

## COPPER NITRATE TRIHYDRATE CATALYZED EFFICIENT ONE POT SYNTHESIS OF AMIDOALKYL NAPHTHOL DERIVATIVES.

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### ABSTRACT:

In the present study we extend the scope of the  $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$  catalyzed synthesis of amido alkyl naphthols and the results are presented here. In order to optimize the reaction conditions initially we studied the efficacy of  $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$  by taking catalytic amount of 10 mol% and benzaldehyde (1 mmol)  $\beta$ -naphthol (1 mmol) and acetamide (1.2 mmol) in acetonitrile (10 ml) as model reaction. We have developed a practical and new, general efficient procedure for the one pot synthesis of amidoalkyl naphthols by coupling of various aromatic aldehydes with amides urea and  $\beta$ -naphthol using  $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$  as catalyst. The present protocol has several advantages of readily available, inexpensive catalyst, mild reaction conditions, easy handling, excellent yields, greater selectivity, operational and experimental simplicity. We believe that  $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$  catalyzed methodology will definitely be a valuable addition to the existing process in the field of amidoalkyl naphthols.

**Keywords:** Amidoalkyl naphthols,  $\beta$ -naphthol, Multi component reaction,  $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ .

### INTRODUCTION

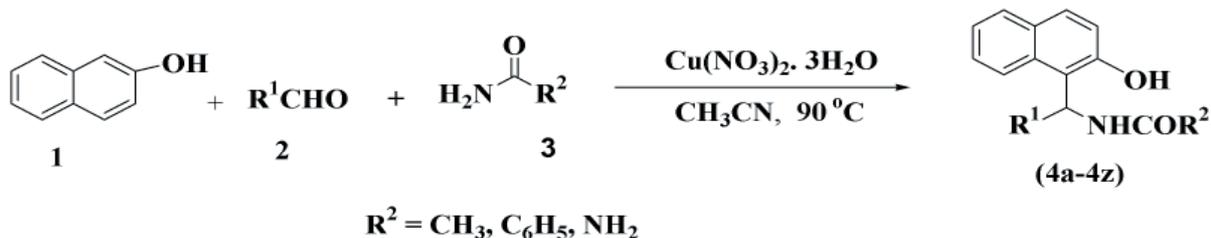
Multicomponent reactions (MCRs) have been proved to be a very elegant and rapid way to access complex structures in a single synthetic operation from simple building blocks, and show high atom-economy, high selectivity and procedural simplicity due to the formation of carbon-carbon and carbon-heteroatom bonds in one-pot and generally afford good yields.<sup>[1]</sup> Multi-component reactions have attracted considerable attention in organic synthesis as they can produce target products in a single operation without isolating the intermediates and thus reducing the reaction times and energy input.<sup>[2, 3]</sup>

Compounds bearing 1,3-amino-oxygenated functional motifs are common in a variety of biologically important natural products and potent drugs, including a number of nucleoside antibiotics and HIV protease inhibitors, such as ritonavir and lipinavir.<sup>[4]</sup> Moreover, 1-amidoalkyl naphthol can be easily hydrolyzed to 1-aminoalkyl naphthol, which shows biological activities like hypotensive, bradycardiac effect and catalyst in asymmetric synthesis.<sup>[5,6]</sup>

The importance of amidoalkyl naphthols for their synthesis has attracted renewed attention and various improved procedures have been reported. Several alternative and efficient methods have been developed for the

