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Novel Synthesis of Aurones by 2-PyONa-catalyzed Regioselective Cyclization of *o*-(Alkynon-1-yl)phenols

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Aurones, 2-benzylidenebenzofuran-3(2*H*)-ones, as structural isomers of flavones are distributed in plants and contribute to yellow coloration of flowers and fruits.[1] They possess diverse biological activities,[2] including antioxidant,[3] anticancer,[4] and antimalarial properties.[5] They are also described as phytoalexins against infections of plants[6] and used for the treatment of Alzheimer's disease[7] and as inhibitors of hepatitis C virus.[8]

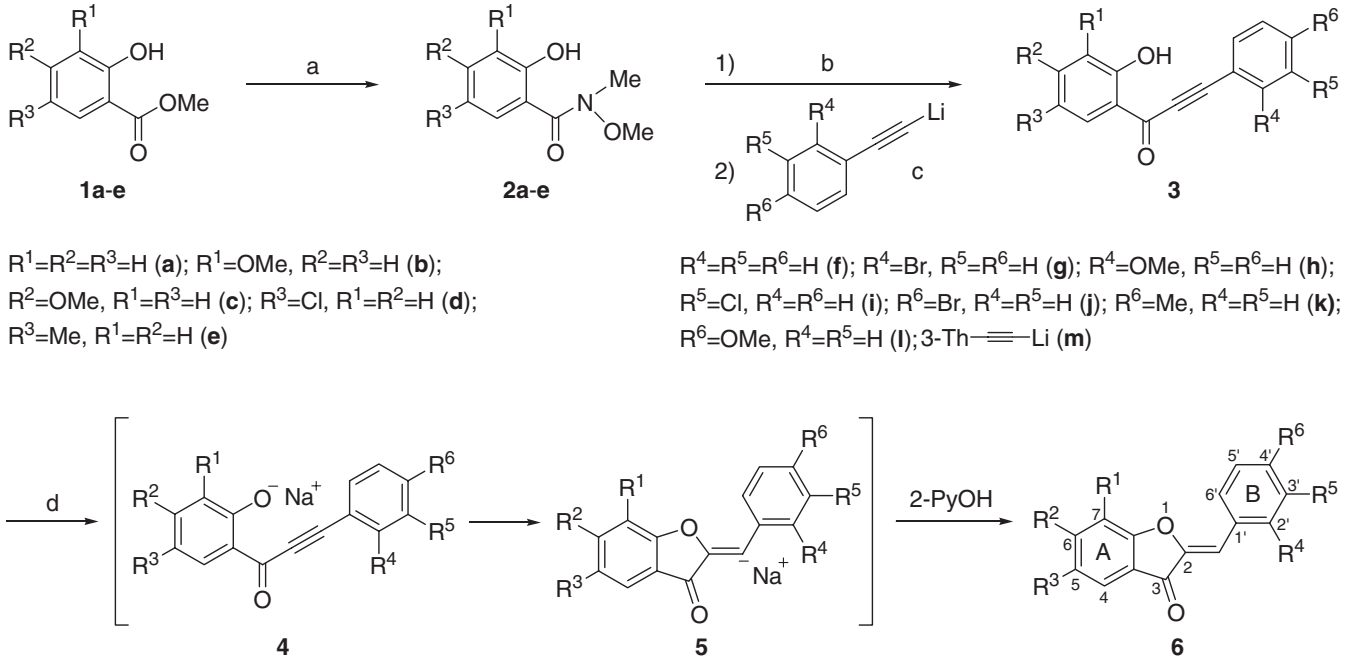
The most common methods for the synthesis of aurones are the condensation of benzofuran-3(2*H*)-ones with aryl aldehydes and the oxidative cyclization of 2′-hydroxychalcones.[9] The intramolecular cyclization of 2-hydroxyphenacyl chlorides with bases such as NaOAc[7] and NaOMe[10] in refluxing CH_3OH or 2-phenoxyacetic acids with polyphosphoric acid[11] at 80°C provided benzofuran-3(2*H*)-ones. These intermediates were then condensed with aryl aldehydes using basic reagents such as KOH[7,10a] and $Ba(OH)_2$[10b] or acidic reagents such as Al_2O_3[11] and ethanolic HCl[12] to afford aurones. The condensation of 2′-hydroxyacetophenones with aryl aldehydes in ethanolic KOH afforded 2′-hydroxychalcones, which were dehydrogenatively cyclized to give aurones by transition metals such as 2 equiv of $Hg(OAc)_2$[13] in HOAc or DMSO and $CuBr_2$[14] in DMF at reflux. 2′-Hydroxychalcones were also cyclized by 3 equiv of $Tl(NO_3)_3$ to give aurones after treatment with *aq* HCl for 10 h at 65°C.[15]

