamide was formed (scheme 5). So we have established a novel and reasonable mechanism for the formation of 1-amidoalkyl-2-naphthols in scheme 6.

To show the merit of the present work in comparison with reported results in the literature, we have compared the reactions of $\text{Mg}(\text{ClO}_4)_2$ with the other catalysts in the synthesis of 1-amidomethyl-2-naphthol derivatives (table 1) and have found that $\text{Mg}(\text{ClO}_4)_2 \cdot x\text{H}_2\text{O}$ is more efficient catalyst for the synthesis of amidoalkyl naphthols than the others.

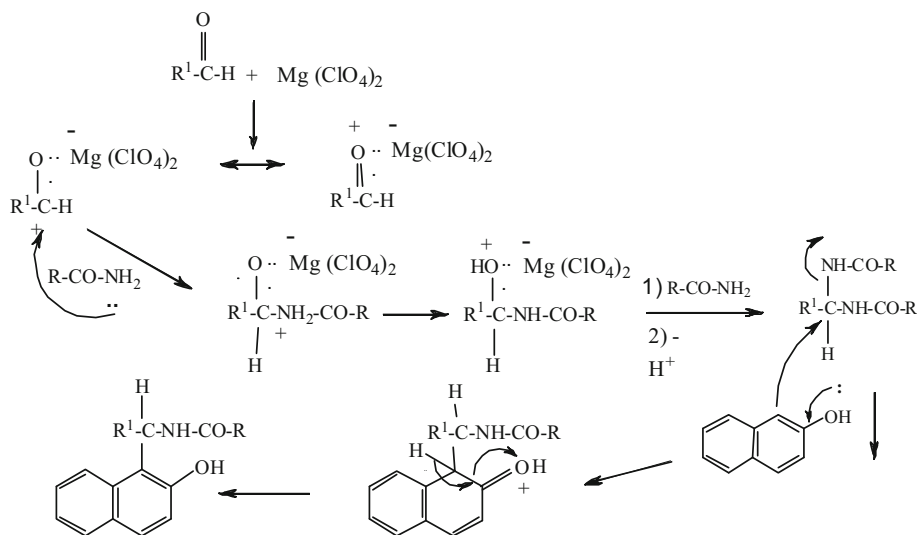
This catalyst is more effective electrophilic activation agent and can accelerate the overall reaction rate. Furthermore, the corresponding amidoalkyl naphthols are produced in high yields and in short reaction time. $\text{Mg}(\text{ClO}_4)_2$ is an inexpensive solid acid catalyst which can be handled easily and removed from the reaction mixtures by simple filtration which makes it a simple,



Scheme 4. One-pot synthesis of amidoalkylphenols.



Scheme 5. Diamid (2) as an intermediate in the preparation of 1-amidoalkyl-2-naphthols.



Scheme 6. Suggested mechanism for preparation of 1-amidoalkyl-2-naphthols.

useful and attractive process for the rapid synthesis of substituted amidoalkyl naphthols.

5. Conclusions

We have developed an improved, and environmentally friendly procedure for the solventless synthesis of 1-amidoalkyl-2-naphthols in the presence of $(\text{Mg}(\text{ClO}_4)_2)$. This methodology offers several advantages such as excellent yields, short reaction times, mild reaction conditions and use of cheap, commercially available, and non-corrosive catalyst.

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