synthesis of amidoalkyl naphthol derivatives by multi component reaction of  $\beta$ -naphthol, aldehyde and amide in the presence of different catalysts such as Montmorillonite K10clay,<sup>[7]</sup>  $\text{Ce}(\text{SO}_4)_2$ ,<sup>[8]</sup>  $\text{K}_5\text{CoW}_{12}\text{O}_{40} \cdot 3\text{H}_2\text{O}$ ,<sup>[9]</sup> p-TSA,<sup>[10]</sup> Sulfamic acid/ultrasound,<sup>[11]</sup> Ionic liquids,<sup>[12]</sup> Indion-130,<sup>[13]</sup>  $\text{Al}(\text{H}_2\text{PO}_4)_3$ ,<sup>[14]</sup>  $\text{Fe}(\text{HSO}_4)_3$ ,<sup>[15]</sup>  $\text{Yb}(\text{OTf})_3$ ,<sup>[16]</sup> Wetcyanuric chloride,<sup>[17]</sup>  $\text{Al}_2\text{O}_3\text{-HClO}_4$ ,<sup>[18]</sup> Silica chloride ( $\text{SiO}_2\text{-Cl}$ )/ultrasound,<sup>[19]</sup> indium(III)chloride,<sup>[20]</sup>  $\text{Sr}(\text{OTf})_2$ ,<sup>[21]</sup>  $\text{P}_2\text{O}_5$ ,<sup>[22]</sup>  $\text{NaHSO}_4 \cdot \text{SiO}_2$ ,<sup>[23]</sup>  $\text{H}_4\text{SiW}_{12}\text{O}_{40}$ ,<sup>[24]</sup> Zeolite H-BEA,<sup>[25]</sup> N,N,N',N'-Tetrabromobenzene-1,3-disulfonamide (TBBD),<sup>[26]</sup>  $\text{KHSO}_4$ ,<sup>[27]</sup> tritylchloride<sup>[28]</sup> Bismuth(III) nitrate pentahydrate,<sup>[29]</sup>  $\text{H}_3\text{BO}_3$ ,<sup>[30]</sup>  $\text{Mg}(\text{ClO}_4)_2$ ,<sup>[31]</sup> MNPs- $\text{SO}_3\text{H}$ ,<sup>[32]</sup> and Succinic acid.<sup>[33]</sup> In which some of often involves the use of expensive reagents, hazard solvents and tedious workup.

$\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$  has been extensively used as a mild catalyst for a variety of organic transformations.<sup>[34,35b,37-40]</sup> In continuation of our work to develop new organic transformations,<sup>[34-37]</sup> we would like to report a highly efficient route for the synthesis of amidoalkyl naphthol derivatives by one pot multi component-coupling of  $\beta$ -naphthol, aldehydes, amides/urea catalyzed by commercially available, inexpensive, mild  $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$  as catalyst in good to excellent yields. (Scheme-1)

**Scheme-1****Experimental****MATERIALS AND METHODS**

Chemicals were purchased from Merck and Fluka and directly used for the synthesis. Thin layer chromatography (TLC): precoated silica gel plates (60 F254, 0.2mm layer; E. Merck). <sup>1</sup>HNMR (Avance 300 MHz) spectra were recorded in DMSO using TMS as internal standard. Melting points (m.p.) were determined on a Fischer-Johns melting point apparatus. IR and MS were recorded on a Thermo Nicolet Nexus 670 FT-IR Spectrometer and Finnegan MAT 1020 Mass spectrometer operating at 70 eV.

General procedure for the synthesis of amidoalkyl naphthol derivatives: A mixture of β-naphthol (1 mmol) aldehyde (1mmol) amides (1.2 mmol) or urea (1.2 mmol) and Cu(NO<sub>3</sub>)<sub>2</sub>·3H<sub>2</sub>O (10mol%) in acetonitrile (10 ml) was refluxed at 90°C for the time specified in (Table-3) after completion of the reaction as indicated by TLC, the reaction mixture was cooled to room temperature and washed with water and extracted with ethyl acetate, dried over Na<sub>2</sub>SO<sub>4</sub> concentrated under vacuum and the crude mixture was purified by recrystallization from ethanol to afford pure product.

**Spectral data of the selected compounds:**

*N*-[phenyl-(2-hydroxy-naphthalen-1-yl)methyl]acetamide (**4a**): IR(KBr): 3406, 3250, 2920, 1640, 1583, 1519, 1437, 1369, 1338, 1278, 1275, 1190, 1029, 987, 940, 875, 840, 810, 743 cm<sup>-1</sup>; <sup>1</sup>HNMR (300 MHz, DMSO-d<sub>6</sub>) δ = 9.98 (s, 1H), 8.45–8.42 (d, 1H), 7.84–7.74(m, 3H), 7.37–7.11 (m, 9H), 1.97 (s, 3H) ppm; MS(EI): m/z(%) 292 (M<sup>+</sup>).

