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Synthesis of bis coumarinyl methanes using of potassium 2-oxoimidazolidine-1,3-diide as a novel, efficient and reusable catalyst

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ABSTRACT

At first, potassium 2-oxoimidazolidine-1,3-diide (POImD) was prepared of stirring a mixture of imidazolidin-2-one, KOH and H_2O overnight. Then, Potassium 2-oxoimidazolidine-1,3-diide was used as a green, novel, fast, efficient and mild catalyst for the synthesis of bis coumarinyl methanes via a one-pot reaction of one equivalent of various aromatic aldehydes and two equivalents of 4-hydroxycoumarin at room temperature in aqueous media. All reactions are performed in the absence of organic solvent in high to excellent yield during short reaction time. The procedure was readily conducted and affords remarkable advantages such as simple work-up, green media and eco-friendly procedure. The catalyst was recovered and reused. Apart from the mild conditions of the process and its excellent results, the simplicity of product isolation and the possibility to recycle the catalyst offer a significant advantage. To the best of our knowledge this is the first report on synthesis of POImD. All of synthesized compounds were characterized by IR, ^1H and ^{13}C NMR spectroscopy and elemental analyses.

1. Introduction

Coumarin constitutes one of the great classes of natural compounds. In the well-known family of coumarins, dimeric coumarins (bis coumarins) occupy an interesting position. Although some types of these compounds could be extracted from plants [1] and interest in its chemistry because of its fitness as pharmaceutically activities. Coumarin has been reported to serve as anti-microbial [2], anti-cancer [3], anticoagulant [4], anti-inflammatory [2] agents. These biological activities of coumarins raised our interest in synthesizing some new coumarins.

Water has a unique media in chemistry and is one of the best solvents, owing to its features such as being eco-friendly, clean, green, nontoxic, non-flammable, safe, low-cost and readily available in organic transformations. Also, the use of aqua media not only diminishes the risk entailed in the use of organic solvents but also improves the rate of many chemical reactions [5-8].

2. Results and Discussion

In continuation of our research for the green synthesis of the spread of neat and efficient procedures for the synthesis of organic and pharmaceutical compounds [9-14], an solvent free, facile and efficient procedure was introduced for the synthesis of bis coumarinylmethane by the POImD catalyzed reaction of two equivalent of 4-hydroxycoumarins and one equivalent of synthesized aldehyde.

Although, synthesis of bis coumarins using catalysts and ionic liquids such as SO_3H-functionalized benzimidazolium cation[15], [bmim]BF_4[16], [MIM$(CH_2)_4SO_3H$][HSO_4] [17], sulfonic acid-functionalized pyridinium chloride[18], choline chloride–oxalic acid [19], $RuCl_3{\cdot}nH_2O$[20] and $NaHSO_4/SiO_2$/Indion 190 resin [21] was reported, However, most of these reported methods suffer from expensive reagents or catalysts, environmental pollution, exotic reaction conditions, long reaction time, complicated operations and unsatisfactory yields. In order to make this reaction simple and green, herein,

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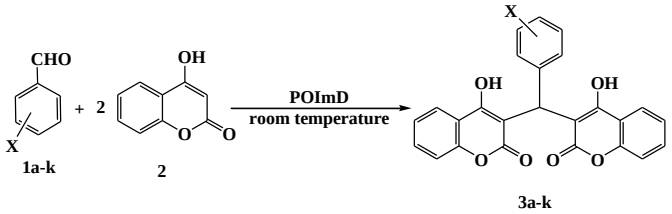
POImD was used to synthesis of bis coumarinylmethanes by the one-pot reaction of 4-hydroxycoumarin and various benzaldehydes at room temperature.



Scheme 1. Synthesis of bis coumarinylmethanes **3a-k** using POImD at room temperature.

The efficiency of POImD rather than some of other catalysts in their catalytic amount was checked for the synthesis of **3a**. As summarized in Table 1, the best result is related to usage of 0.2mmol% of POImD (Table 1; Entry 11).