The gold(I) chloride[16] or silver[17] complex-catalyzed cyclization of 2-(1-hydroxy-3-arylprop-2-ynyl)phenols, prepared by the addition of 2 equiv of lithium arylacetylides to salicylaldehydes, in the presence of K_2CO_3 or *i*-Pr_2NEt and subsequent oxidation of the cyclized products with MnO_2 afforded aurones. Alternatively, 2-(1-hydroxy-3-arylprop-2-ynyl)phenols were directly cyclized using 2 equiv of $AgNO_3/K_2CO_3$ in refluxing toluene to give aurones.[18] The intramolecular cyclization of *o*-(alkynon-1-yl)phenols is also useful for the synthesis of aurones. However, the cyclization of *o*-(alkynon-1-yl)phenols using K_2CO_3 or NaOEt was problematic because it provided a mixture of aurones and flavones by 5-*exo* and 6-*endo* attack, respectively.[19] Recently, the use of Lewis bases such as a catalytic PBu_3[20] and 3 equiv of Cs_2CO_3[21] on the 5-*exo* cyclization of *o*-(alkynon-1-yl)phenols afforded aurones together with trace amounts of flavones.

To date, several synthetic methods for aurones have been reported, but some suffer from the use of excess reagent, harsh conditions, and tedious separation. The intramolecular cyclization of *o*-(alkynon-1-yl)phenols is a very convenient method for the synthesis of aurones, but their reports are rare. In this paper, we report that aurones can be novely synthesized by regioselective cyclization of *o*-(alkynon-1-yl)phenols using 0.1 equiv of sodium 2-pyridyloxide (2-PyONa) as a base in high yields.

N-Methoxy-*N*-methyl *o*-hydroxybenzamides (**2a**–**e**) as precursors of *o*-(alkynon-1-yl)phenols (**3**) were easily prepared by treating a mixture of methyl *o*-hydroxybenzamides (**1a**–**e**) and $Me(OMe)NH_2Cl$ with 3 equiv of isopropylmagnesium chloride (Scheme 1). The slow addition of isopropylmagnesium chloride to a slurry solution of **1** and $Me(OMe)NH_2Cl$ in THF at −10°C produced the corresponding phenoxymagnesium chlorides and Me(OMe)NMgCl. The acyl substitution of methoxy group in **1** by Me(OMe)NMgCl proceeded rapidly within 0.5 h to afford **2** in 64–87% yields after the acidic work-up and chromatographic separation (**2a**: 82%, **2b**: 64%, **2c**: 75%, **2d**: 87%, **2e**: 81%). The synthesis of **3** was efficiently accomplished by the reaction of pretreated **2** with lithium diisopropylamide (LDA) and arylethynyllithiums. The reaction seemed to proceed *via* the intermediacy of 5-membered chelates between lithium atom and two oxygen atoms of carbonyl/methoxy groups in **2**, which were converted to **3** by quenching with 1 N HCl solution. The kinds of electron-donating or electron-withdrawing groups in both phenyl rings were compatible for the synthesis of **3**. After the acidic work-up and chromatographic separation, **3** was obtained in 71–90% yields.