*N*-[4-Methyl phenyl-(2-hydroxy-naphthalen-1-yl)methyl]acetamide (**4b**): IR (KBr): 3420, 3320, 3058, 1621, 1580, 1561, 1520, 1466, 1392, 1298, 1202, 1145, 1051, 945, 890, 748, 745, 719

cm<sup>-1</sup>; <sup>1</sup>HNMR (300 MHz, DMSO-d<sub>6</sub>): δ = 1.96 (s, 3H), 2.21 (s, 3H), 7.08-7.03 (m, 5H), 7.19 (d, *J* = 8.8 Hz, 1H), 7.24 (t, *J* = 7.1 Hz, 1H), 7.34 (m, 1H), 7.74 (d, *J* = 8.8 Hz, 1H), 7.78 (d, *J* = 7.9 Hz, 1H), 7.82 (br, 1H), 8.36 (d, *J* = 8.1 Hz, 1H), 9.91 (s, 1H) ppm; MS(EI): m/z (%) 305 (M<sup>+</sup>).

*N*-[4-Methoxy phenyl-(2-hydroxy-naphthalen-1-yl)methyl]acetamide (**4c**): IR(KBr): 3396, 3078, 3002, 2967, 2832, 2787, 2704, 2614, 1627, 1581, 1515, 1438, 1378, 1334, 1304, 1279, 1268, 1179, 1088, 1075, 1043, 983, 930, 880, 850, 822, 814, 8035, 745 cm<sup>-1</sup>; <sup>1</sup>HNMR (300 MHz, DMSO-d<sub>6</sub>) δ = 10 (s, 1H), 8.4 (d, 1H), 7.80–7.73 (m, 4H), 7.35–7.04 (m, 6H), 2.5 (s, 3H), 1.953 (s, 3H) ppm; MS(EI) m/z: 322 (M<sup>+</sup>).

*N*-[4-Nitro phenyl-(2-hydroxy-naphthalen-1-yl)methyl]acetamide (**4i**): IR(KBr): 3391, 3072, 1640, 1602, 1524, 1439, 1352, 1281, 1246, 1167, 1066, 1093, 983, 935, 883, 855, 825, 734, 750 cm<sup>-1</sup>; <sup>1</sup>HNMR (300 MHz, DMSO-d<sub>6</sub>) δ = 10.11 (s, 1H), 8.59–8.56 (d, 1H), 8.14–8.12 (m, 2H), 7.83–7.79 (m, 3H), 7.41–7.38 (m, 3H), 7.28–7.16 (m, 2H), 2.02 (s, 3H) ppm; MS(EI) m/z: 337 (M<sup>+</sup>).

*N*-[2-Hydroxy naphthalen-1-yl-(naphthalen-1-yl)methyl]acetamide (**4m**): IR(KBr): 3420, 3300, 2316, 1659, 1640, 1449, 1523, 748 cm<sup>-1</sup>; <sup>1</sup>HNMR (300 MHz, DMSO-d<sub>6</sub>): δ = 9.41 (s, 1H, OH), 8.52 (d, *J* = 8.5 Hz, 1H), 8.12 (d, *J* = 8.1 Hz, 1H), 7.81–7.85(m, 7H, Ar-H), 7.28–7.34 (m, 6H, Ar-H), 2.88 (s, 3H, CH<sub>3</sub>) ppm; MS(EI) m/z: 341(M<sup>+</sup>).

*N*-[3-Nitro phenyl-(2-hydroxy-naphthalen-1-yl)methyl]benzamide (**4u**): IR (KBr): 3369, 3278, 3099, 2978, 2956, 1640, 1578, 1543, 1522, 1521, 1480, 1439, 1347, 1308, 1280, 1207, 1171, 1093, 1070, 963, 934, 867, 820, 740 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, DMSO-d<sub>6</sub>) δ 10.42 (s, 1H), 9.15–9.13 (d, 1H), 8.11–8.08 (m, 4H), 7.90–7.82 (m, 5H), 7.73–7.71 (m, 2H), 7.61–7.38 (m, 6H) ppm; MS(EI) m/z: 398 (M<sup>+</sup>).