Table 1. Effect of catalyst on the synthesis of **3a** at room temperature

Entry	Catalyst	Catalyst amount[a]	Time (min)	Yield (%)
1	HCl	5 drops	360	52
2	*L*-proline	0.1mmol	360	65
3	I_2	0.1mmol	480	48
4	K10	0.1g	210	63
5	$ZnCl_2$	0.1g	360	63
6	[BMIm]Br	0.1mmol	150	67
7	[BMIm]OH	0.1mmol	160	72
8	SiO_2	0.1g	360	63
9	PPI[19]	0.3mmol	120	75
10	POImD	0.3mmol	60	98
11	POImD	0.2mmol	60	98
12	POImD	0.1mmol	120	73

[a]Catalyst amount per 1mmol of aldehyde; [b]solvent in the entries 1-5 and 8-12 was water.

To compare this method with catalyst-free reaction, the achieved yields increased and the reaction times were shortened to 60-90 min. On the other hand, this catalyst showed more satisfactory results rather than catalysts such as Fe_3O_4, SiO_2, K10 montmorillonite and potassium phthalimide (PPI) [19]. Because nucleophilic centers in POImD is more powerful for the ionization of 4-hydroxycoumarin (Scheme 2).

Initially, we carried out the reaction of **1a** and 4-hydroxycoumarin in the presence of 0.002 mol of POImD at room temperature under solvent free reaction. This reaction was carried out with 0.001, 0.002 and 0.003mol of POImD, respectively. The best results were obtained using 0.2mmol of the catalyst with complete conversion within 60 min and in 98% yields (Table 2).

Table 2. Synthesis of biscoumarinylmethane **3a-l** using POImD at room temperature

Entry	Product[a]	X	Time (min)	Yield[b] (%)
1	**3a**	4-NO_2	60	98
2	**3b**	3-NO_2	60	96
3	**3c**	2-NO_2	60	94
4	**3d**	4-Br	60	97
5	**3e**	3-Br	75	90
6	**3f**	4-Cl	60	98
7	**3g**	3-Cl	60	92
8	**3h**	2-Cl	90	90
9	**3i**	4-F	60	92
10	**3j**	3-OH	75	90
11	**3k**	2-OH	90	87
12	**3l**	-	60	94

[a]All products were characterized by their physical constant, comparison with authentic samples, IR and NMR spectroscopy; [b]Yields based upon starting aldehyde

To expand the scope and generality of this method, some aldehydes were used as substrate in this reaction. The results were summerized in Table 2. In continuation of our studies, we triggered to synthesize tris-(bis coumarinyl)methane using POImD (Figure 1).

All of compounds summarized in Table 2 were characterized by spectroscopic methods (IR, ^{1}H NMR and ^{13}C NMR) and elemental analysis. So, all of synthesized compounds are new. They were prepared

from pyrazolecarbaldehydes that most of them are not commercially available material. Our experiments also indicated that after five successive runs, recycled ionic liquid showed no loss of efficiency with regard to reaction time and yield (Table 3).

Table 3. Evaluation of reusability of POImD for the synthesis of **3a**

run	1	2	3	4	5
Time(min)	60	60	60	60	60
Yield(%)	98	96	98	97	96

A possible mechanism for the synthesis of bis (coumarinyl)methane derivatives was proposed (Scheme 2). Initially, 4-hydroxycoumarine (**2**) was converted to active form **6** by hydrogen abstraction in the presence of (POImD **4**). Then, the nucleophilic attack of C-3 of intermediate **6** to carbonyl moiety of arylaldehyde (**1**) lead to compound **7**. Finally, nucleophilic attack of the second molecule of activated 4-hydroxycoumarine to intermediate **7** lead to product **3**. It is mentionable that under this procedure catalyst POImD reversibly converts from **4** to **5**.