To investigate the optimum conditions for the 5-*exo* cyclization of **3**, the effect of bases and solvents was examined for the reaction of 1-(2-hydroxyphenyl)-3-phenyl-2-propyn-1-one (**3af**) (Table 1). The cyclization of **3af** with 0.1 equiv of bases such as 2-PyOLi, 2-PyONa, and 2-PyOK in THF afforded aurone (**6af**, R_f 0.56 in 30% EtOAc/*n*-hexane) in 68, 93, and 57% yields, respectively, after 65°C/10 h, rt/6 h, and rt/1 h, respectively, together with



R^1=R^2=R^3=H (**a**); R^1=OMe, R^2=R^3=H (**b**); R^2=OMe, R^1=R^3=H (**c**); R^3=Cl, R^1=R^2=H (**d**); R^3=Me, R^1=R^2=H (**e**)

R^4=R^5=R^6=H (**f**); R^4=Br, R^5=R^6=H (**g**); R^4=OMe, R^5=R^6=H (**h**); R^5=Cl, R^4=R^6=H (**i**); R^6=Br, R^4=R^5=H (**j**); R^6=Me, R^4=R^5=H (**k**); R^6=OMe, R^4=R^5=H (**l**); 3-Th—≡—Li (**m**)

Scheme 1. Reagents and conditions: (a) Me(OMe)NH_2Cl, 3 equiv of *i*-PrMgCl, THF, −10°C, 0.5 h; 0.5 N HCl; (b) LDA, THF, 0°C, 10 min; (c) THF, −10°C, 20 min; 1 N HCl; (d) 0.1 equiv of 2-PyONa, THF, rt, 1–12 h; 50°C, 3–7 h for **5bf**, **5ck**, **5dh**.

flavone (R_f 0.31 in 30% EtOAc/*n*-hexane) in 21, 3, and 34% yields, respectively. When DME, CH_3OH, and CH_3CN were employed as solvents using 0.1 equiv of 2-PyONa, **6af** was obtained in 89, 48, and 43% yields, respectively, after 1, 1, and 2 h, respectively, at room temperature. Thus, the highest 5-*exo* cyclization of **3** was accomplished with 0.1 equiv of 2-PyONa, which might provide the sodium phenoxides (**4**) having optimized nucleophilicity in less polar THF solvent. The reaction seemed to proceed by the abstraction of hydroxyl proton in **3** by 2-PyONa as a base to produce the corresponding sodium phenoxides (**4**), which underwent 5-*exo* cyclization through kinetic control to afford vinyl carbanion intermediates (**5**). Compound **5** was rapidly protonated by 2-PyOH as a proton source to give aurones (**6**).

The configuration of C=C double bond in synthesized aurones was appeared as (*Z*)-form, which was expected to be more stable than (*E*)-form, avoiding the repulsion between B-ring and carbonyl group in **6**.[22] (*Z*) Assignment of C=C in aurones was based on the ^{1}H chemical shift of the vinylic proton. For example, the chemical shift of the vinylic Cβ proton in (*Z*)-aurone (**6af**) was observed as δ 6.90 ppm, which was consistent with the previous results.[16,20a] This observation could be explained by the diamagnetic shielding of the vinylic proton by carbonyl group and thus the chemical shift of the vinylic proton in (*Z*)-isomer was found at a lower value than that in (*E*)-isomer. In cases of (*Z*)-aurones with 2′-substituents such as methoxy (**5ah**, **5dh**) and bromo (**5eg**), the chemical shifts of the vinylic proton were observed as δ 7.49 (Ref. 3—7.49, Ref. 16—7.48), 7.50, and 7.59 ppm, respectively, and the downfield shift might be attributed to anomeric "*ortho* effect". The cyclization of *o*-(alkynon-1-yl)phenols using 0.1 equiv of 2-PyONa exclusively afforded aurones by highly regioselective 5-*exo* attack and the 6-*endo* cyclized flavones were not obtained to isolable amounts in most cases. As shown in Table 2, various aurones were synthesized from methyl *o*-hydroxybenzoates in high overall yields (47–68%). The conversion of **3** to **6** worked well with both electron-donating methoxy or methyl group and electron-withdrawing chloro group on the A-ring. *o*-(Alkynon-1-yl)phenols possessing methoxy, methyl, bromo, and chloro group on the B-ring were also cyclized as well under the same conditions. Furthermore, the cyclization of 1-(2-hydroxy-5-methylphenyl)-3-(3-thienyl)-2-propyn-1-one proceeded well to give (*Z*)-2-(3-thienylmethylene)-5-methylbenzofuran-3(2*H*)-one (**6em**) in 93% yield.