*N*-[4-Nitro phenyl]- (2-hydroxy-naphthalen-1-yl)methyl]urea (**4w**) : IR(KBr): 3481, 3405, 3377, 3179, 3062, 2924, 2849, 1716, 1655, 1600, 1517, 1439, 1346, 1257, 1140, 1109, 1018, 853, 825, 746 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, DMSO-d<sub>6</sub>) δ 9.957 (s, 1H), 7.82–7.73 (m, 3H), 7.41–7.27 (m, 1H), 7.24–7.11 (m, 3H), 6.89(m, 2H), 6.75–6.72 (m, 3H), 5.82 (s, 2H), 3.65 (s, 3H) ppm; MS(EI): m/z (%) 322(M<sup>+</sup>).

## RESULTS AND DISCUSSION

In the present study we extend the scope of the Cu(NO<sub>3</sub>)<sub>2</sub>·3H<sub>2</sub>O catalyzed synthesis of amido alkynaphthol derivatives and the results are presented here. In order to optimize the reaction conditions initially we studied the efficiency of Cu(NO<sub>3</sub>)<sub>2</sub>·3H<sub>2</sub>O by taking catalytic amount of 10 % mol and benzaldehyde (1 mmol) β-naphthol (1 mmol) and acetamide (1.2 mmol) in acetonitrile (10 ml) as model reaction, the reaction gave the corresponding *N*-[phenyl-(2-hydroxynaphthalen-1-yl) methyl]-acetamide with 92% yield in 4hours, with refluxing at 90°C (Table-3). In the absence of Cu(NO<sub>3</sub>)<sub>2</sub>·3H<sub>2</sub>O even up to 6 h no reaction was observed although the amount of catalyst has been optimized to 10% mol, (5% mol) also worked when longer reaction times were employed, while screening of various catalysts we found that Cu(NO<sub>3</sub>)<sub>2</sub>·3H<sub>2</sub>O was more effective than other nitrates tested in terms of isolated yields (92%) (10 Table-1), we choose Cu(NO<sub>3</sub>)<sub>2</sub>·3H<sub>2</sub>O as the suitable catalyst for further reactions due to its easy availability, cost effectiveness, easy handling intrigued by these observations we have then tested the efficiency of several copper salts such as Cu(OAc)<sub>2</sub>, Cu (Cl, Br, I) along with Cu(NO<sub>3</sub>)<sub>2</sub>·3H<sub>2</sub>O on the model reaction and among the copper salts screened Cu(NO<sub>3</sub>)<sub>2</sub>·3H<sub>2</sub>O was found to be best both in terms of reaction time and yields. The model reaction was performed in various solvents using Cu(NO<sub>3</sub>)<sub>2</sub>·3H<sub>2</sub>O as the catalyst to identify the best conditions for the reaction. A range of solvents such as CHCl<sub>3</sub>, DCM, THF, DMSO, toluene, H<sub>2</sub>O and acetonitrile were examined and acetonitrile emerged as the solvent of choice in terms of reaction kinetics and product yields (Table-2). Encouraged by the results obtained for benzaldehyde we generalized the reaction scope

for a number of other aromatic functionalities chloro, fluoro, methyl, methoxy nitro were tolerated and gave good yields.

