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ORIGINAL ARTICLE

Lanthanum(III) chloride/chloroacetic acid as an efficient and reusable catalytic system for the synthesis of new 1-((2-hydroxynaphthalen-1-yl)(phenyl)methyl) semicarbazides/thiosemicarbazides

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Multi-component reaction;
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Abstract A one-pot, multi-component coupling reaction of aromatic aldehydes, 2-naphthol and semicarbazide (hydrochloride)/thiosemicarbazide using $LaCl_3/ClCH_2COOH$ as an efficient catalyst system under solvent free condition at elevated temperature was investigated. High yields, short reaction time, easy work-up, green environment which requires no toxic organic solvents and reusability of the catalyst are the advantages of this procedure.

1. Introduction

Semicarbazide derivatives are an important class of compounds because they have been found to possess useful biological activities (Bonaiuto et al., 2010; Takahashi et al., 2007; Olivieri et al., 2010). Their tendency to be potent inhibitors of metallo-enzymes has been approved. Thus, the synthesis of semicarbazide derivatives is an important and useful task in organic chemistry.

Multi-component reactions (MCRs) are powerful and useful synthetic tool to produce complex organic molecules due to the formation of carbon–carbon and carbon–heteroatom bonds in a one-pot pathway (Lu et al., 2000; Domling and Ugi, 2000; Zhu and Bienaymé, 2005).

Therefore, the design of novel MCRs has attracted a great attention of the research groups working in medicinal chemistry and drug discovery.

One example of the MCRs is the synthesis of 1-amidoalkyl 2-naphthols which can be carried out by condensation of aryl aldehydes, 2-naphthol and acetonitrile or amide in the presence of different catalysts such as montmorillonite K10 clay (Nagarapu et al., 2007), $HClO_4$–SiO_2 (Shaterian et al., 2008), Iodine (Das et al., 2007), *p*-toluene sulfonic acid (*p*-TSA) (Khodaei et al., 2006), sulfamic acid/ultrasound (Patil et al., 2007a,b), cation-exchange resins (Patil et al., 2007a,b), heteropoly acid (Dorehgiraee et al., 2009; Nagarapu et al., 2007), wet cyanuric chloride (Mahdavinia and Bigdeli, 2009), triytl chloride (Khazaei et al., 2010), P_2O_5 (Nandi et al., 2009),

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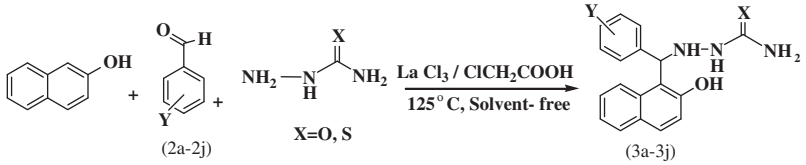


Scheme 1 Synthesis of 1-((2-hydroxynaphthalen-1-yl)(phenyl)methyl)semicarbazides/thiosemicarbazides catalyzed by $LaCl_3$/$ClCH_2COOH$ under solvent free conditions.

4-(1-imidazolium)butane sulfonate (Kundu et al., 2010), *N*-(4-sulfonic acid)butyl triethylammonium hydrogensulfate (Hajipour et al., 2009), 1-butyl-3-methylimidazolium hydrogen sulfate (Sapkal et al., 2009), 1-butyl-3-methylimidazolium bromide ([Bmim] Br) (Zare et al., 2011), and 1-Hexanesulfonic acid sodium salt (Niralwad et al., 2011).

Nevertheless, development and discovery of new MCRs are still in demand. Recently a one-pot, multi-component reaction for the preparation of 4-semicarbazonoalkyl-2-naphthols by reacting aldehydes, 2-naphthol and semicarbazide (hydrochloride) in stoichiometric ratio of 2:1:1 in the presence of PTSA/NaOAC as a catalyst was reported (Foroughifar et al., 2008).

Herein we wish to report the synthesis of 1-((2-hydroxynaphthalen-1-yl)(phenyl)methyl)semicarbazides/thiosemicarbazides, from condensation reaction of aldehyde, 2-naphthol and semicarbazide (hydrochloride) or thiosemicarbazide in stoichiometric ratio of 1:1:1.1 in the presence of $LaCl_3$/$ClCH_2COOH$ as a catalyst (Scheme 1).