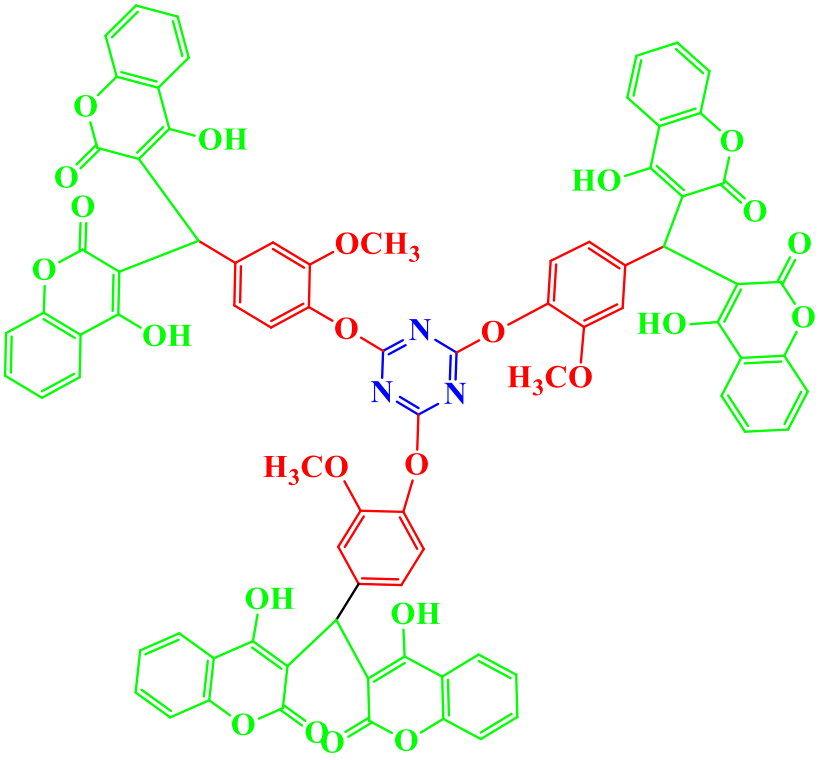
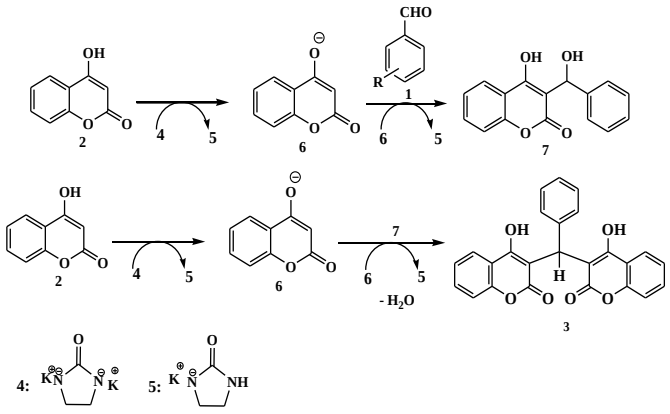


Figure 1. Structure of novel tris-(bis coumarinyl)methane **3l.**



Scheme 2. Proposed mechanism for the synthesis of biscoumarinylmethane using POImD

3. Experimental

3.1. General

Chemicals were purchased from Fluka and Merck. Melting points were measured on an Electrothermal 9100 apparatus. IR spectra were determined on a Shimadzo FT-IR 8600 spectrophotometer. ^{1}H and ^{13}C NMR spectra were determined on a Bruker 400 DRX Avance instrument at 500 and 125 MHz. Elemental analyses were done on a Carlo-Erba EA1110CNNO-S analyzer and agreed with the calculated values. All solvents used were dried and distilled according to standard procedures.

3.2. General procedure for the synthesis of potassium 2-oxoimidazolidine-1,3-diide (POImD)

A mixture of imidazolidin-2-one (20 mmol), KOH (20 mmol) and H_2O (10 mL) was stirred overnight. Following the completion of the reaction, as indicated by TLC, potassium 2-oxoimidazolidine-1,3-diide (POImD) was separated from the reaction mixture by filtration. POImD was purified by recrystallization from EtOH to afford pure products [9].

3.3. General procedure for the synthesis of Compounds 3a-l

A mixture containing aldehyde (1 mmol), 4-hydroxycoumarin (2 mmol), 2mmol% of POImD and 10mL H_2O were stirred at room temperature for the required reaction times. The progress of the reaction was monitored by TLC (EtOAc: petroleum ether 1:3). Having completed the reaction, we extracted the product with $CHCl_3/H_2O$. After separation of phases and evaporation of the organic phase and recrystallization of the residue, the pure product was obtained. The aqueous phase was concentrated under reduced pressure to recover the catalyst for subsequent use.