**Table- 1: Screening of various catalysts (metal nitrates) (10% mol) on model reaction between β -naphthol (1 mmol) benzaldehyde (1mmol), acetamide (1.2 mmol), in acetonitrile (10 ml) heating at 90 °C .**

Entry	Catalyst	Time (h)	Yield (%) <sup>a</sup>
1	Ce(NH <sub>4</sub> ) <sub>2</sub> .(NO <sub>3</sub> ) <sub>6</sub>	6	45
2	Ni(NO <sub>3</sub> ) <sub>3</sub> .6H <sub>2</sub> O	5.5	50
3	Al(NO <sub>3</sub> ) <sub>3</sub> .9H <sub>2</sub> O	6	50
5	Zn(NO <sub>3</sub> ) <sub>2</sub> .6H <sub>2</sub> O	5	60
6	AgNO <sub>3</sub>	5	50
7	Fe(NO <sub>3</sub> ) <sub>3</sub> .9H <sub>2</sub> O	5.5	60
8	Rh(NO <sub>3</sub> ) <sub>3</sub> .2H <sub>2</sub> O	5	76
9	La(NO <sub>3</sub> ) <sub>3</sub> .6H <sub>2</sub> O	5.5	65
10	Cu(NO <sub>3</sub> ) <sub>2</sub> .3H <sub>2</sub> O	4	92

**a = Isolated yields**

**Table- 2: Screening of various solvents ( 10ml) on model reaction between β-naphthol(1 mmol) benzaldehyde (1mmol), acetamide (1.2 mmol), in the presence of Cu(NO<sub>3</sub>)<sub>2</sub>·3H<sub>2</sub>O (10 mol %) heating at 90°C.**

Entry	Solvent	Time (h)	Yield (%) <sup>a</sup>
1	DCM	5.5	40
2	DMF	6	60
3	THF	6	55
4	H <sub>2</sub> O	7	40
5	CH <sub>3</sub> CN	4	92
6	EtOAc	5	54
7	CHCl <sub>3</sub>	5	70
8	DEA	5	68
9	Ethanol	5.5	55

**a = Isolated yields**

Aliphatic aldehydes such as butyraldehyde, propanaldehyde (Table-3) the reaction gave the corresponding amidoalkynaphthol derivatives in

good yield. In case of hereoaromatic aldehyde we have isolated the product with impurity (Table-3). Aromatic aldehydes carrying either electron-withdrawing or electron-donating groups reacted successfully and gave the desired product in high yields. A possible mechanism for this transformation is proposed in the literature,<sup>[7,10, 19, 29, 32]</sup> reaction of  $\beta$ -naphthol with aldehydes

in the presence of lewis acid catalyst is known to give *ortho*-quinone methides (*o*-QMs). The (*o*-QMs) generated insitu, have been reacted with amides via conjugate addition to form 1-amidoalkyl-2-naphthol derivatives (Table-3). On the other hand, the reaction with amine such as aniline was utilized and no amino alkyl naphthols were obtained. (Scheme-2)

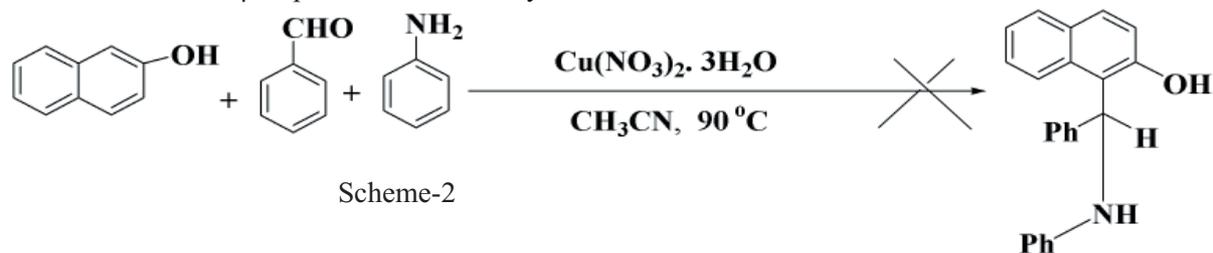


Table-3 Synthesis of amidoalkyl naphthols in the presence of Cupric Nitrate Trihydrate (10mol%)