2. Experimental section

2.1. Reagents and analysis

Starting materials, solvents, and reagents were either prepared in our laboratories or purchased from Merck, Fluka chemical companies, and used without purification. $LaCl_3$ was prepared by unhydrating $LaCl_3{\cdot}7H_2O$ at 140 °C for 24 h. IR spectra were recorded by using a BRUKER FT-IR spectrophotometer with KBr plates, ^1H and ^{13}C NMR spectra were recorded on a Bruker 400-MHz spectrometer in chloroform as a solvent and tetramethylsilan (TMS) as an internal standard. Melting points were recorded on an electrothermal apparatus and were uncorrected.

2.2. General procedure

A mixture of 2-naphthol (1 mmol), semicarbazide/thiosemicarbazide (1.1 mmol), aldehyde (1 mmol), lanthanum(III) chloride (0.03 g, 0.12 mmol), in chloroacetic acid (2 mmol) was heated at 125 °C for an appropriate time (Table 2). The progress of the reaction was checked by TLC (chloroform/petroleum 2/1) and after completion of the reaction, the mixture was diluted with EtOH/H_2O (2/1) and then the crude product was recrystallized from EtOH (96%) to afford the pure product. All of the compounds which were synthesized are unknown compounds and were fully characterized by IR, Mass, ^1H NMR, ^{13}C NMR spectral data and also micro elemental analyses.

Table 1 Optimization of the reaction condition.

No.	Solvent	Condition	Time (h)	Yield (%)
1	THF	Reflux	6 h	0
2	CH_2Cl_2	Reflux	5 h	Trace
3	EtOH	Reflux	5 h	15%
4	$[ET_3N^+][HSO_4^-]$	125 °C	3 h	30%
5	$ClCH_2COOH$	125 °C	55–95 min	95%

Note: ^1H NMR spectra of some compounds clearly exhibit hydrogen bonding between phenolic OH and neighboring N which causes an extensive signal overlap in the aromatic region, therefore complete ^1H NMR characterization was not possible.

2.3. 1-((2-Hydroxynaphthalen-1-yl)(phenyl)methyl) semicarbazide (**3a**)

Mp 183–185 °C; IR (KBr): 3492, 3234, 3063, 1674, 1591, 1247, 815 cm^{-1}; ^1H NMR (400 MHz; $((CD_3)_2CO)$: δ = 6.74(s, 1H, CH), 7.14(brd, 1H, NH), 7.16–7.30(m, 5H, ArH, NH), 7.53(d, J = 7.8 Hz, 2H, ArH), 7.62(t, J = 8.7 Hz, 2H, ArH), 7.67(d, J = 7.5 Hz, 2H, ArH), 7.8(br, 1H, NH), 7.91(d, J = 8 Hz, 2H, ArH), 8.68(s, 1H, OH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 35.40 (CH), 117.62, 117.74, 123.25, 124.40, 125.18, 126.05, 126.29, 126.8, 128.24, 128.33, 128.50, 128.68, 129.01, 131.22, 131.48, 145.69 (C, CH, Ar), 148.54(CO); MS (ESI): m/z (%): 307 [M^+, 3]; Anal. Calcd for $C_{18}H_{17}N_3O_2$: C, 70.36; H, 5.54; N,13.68%. Found: C, 70.53; H, 5.41; N, 13.54%.

2.4. 1-((2-Hydroxynaphthalen-1-yl)(2-chlorophenyl)methyl) semicarbazide (**3b**)

Mp 197–199 °C; IR (KBr): 3450, 3359, 3031, 1590, 1564, 1187, 854 cm^{-1}; ^1H NMR (400 MHz; $CDCl_3$): δ 6.84(s, 1H, CH), 6.94(brd, 2H, NH_2), 7.43–7.51(m, 4H, ArH),7.53(d, J = 8.7 Hz, 2H, ArH), 7.64(d, J = 7.5 Hz, 2H, ArH), 7.76(brd, 1H, NH), 7.84(t, J = 7.5 Hz, 3H, ArH), 8.78(s, 1H, OH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 34.65(CH), 118.04, 118.11, 123.45, 124.44, 126.93, 127.55, 127.87, 127.95, 128.65, 129.07, 129.61, 130.91, 131.77, 131.80(C, CH, Ar), 148.99(CO); MS (ESI): m/z (%) = 342 [M^+, 4]; Anal. Calcd for $C_{18}H_{16}N_3O_2Cl$: C, 63.25; H, 4.68; N,12.30%. Found: C, 63.97; H, 4.92; N; 12.63%.