Table 1. The effect of bases and solvents for the cyclization of 1-(2-hydroxyphenyl)-3-phenyl-2-propyn-1-one (**3af**).[a]

Bases	Solvents	temp, °C; time, h	Yields, %	
			Aurone	Flavone
None	THF	65; 18	0	0
2-PyOLi	THF	65; 10	68	21
2-PyONa	THF	rt; 6	93	3
	DME	rt; 1	89	7
	CH_3OH	rt; 1	48	43
	CH_3CN	rt; 2	43	50
2-PyOK	THF	rt; 1	57	34

[a] 0.1 equiv of bases was used.

Table 2. Synthesis of **3** and aurones **6** from methyl *o*-hydroxybenzoates **1**.[a]

Entry	Chemical name	R^1	R^2	R^3	R^4	R^5	R^6	Isolated yields of **3** and **6** (%)[b]
af	Aurone	H	H	H	H	H	H	87; 93 (66)
ah	2′-Methoxyaurone	H	H	H	OMe	H	H	90; 92 (68)
ai	3′-Chloroaurone	H	H	H	H	Cl	H	86; 95 (67)
al	4′-Methoxyaurone	H	H	H	H	H	OMe	82; 84 (56)
bf	7-Methoxyaurone	OMe	H	H	H	H	H	85; 88[c](48)
ci	3′-Chloro-6-methoxyaurone	H	OMe	H	H	Cl	H	71; 94 (50)
ck	6-Methoxy-4′-methylaurone	H	OMe	H	H	H	Me	76; 82[d](47)
dh	5-Chloro-2′-methoxyaurone	H	H	Cl	OMe	H	H	80; 90 (63)
dj	4′-Bromo-5-chloroaurone	H	H	Cl	H	H	Br	74; 88 (57)
eg	2′-Bromo-5-methylaurone	H	H	Me	Br	H	H	81; 97 (64)
em	2-(3-Thienylmethylene)-5-methylbenzofuran-3(2*H*)-one	H	H	Me	—	3-Th	—	75; 93 (56)

[a] The conversion of **3** to **6** was carried out using 0.1 equiv of 2-PyONa in THF.
[b] The numbers in parentheses indicate the overall yields from methyl *o*-hydroxybenzoates **1**.
[c] 8-Methoxyflavone was obtained in 5% yield.
[d] 7-Methoxy-4′-methylflavone was obtained in 13% yield.