3.3.1.((4-nitrophenyl)methylene)bis(4-hydroxy-2H-chromen-2-one) (Table 2, Entry 1): Yellow solid; m.p. = 234-236 ˚C; FT-IR: ν_{max} (KBr): 3433, 1662, 1561, 1608, 1520, 1348, 1103 cm^{-1}, ^{1}H NMR (400 MHz, DMSO-d_6): δ= 6.40 (s, 1H), 6.84 (s, br, 2H), 7.30-7.42 (m, 6H), 7.58 (s, br, 2H), 7.88 (d, *J*= 6.7 Hz, 2H), 8.08 (d, *J*= 6.7 Hz, 2H) ppm. ^{13}C NMR (100 MHz, DMSO-d_6): δ= 36.8, 103.4, 116.0, 118.5, 123.3, 123.7, 124.1, 128.2, 131.9, 145.6, 150.1, 152.5, 164.5, 166.4 ppm. Anal Calc. for $C_{25}H_{15}NO_8$: C, 65.65; H, 3.31; N, 3.06. Found: C, 65.63; H, 3.29; N, 3.08.

3.3.2.((3-nitrophenyl)methylene)bis(4-hydroxy-2H-chromen-2-one) (Table 2, Entry 2): Off white solid; mp 234-236 ˚C; FT-IR: ν_{max} (KBr): 3428, 3079, 1658, 1611, 1564, 1528, 1347, 1102, 1058 cm^{-1},^{1}H NMR (400 MHz, DMSO-d_6): δ= 7.27-7.35 (m, 4H), 7.50-7.57 (m, 4H), 7.86 (d, *J*= 8.0 Hz, 1H), 7.92 (s, 1H), 8.03 (d, *J*= 8.0 Hz, 1H). Anal Calc. for $C_{25}H_{15}NO_8$: C, 65.65; H, 3.31; N, 3.06. Found: C, 65.64; H, 3.29; N, 3.07.

3.3.3((2-nitrophenyl)methylene)bis(4-hydroxy-2H-chromen-2-one) (Table 2, Entry 3): Off white solid; m.p. = 245-247 ˚C; FT-IR: ν_{max} (KBr): 3429, 3075, 2930, 1656, 1609,1562, 1354, 1522, 1069 cm^{-1}, ^{1}H NMR (400 MHz, DMSO-d_6): δ= 5.14 (s, 1H), 7.26-7.33 (m, 4H), 7.40 (d, *J*= 7.6 Hz, 2H), 7.42-7.58 (m, 3H), 7.66 (d, *J*= 7.6 Hz, 1H), 7.84 (d, *J*= 8.0 Hz, 2H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ= 34.4, 103.6, 116.1, 117.7, 123.7, 123.8, 124.1, 127.2, 130.0, 131.9, 132.2, 134.7, 149.5, 152.3, 163.4, 165.0. Anal Calc. for $C_{25}H_{15}NO_8$: C, 65.65; H, 3.31; N, 3.06. Found: C, 65.63; H, 3.33; N, 3.05.

3.3.4.((4-bromophenyl)methylene)bis(4-hydroxy-2H-chromen-2-one) (Table 2, Entry 4): Off white solid; m.p. = 285-287 ˚C; FT-IR: ν_{max} (KBr): 3421, 3071, 2938, 1669, 1610, 1561, 1488, 1096, 765 cm^{-1}, ^{1}H NMR (400 MHz, DMSO-d_6): δ= 6.33 (s, 1H), 7.12 (d, *J*= 8.0 Hz, 2H), 7.34-7.42 (m, 4H), 7.59 (t, *J*= 7.6 Hz, 4H), 7.90 (d, *J*= 7.6 Hz, 2H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ= 35.8, 104.0, 116.1, 117.9, 118.7, 123.9, 124.0, 129.2, 131.0, 132.1, 139.7, 152.3, 164.8, 165.4. Anal Calc. for $C_{25}H_{15}BrO_6$: C, 61.12; H, 3.08. Found: C, 61.14; H, 3.10.