2.5. 1-((2-Hydroxynaphthalen-1-yl)(4-nitrophenyl)methyl) semicarbazide (**3c**)

Mp 325–327 °C. IR (KBr): 3471, 3163, 3064, 1687, 1514,,1249, 750 cm^{-1}; ^1H NMR (400 MHz; $CDCl_3$): δ 6.64(s, 1H, CH), 7.46–7.62(m, 4H, ArH, NH_2), 7.65(t, J = 7.8 Hz, 2H, ArH), 7.71(d, J = 7.7, 2H, ArH), 7.81(brd, 1H, NH), 7.89 (t, J = 8 Hz, 2H, ArH), 8.04 (d, J = 8.7 Hz, 2H, ArH),

Table 2 Synthesis of 1-((2-hydroxynaphthalen-1-yl)(phenyl)methyl)semicarbazides/thiosemicarbazides.

Entry	Aldehyde	Time (min)	Yield[a] (%)	X	Mp (°C)
2a	PhCHO	70	94	O	183–185
2b	2-ClC_6H_4CHO	50	97	O	292–294
2c	4-$O_2NC_6H_4CHO$	55	94	O	325–327
2d	4-$OMeC_6H_4CHO$	75	87	O	146–148
2e	4-MeC_6H_4CHO	60	92	O	210–212
2f	PhCHO	60	94	S	252–253
2g	4-ClC_6H_4CHO	65	93	S	245–247
2h	4-OHC_6H_3CHO	95	94	S	166–168
2i	3-$O_2NC_6H_4CHO$	65	92	S	201–203

[a] Yields refer to the pure isolated products.

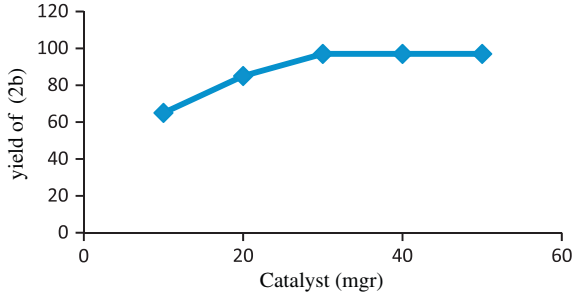


Figure 1 Effect of $LaCl_3$ on the reaction of 2-chlorobenzaldehyde, 2-naphthol and semicarbazide (as hydrochloride) (reactions carried out at 125 °C for 50 min).

8.27(brd, 1H, NH), 8.32(s, 1H, OH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 38.28(CH), 116.19, 118.48, 122.46, 123.32, 124.27, 125.00, 126.65, 127.02, 127.61, 128.43, 129.37, 129.48, 130.01, 131,50, 149.21(C, CH, Ar), 152.41(CO); MS (ESI): *m*/*z* (%) = 353 [M + H, 4]; Anal. Calcd for $C_{18}H_{16}$ N_4O_4: C, 61.36; H, 4.54; N, 15.91%. Found: C,61.97; H, 4.78; N, 15.64%.