Entry	R <sup>1</sup>	R <sup>2</sup>	Product <sup>a</sup>	Time (h) <sup>c</sup>	Yield <sup>b</sup> (%)	m.p.	[Ref]
1	C <sub>6</sub> H <sub>5</sub>	CH <sub>3</sub>	4a	4	92	242-244	32
2	4-CH <sub>3</sub> C <sub>6</sub> H <sub>4</sub>	CH <sub>3</sub>	4b	4	90	222-223	32
3	4-OCH <sub>3</sub> C <sub>6</sub> H <sub>4</sub>	CH <sub>3</sub>	4c	4	90	183-184	32
4	2,4 -Cl <sub>2</sub> C <sub>6</sub> H <sub>3</sub>	CH <sub>3</sub>	4d	4.5	85	202-204	32
5	4-ClC <sub>6</sub> H <sub>4</sub>	CH <sub>3</sub>	4e	4.5	89	237-239	33
6	4 -N(CH <sub>3</sub> ) <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	CH <sub>3</sub>	4f	4.5	82	120-123	32
7	C <sub>6</sub> H <sub>4</sub> N	CH <sub>3</sub>	4g	4.5	63	193-194	31
8	2 -HOC <sub>6</sub> H <sub>4</sub>	CH <sub>3</sub>	4h	4	80	216-218	31
9	4 -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	CH <sub>3</sub>	4i	4	92	251-253	33
10	C <sub>6</sub> H <sub>4</sub> -CH=CH	CH <sub>3</sub>	4j	4.5	75	185-188	31
11	2 -ClC <sub>6</sub> H <sub>4</sub>	CH <sub>3</sub>	4k	4.5	80	213-214	32
12	4-FC <sub>6</sub> H <sub>4</sub>	CH <sub>3</sub>	4l	4.5	90	229-231	32
13	C <sub>10</sub> H <sub>7</sub>	CH <sub>3</sub>	4m	4.5	80	228-230	32
14	3-OH4-CH <sub>3</sub> OC <sub>6</sub> H <sub>3</sub>	CH <sub>3</sub>	4n	4	83	237-239	32
15	CH <sub>3</sub> -CH <sub>2</sub> -CH <sub>2</sub>	CH <sub>3</sub>	4o	5	78	224-227	32
16	CH <sub>3</sub> -CH <sub>2</sub>	CH <sub>3</sub>	4p	5	75	178-180	32
17	C <sub>6</sub> H <sub>5</sub>	C <sub>6</sub> H <sub>5</sub>	4q	4	92	238-240	32
18	4 -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	C <sub>6</sub> H <sub>5</sub>	4r	4	90	240-242	33
19	4-CH <sub>3</sub> OC <sub>6</sub> H <sub>4</sub>	C <sub>6</sub> H <sub>5</sub>	4s	4	90	209-212	32
20	2 -ClC <sub>6</sub> H <sub>4</sub>	C <sub>6</sub> H <sub>5</sub>	4t	4.5	85	286-288	32
21	3-NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	C <sub>6</sub> H <sub>5</sub>	4u	4.5	89	233-235	33
22	C <sub>6</sub> H <sub>5</sub>	NH <sub>2</sub>	4v	4	82	179-181	32
23	4 -NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	NH <sub>2</sub>	4w	4	79	163-165	33
24	3-CH <sub>3</sub> OC <sub>6</sub> H <sub>4</sub>	NH <sub>2</sub>	4x	4	80	243-245	33
25	3-NO <sub>2</sub> C <sub>6</sub> H <sub>4</sub>	NH <sub>2</sub>	4y	4	92	191-193	33
26	4-CH <sub>3</sub> C <sub>6</sub> H <sub>4</sub>	NH <sub>2</sub>	4z	4.5	88	115-118	33

a = All the products are characterized by spectral analysis, b = Isolated yields , c = reflux at 90°

**CONCLUSION:**

We have developed a practical and new, general efficient procedure for the one pot synthesis of amidoalkyl naphthol derivatives by coupling of various aromatic aldehydes with amides urea and  $\beta$ -naphthol using  $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$  as catalyst. The present protocol has several advantages of readily available, inexpensive catalyst, mild reaction conditions, easy handling, excellent yields, greater selectivity, operational and experimental simplicity. We believe that  $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$  catalyzed methodology will definitely be a valuable addition to the existing process in the field of amidoalkyl naphthol derivatives.

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