3.3.5.((3-bromophenyl)methylene)bis(4-hydroxy-2H-chromen-2-one) (Table 2, Entry 5): Off white solid; m.p. = 242-244 ˚C; FT-IR: ν_{max} (KBr): 3421, 3074, 1664, 1609, 1561, 1498, 1095, 765 cm^{-1},^{1}H NMR (400 MHz, DMSO-d_6): δ= 6.35 (s, 1H), 7.10 (s, br., 3H), 7.31-7.36 (m, 7H), 7.58 (t, *J*= 7.2 Hz, 2H), 7.90 (d, *J*= 7.2 Hz, 2H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ= 36.1, 103.7, 116.0, 118.1, 121.7, 123.8, 124.0, 126.1, 128.6, 129.4, 130.3, 132.0, 143.6, 152.3, 164.7, 165.7. Anal Calc. for $C_{25}H_{15}BrO_6$: C, 61.12; H, 3.08. Found: C, 61.10; H, 3.07.

3.3.6.((4-chlorophenyl)methylene)bis(4-hydroxy-2H-chromen-2-one) (Table 2, Entry 6): Off white solid; m.p. = 256-258 ˚C; FT-IR: ν_{max} (KBr): 3434, 1668, 1613, 1562, 1495, 1093, 767 cm^{-1}, ^{1}H NMR (400 MHz, DMSO-d_6): δ= 6.28 (s, 1H), 7.14 (d, *J*= 8.0 Hz, 2H), 7.24-7.35 (m, 6H), 7.57 (t, *J*= 8.0 Hz, 2H), 7.88 (d, *J*= 8.0 Hz, 2H). Anal Calc. for $C_{25}H_{15}ClO_6$: C, 67.20; H, 3.38. Found: C, 67.22; H, 3.35.

3.3.7.((3-chlorophenyl)methylene)bis(4-hydroxy-2H-chromen-2-one) (Table 2, Entry 7): Off white solid; m.p. = 233-235 ˚C; FT-IR: ν_{max} (KBr): 3398, 3074, 1665, 1610, 1562, 1489, 1098, 764 cm^{-1}, ^{1}H NMR (400 MHz, DMSO-d_6): δ= 6.34 (s, 1H), 7.12-7.37 (m, 10H), 7.59 (t, *J*= 7.2 Hz, 2H), 7.92 (d, *J*= 8.0 Hz, 2H). Anal Calc. for $C_{25}H_{15}ClO_6$: C, 67.20; H, 3.38. Found: C, 67.18; H, 3.37.

3.3.8.((2-chlorophenyl)methylene)bis(4-hydroxy-2H-chromen-2-one) (Table 2, Entry 8): Off white solid; m.p. = 225-227 ˚C; FT-IR: ν_{max} (KBr): 3429, 1658, 1612, 1562, 1495, 1053, 759 cm^{-1}, ^{1}H NMR (400 MHz, DMSO-d_6): δ= 6.16 (s, 1H), 7.16-7.23 (m, 2H), 7.27-7.37 (m, 6H), 7.56 (t, *J*= 7.6 Hz), 7.89 (d, *J*= 8.0 Hz). Anal Calc. for $C_{25}H_{15}ClO_6$: C, 67.20; H, 3.38. Found: C, 67.19; H, 3.32.

3.3.9.((4-fluorophenyl)methylene)bis(4-hydroxy-2H-chromen-2-one) (Table 2, Entry 9): Off white solid; m.p. = 213-215 ˚C; FT-IR: ν_{max} (KBr): 3424, 1670, 1610, 1561, 1501, 1099 cm^{-1}, ^{1}H NMR (400 MHz, DMSO-d_6): δ= 6.36 (s, 1H), 7.05 (t, *J*= 8.0 Hz, 2H), 7.19-7.29 (m, 2H), 7.31 (t, *J*= 8.8 Hz, 2H), 7.37 (d, *J*= 8.0 Hz, 2H), 7.59 (td, *J*= 8.4 , 2.0 Hz, 2H), 7.91 (dd, *J*= 8.0, 2.0 Hz, 2H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ= 35.5, 104.3, 115.0, 116.1, 117.7, 124.0, 128.8,

132.2, 132.8, 135.7, 152.3, 159.5, 161.9, 165.1. Anal Calc. for $C_{25}H_{15}FO_6$: C, 69.77; H, 3.51. Found: C, 69.78; H, 3.48.