*2.6. 1-((2-Hydroxynaphthalen-1-yl)(4-methoxyphenyl)methyl) semicarbazide (**3d**)*

Mp 146–148 °C. IR (KBr): 3381, 3060, 1627, 1595, 1515, 1247, 745 cm^{-1}; ^{1}H NMR (400 MHz; $CDCl_3$): δ 3.78(s, 3H, OMe) 6.46(s, 1H, CH), 7.08 (d, J = 7.5 Hz, 1H, ArH), 7.17(brd, 1H, NH), 7.36–7.57(m, 5H, ArH, NH), 7.61(t, J = 7.5 Hz, 2H, ArH), 7.63(d, J = 7.5 Hz, 2H, ArH), 7.76(brd, 1H, NH), 7.83(d, J = 7.8 Hz, 2H, ArH), 8.32(s, 1H, OH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 48.79(CH), 53.45, 106.45, 109.93, 118.15, 124.06, 126.50, 126.79, 128.19, 129.39, 130.28, 135.02(C, CH, Ar), 155.79(CO); MS (ESI): *m*/*z* (%) = 337 [M, 3]$^+$; Anal. Calcd for $C_{19}H_{19}N_3O_3$: C, 67.65; H, 5.64; N, 12.46%. Found: C, 68.02; H, 5.78; N, 12.94%.

*2.7. 1-((2-Hydroxynaphthalen-1-yl)(4-Methylphenyl)methyl) semicarbazide (**3e**)*

Mp 210–212 °C; IR (KBr): 3463, 3333, 1690, 1572, 1476, 1294, 725 cm^{-1}; ^{1}H NMR (400 MHz; $CDCl_3$): δ = 2.15(s, 3H, Me), 6.49(s, 1H, CH), 6.89(d, J = 8 Hz, 2H, ArH), 7.15(br, 2H, NH_2),7.40–7.58(m, 4H, ArH, NH), 7.60(t, J = 7.5 Hz, 2H, ArH), 7.7–7.8(brd, 1H, NH), 7.82(m, 3H, ArH), 8.43(s, 1H, OH);^{13}C NMR (100 MHz, $CDCl_3$): δ 20.89(Me), 37.62 (CH), 117.45, 118.00, 122.70, 124.21, 126.76, 126.87, 128.09, 128.75, 128.78, 129.17, 129.50, 131.08, 131.45, 135.89, 142.00, 142.11(C, CH, Ar), 148.68(CO); MS (ESI): *m*/*z* (%) = 277, 235, 167, base peak: 143(100%); Anal. Calcd for $C_{19}H_{19}$ N_3O_2: C, 71.03; H, 5.92; N, 13.08%. Found: C, 71.54; H, 5.31; N, 13.73%.

Table 3 Recyclability of the catalyst in the reaction of benzaldehyde and 2-naphthol in the presence of semicarbazide in the presence of $LaCl_3$/$ClCH_2COOH$ under solvent free condition at 125 °C.