Experimental

Preparation of 1-(2-Hydroxyphenyl)-3-phenyl-2-propyn-1-one (3af). To a pretreated solution of *N*-methoxy-*N*-methyl *o*-hydroxybenzamide (**2a**, 725 mg, 4.0 mmol) with LDA (2.0 M in THF, 2.0 mL, 4.0 mmol) in THF (12 mL) was added a solution of phenylethynyllithium, which was generated from phenylacetylene (490 mg, 4.8 mmol) and methyllithium (1.5 M in Et_2O, 3.2 mL, 4.8 mmol), at −10°C under argon atmosphere. After being stirred for 20 min, the mixture was quenched with 1 N HCl solution, and the solvents were evaporated *in vacuo*. The mixture was diluted with 0.1 N HCl solution (40 mL) and extracted with methylene chloride (3×20 mL). The organic phases were dried over anhydrous $MgSO_4$ and filtered. The filtrate was concentrated and purified by column chromatography on silica gel using 30% EtOAc/*n*-hexane to give **3af** (773 mg, 87%). mp 63-64°C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 11.76 (s, 1H), 8.13 (dd, *J* = 8.3, 1.7 Hz, 1H), 7.67–7.72 (m, 2H), 7.48–7.56 (m, 2H), 7.41–7.47 (m, 2H), 6.96–7.03 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 182.3, 162.8, 137.1, 133.1, 133.0, 131.2, 128.8, 120.8, 119.7, 119.4, 118.2, 96.0, 85.7; FT-IR (KBr) 2207 (C≡C), 1624 (C═O) cm^{-1}; Ms *m/z* (%) 222 (M^+, 100), 194 (61).

Preparation of (*Z*)-Aurone (6af). To a solution of **3af** (667 mg, 3.0 mmol) in THF (9 mL) was added sodium 2-pyridyloxide (35 mg, 0.3 mmol) and stirred for 6 h at room temperature. After evaporation of THF, the mixture was directly subjected to silica gel column chromatography using 30% EtOAc/*n*-hexane to give **6af** (620 mg, 93%) as a yellow solid. mp 110-111°C (Ref. 13a, 108-109°C); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.93 (d, *J* = 7.7 Hz, 2H), 7.81 (d, *J* = 7.7 Hz, 1H), 7.60–7.69 (m, 1H), 7.37–7.50 (m, 3H), 7.34 (d, *J* = 8.4 Hz, 1H), 7.18–7.25 (m, 1H), 6.90 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 184.9, 166.2, 146.9, 137.0, 132.3, 131.6, 130.0, 128.9, 124.7, 123.5, 121.6, 113.1, 113.0; FT-IR (KBr) 1703 (C═O), 1655 (C═C) cm^{-1}; Ms *m/z* (%) 222 (M^+, 88), 221 (100).

6ah, 6al, 6bf. Known compounds.[3,16,20]

(*Z*)-3′-Chloroaurone (**6ai**). mp 108-109°C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.94 (s, 1H), 7.80 (d, *J* = 7.6 Hz, 1H), 7.64–7.75 (m, 2H), 7.33–7.39 (m, 3H), 7.20–7.26 (m, 1H), 6.79 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 184.7, 166.2, 147.3, 137.2, 134.8, 134.0, 130.9, 130.1, 129.8, 129.6, 124.8, 123.8, 121.4, 113.1, 111.2; FT-IR (KBr) 1707 (C═O), 1653 (C═C) cm^{-1}; Ms *m/z* (%) 258 (M^++2, 28), 257 (51), 256 (M^+, 81), 255 (100), 221 (87).

(*Z*)-3′-Chloro-6-methoxyaurone (**6ci**). mp 151-153°C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.93 (s, 1H), 7.64–7.72 (m, 2H), 7.31–7.38 (m, 2H), 6.80 (d, *J* = 2.0 Hz, 1H), 6.76 (dd, *J* = 8.5, 2.1 Hz, 1H), 6.72 (s, 1H), 3.94 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 182.8, 168.7, 167.7, 148.3, 134.8, 134.2, 130.7, 130.0, 129.5, 129.4, 125.9, 114.6, 112.5, 110.0, 96.7, 56.1; FT-IR (KBr) 1701 (C═O), 1657 (C═C) cm^{-1}; Ms *m/z* (%) 288 (M^++2, 24), 287 (42), 286 (M^+, 63), 285 (100).