3.3.10.((3-hydroxyphenyl)methylene)bis(4-hydroxy-2H-chromen-2-one) (Table 2, Entry 10): Off white solid; m.p. = 256-258 °C; FT-IR: ν_{max} (KBr): 3397, 1655, 1612, 1569, 1489, 1054 cm^{-1}, ^1H NMR (400 MHz, DMSO-d_6): δ= 6.28 (s, 1H), 6.52-6.58 (m, 3H), 7.0 (t, *J*= 8.0 Hz, 2H), 7.30-7.37 (m, 4H), 7.59 (t, *J*= 8.0 Hz, 2H), 7.91 (d, *J*= 8.0 Hz, 2H). Anal Calc. for $C_{25}H_{16}O_7$: C, 70.09; H, 3.76. Found: C, 70.08; H, 3.78.

3.3.11.((2-hydroxyphenyl)methylene)bis(4-hydroxy-2H-chromen-2-one) (Table 2, Entry 11): Off white solid; m.p. = 258-260 °C; FT-IR: ν_{max} (KBr): 3251, 3078, 1710, 1671, 1634, 1571, 1220 cm^{-1}, ^1H NMR (400 MHz, DMSO-d_6): δ= 5.74 (s, 1H), 7.14 (t, *J*= 7.8 Hz, 1H), 7.19 (d, *J*= 8.0 Hz, 1H), 7.31-7.45 (m, 5H), 7.43 (d, *J*= 8.4 Hz, 1H), 7.48 (d, *J*= 8.0 Hz, 1H), 7.59 (t, *J*= 8.0 Hz, 1H), 7.69 (t, *J*= 7.4 Hz, 1H), 8.07 (s, 1H), 8.08 (d, *J*= 7.6, 1H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ= 28.7, 113.8, 116.1, 116.3, 116.5, 122.7, 124.0, 124.6, 125.4, 128.7, 132.3, 132.6, 149.2, 152.0, 156.3, 160.5. Anal Calc. for $C_{25}H_{16}O_7$: C, 70.09; H, 3.76. Found: C, 70.11; H, 3.74.

3.3.12.((((1,3,5-triazine-2,4,6-triyl)tris(oxy))tris(3-methoxybenzene-4,1-diyl))tris(methane triyl))hexakis (4-hydroxy-2H-chromen-2-one) (Table 2, Entry 12): Off white solid; m.p. = 284-286°C; FT-IR: ν_{max} (KBr): 1712, 1652, 1618, 1357, 1263, 1217 cm^{-1},^1H NMR (400 MHz, DMSO-d_6): δ= 8.22-8.41 (m, 4H), 8.14-8.03 (m, 4H), 7.72 (t, 4H, *J*= 7.6 Hz), 7.59 (d, 4H, *J*= 8.2, 2.4 Hz), 7.50 (dd, 3H, *J*= 7.8, 2.2 Hz), 7.45 (d, 3H, *J*= 7.8 Hz), 7.34 (d, 3H, *J*= 2.2 Hz), 5.78 (s, 3H), 3.29 (s, 9H) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ= 169.7, 163.5, 157.6, 154.6, 152.3, 147.6, 136.4, 133.4, 129.5, 127.4, 125.3, 124.2, 123.4, 121.5, 118.5, 113.2, 59.4, 31.9. Anal Calc. for $C_{81}H_{51}N_3O_{24}$: C, 67.08; H, 3.54; N, 2.90. Found: C, 67.05; H, 3.57; N, 2.88.

4. Conclusion

In conclusion, the POImD was investigated as a mild and efficient catalyst for the synthesis of substituted bis coumarin compounds. The remarkable advantages offered by this method are: the catalyst is non-toxic, inexpensive and reusable. Simple work-up procedure, short reaction time, high yields of product with better purity and green aspect by avoiding toxic catalyst and hazardous solvent are another benefits of this procedure. To the best of our knowledge this is the first report on synthesis of bis coumarin compounds using POImD.

Acknowledgements

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