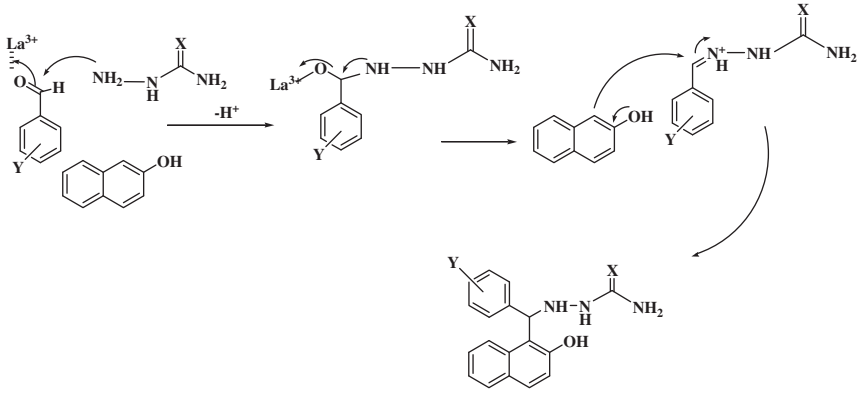
Run no.	Yield (%)
1	94
2	89
3	85

*2.8. 1-((2-Hydroxynaphthalen-1-yl)(phenyl)methyl) thiosemicarbazide (**3f**)*

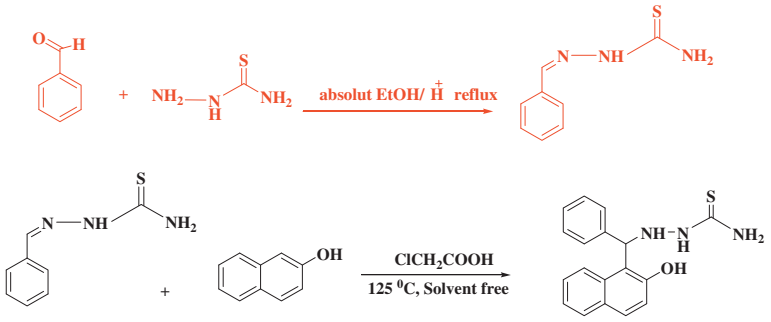
Mp 252–253 °C; IR (KBr): 3487, 3221, 3059, 1665, 1594, 1519, 1247, 748 cm^{-1}; ^{1}H NMR (400 MHz; $CDCl_3$): δ 6.63(s, 1H, CH), 7.35(s, 1H, NH), 7.46(t, J = 7.7 Hz, 2H, ArH), 7.53(d, J = 8.1 Hz, 2H, ArH), 7.62(t, J = 8.9 Hz, 2H, ArH), 7.71–8.1(m, 6H, ArH, NH), 8.27(brd, 1H, NH), 8.32(s, 1H, OH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 53.40(CH), 106.45, 109.93, 118.53, 124.06, 126.50, 126.79, 126.97, 128.19, 129.39, 130.28, 135.02 (C, CH, Ar), 153.74(CO); Anal. Calcd for $C_{18}H_{17}$ N_3OS: C, 66.78; H, 5.26; N, 13.00%. Found: C, 66.47; H, 5.46; N, 12.89%.

*2.9. 1-((2-Hydroxynaphthalen-1-yl)(3-nitrophenyl)methyl) thiosemicarbazide (**3g**)*

Mp 201–203 °C; IR (KBr): 3462, 3335, 2822, 1662, 1572, 1249, 725 cm^{-1}; ^{1}H NMR (400 MHz; $CDCl_3$): δ = 6.49(s, 1H, CH), 7.27(brd, 1H, NH), 7.41–7.50(m, 4H, ArH, NH), 7.55(d, J = 8.0 Hz, 2H, ArH), 7.63(t, J = 7.7 Hz, 3H, ArH), 7.82–7.86(m, 3H, ArH, NH), 8.33(s, 1H, NH), 8.35(s, 1H, OH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 38.04(CH), 118.98, 122.05, 122.43, 123.09, 123.27, 124.98, 126.48, 126.65, 127.64, 128.05, 129.44, 129.76, 129.88, 129.96, 131.40, 131.43, 134.62(C, CH, Ar), 155.37(CO); MS (ESI): *m*/*z* (%) = 281, 179, 149, 89, base peak: 42(100%); Anal. Calcd for $C_{18}H_{16}$ N_4O_3S: C, 58.69; H, 4.35; N, 15.22%. Found: C, 58.97; H, 4.58; N, 15.64%.



Scheme 2 A proposed mechanism for the synthesis of 1-((2-hydroxynaphthalen-1-yl)(phenyl)methyl)semicarbazide/thiosemicarbazide.



Scheme 3 Synthesis of 1-((2-hydroxynaphthalen-1-yl)(phenyl)methyl)semicarbazide/thiosemicarbazide by a two-step reaction.

*2.10. 1-((2-Hydroxynaphthalen-1-yl)(4-hydroxyphenyl) methyl)thiosemicarbazide (**3h**)*

Mp 201–203 °C; IR (KBr): 3381, 3102, 3138, 1614, 1550, 1520, 1254, 746 cm^{-1}; ^1H NMR (400 MHz; $CDCl_3$): δ 6.65(s, 1H, CH), 7.25–7.50(m, 3H, ArH,NH_2), 7.49 (t, J = 7.7 Hz, 2H, ArH), 7.54 (d, J = 8.5 Hz, 2H, ArH), 7.65(t, J = 7.5 Hz, 2H, ArH), 7.81(s, 1H, NH), 7.89–7.91(m, 4H, ArH), 8.41(s, 1H, OH), 8.45(s, 1H, NH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 37.87(CH), 117.19, 120.05, 122.80, 124.79, 127.34, 129.32, 129.52, 130.27, 131.48, 131.67, 131.99(C, CH, Ar), 149.13(CO); Anal. Calcd for $C_{18}H_{17}$ N_3O_2 S: C, 63.72; H, 5.015; N, 12.39%. Found: C, 63.98; H, 5.58; N, 12.24%.