(*Z*)-6-Methoxy-4′-methylaurone (**6ck**). mp 159-160°C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.1 Hz, 2H), 7.70 (d, *J* = 8.3 Hz, 1H), 7.25 (d, *J* = 8.3 Hz, 2H), 6.80 (s, 1H), 6.73–6.78 (m, 1H), 6.72 (d, *J* = 2.1 Hz, 1H), 3.92 (s, 3H), 2.40 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 183.1, 168.5, 167.3, 147.4, 140.2, 131.4, 129.7 (overlapped), 125.8, 115.0, 112.2, 112.1, 96.6, 56.0, 21.6; FT-IR (KBr) 1693 (C═O), 1652 (C═C) cm^{-1}; Ms *m/z* (%) 266 (M^+, 84), 265 (100), 251 (76).

(*Z*)-5-Chloro-2′-methoxyaurone (**6dh**). mp 206-208°C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.26 (d, *J* = 7.8 Hz, 1H), 7.76 (d, *J* = 2.3 Hz, 1H), 7.58 (dd, *J* = 8.7, 2.3 Hz, 1H), 7.50 (s, 1H), 7.35–7.43 (m, 1H), 7.27 (d, *J* = 8.7 Hz, 1H), 7.02–7.11 (m, 1H), 6.33 (d, *J* = 8.3 Hz, 1H), 3.90 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 183.4, 164.1, 159,0, 147.0, 136.3, 132.1, 131.9, 129.0, 124.2, 123.1, 121.0, 120.9, 114.2, 110.8, 108.4,

55.6; FT-IR (KBr) 1702 (C═O), 1642 (C═C) cm^{-1}; Ms *m/z* (%) 288 (M^++2, 6), 286 (M^+, 18), 257 (37), 255 (100).

(*Z*)-4′-Bromo-5-chloroaurone (**6dj**). mp 195-197°C; ^1H NMR (300 MHz, $CDCl_3$) δ 7.72–7.80 (m, 3H), 7.55–7.64 (m, 3H), 7.29 (dd, *J* = 8.7, 0.8 Hz, 1H), 6.82 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 183.3, 164.2, 147.2, 136.8, 132.9, 132.3, 130.9, 129.4, 124.8, 124.3, 122.7, 114.3, 112.7; FT-IR (KBr) 1710 (C═O), 1648 (C═C) cm^{-1}; Ms *m/z* (%) 338 (M^++4, 26), 336 (M^++2, 100), 334 (M^+, 78), 310 (12), 308 (49), 306 (36), 257 (14), 255 (42).

(*Z*)-2′-Bromo-5-methylaurone (**6eg**). mp 152-153°C; ^1H NMR (300 MHz, $CDCl_3$) δ 8.32 (d, *J* = 7.9 Hz, 1H), 7.65 (d, *J* = 8.0 Hz, 1H), 7.59 (s, 1H), 7.36–7.48 (m, 2H), 7.14–7.28 (m, 3H), 2.40 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 184.7, 164.7, 148.0, 138.2, 133.5, 133.4, 132.4, 132.1, 130.7, 127.7, 126.5, 124.5, 121.4, 112.5, 110.4, 20.8; FT-IR (KBr) 1704 (C═O), 1651 (C═C) cm^{-1}; Ms *m/z*(%) 316 (M^++2, 8), 314 (M^+, 8), 235 (100).

(*Z*)-2-(3-Thienylmethylene)-5-methylbenzofuran-3(2*H*)-one (**6em**). mp 126-128°C; ^1H NMR (300 MHz, $CDCl_3$) δ 7.30 (d, *J* = 2.4 Hz, 1H), 7.61 (d, *J* = 5.0 Hz, 1H), 7.56 (s, 1H), 7.44 (d, *J* = 8.4 Hz, 1H), 7.33 (dd, *J* = 5.0, 2.9 Hz, 1H), 7.19 (d, *J* = 8.4 Hz, 1H), 6.94 (s, 1H), 2.39 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 184.8, 164.3, 146.6, 137.9, 133.9, 133.1, 130.3, 129.3, 126.3, 124.2, 121.9, 112.5, 106.9, 20.8; FT-IR (KBr) 1700 (C═O), 1648 (C═C) cm^{-1}; Ms *m/z* (%) 242 (M^+, 100), 241 (77), 134 (67).

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