*2.11. 1-((2-Hydroxynaphthalen-1-yl) (4-chlorophenyl)methyl) thiosemicarbazide (**3i**)*

Mp 201–203 °C; IR (KBr): 3288, 3113, 2734, 1620, 1510, 1263, 776 cm^{-1}; ^1H NMR (400 MHz; $CDCl_3$): (δ 6.47s, 1H, CH), 7.08(d, J = 7.8 Hz, 2H, ArH), 7.17(s, 1H, NH), 7.30–7.51(m, 5H, ArH, NH), 7.60(t, J = 7.5 Hz, 2H, ArH), 7.78(brd, 1H, NH), 7.82(m, 2H, ArH), 8.33(s, 1H, OH/NH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 37.87(CH), 117.19, 120.05, 122.80, 124.79, 127.34, 129.32, 129.52, 130.27,131.48, 131.67, 131.99(C, CH, Ar), 149.13(CO); MS (ESI): m/z (%) = 313, 281, 168, base peak: 144(100%); Anal. Calcd for $C_{18}H_{17}$ N_3OS: C, 60.42; H, 4.47; N, 11.75%. Found: C, 60.98; H, 4.58; N, 11.24%.

3. Results and discussion

In order to clarify the roll of $LaCl_3$ as a catalyst, the three-component reaction was carried out in the presence of $ClCH_2COOH$ and in the absence of the catalyst. In this case the reaction needed a longer time to be completed and lower yield was obtained and also increasing reaction temperature does not have any significant affect on the yield of the reaction.

Proper catalyst conditions were obtained by using $LaCl_3$ at different various organic solvents, and the results are shown in Table 1, thus the best results were obtained when the reactions were carried out in the presence of chloroacetic acid.

The amount of $LaCl_3$ was optimized on the reaction of 2-chlorobenzaldehyde, 2-naphtol and semicarbazide (hydrochloride). The results were shown that a proper amount was 0.03 g (0.12 mmol) $LaCl_3$ (Fig. 1).

Encouraged by this result, a wide variety of aromatic aldehydes (**2a**–**2i**) were treated under optimized conditions and afforded the corresponding products (Table 2) in good to excellent yields.

The separated catalyst can be reused after washing with $CHCl_3$ and drying at 100 °C. The reusability of the catalyst was checked by the reaction of benzaldehyde and 2-naphthol in the presence of semicarbazide (hydrochloride) using 0.122 mmol of $LaCl_3/ClCH_2COOH$ under solvent free condition at 125 °C. The results indicate that the catalyst can be used three times without any loss of its activity (Table 3).

A plausible mechanism for the synthesis of 1-((2-hydroxynaphthalen-1-yl)(phenyl)methyl)semicarbazide/thiosemicarbazide has been shown in Scheme 2.

We believe that $LaCl_3$ is coordinated to the carbonyl group of the aromatic aldehyde followed by the attack of the NH_2 group of semicarbazide to the activated carbonyl group, by removing the O that was coordinated to La^{+3}, then the imine group was formed, chloroacetic acid activates imine which will be attacked by 2-naphtol and the final product will be formed.

In order to obtain more information regarding the nature of the reaction, the reaction was run with a different pathway, which was done by a two-step procedure as it has been shown in Scheme 3. The product of two-step reaction and one-pot reaction was identical in both cases.

4. Conclusion

In conclusion, the synthesis of 1-((2-hydroxynaphthalen-1-yl)(phenyl)methyl)semicarbazides/thiosemicarbazides is reported, which is achieved by condensation of semicarbazide (hydrochloride)/thiosemicarbazide, 2-naphthol and aromatic aldehydes in the presence of a catalytic amount of lanthanum(III) chloride/chloroacetic acid as an efficient, green, readily available and environmentally benign catalyst. This new approach provides good yields of the corresponding final products with short reaction times and also simple experimental procedure. In addition it is consistent with the green chemistry approach, since no organic solvent is needed.

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