Lewis Acid Catalyzed Synthesis of 1-Aryl-1,2-dihydro-naphtho[1,2-e][1,3]oxazin-3-ones under Solvent Free Conditions: A Mechanistic Approach

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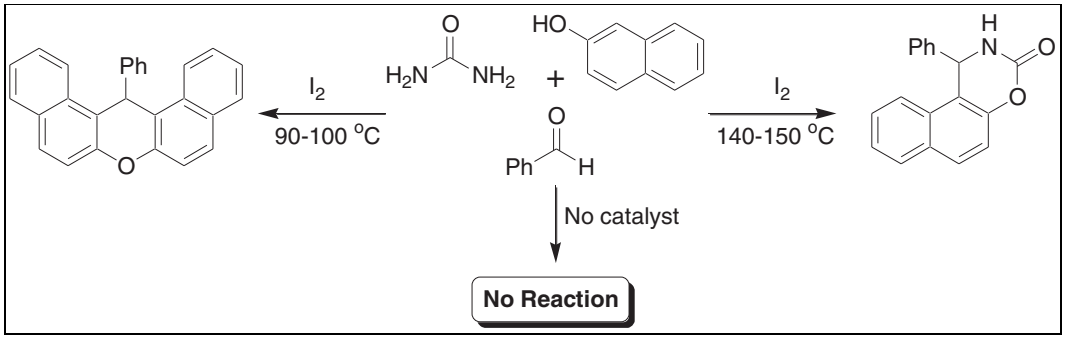

Lewis acids catalyzed highly efficient one-pot three component coupling of β-naphthol, benzaldehydes and urea to produce 1-aryl-1,2-dihydro-naphtho[1,2-e][1,3]oxazin-3-one derivatives under solvent free conditions is described. Mechanistic studies confirmed that product formation is possible only at very high temperature (140–150°C) and at lower temperature (90–100°C) formation of 14-aryl-14*H*-dibenzo(a,j)xanthenes was observed. Among the nine Lewis acids screened, iodine, P_2O_5 and $Yb(OTf)_3$ are found to be most effective catalyst for this multicomponent reaction.

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INTRODUCTION

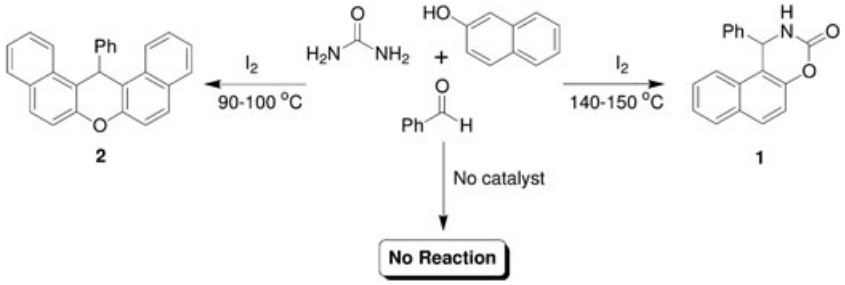
Aromatic-fused oxazinone derivatives belong to relatively small class of heterocycles, but this skeleton has recently been found to be the central part in several antitumor, allergic asthma [1] and antibiotic [2] compounds. Linezolide-based antibiotics and maytansine are important heterocycles that comprises 1,3-oxazinan-2-ones as active pharmacophore [3–5]. 1,3-Oxazinan-2-ones derivatives exhibit a variety of biological activities, and they are being explored as antiinflamatory agents and as agents for treating ulcers, allergies, arthritis, and diabetes [5]. Some 6-phenyl-1,3-oxazinan-2-ones have phosphodiesterase IV inhibitory effects and have been shown to be remedies for inflammatory diseases and antiasthmatics. The six membered 1,3-oxazinan-2-ones are not used as extensively as homologues of 2-oxazolidinones. This is probably due to the difficulties in synthesizing chiral 1,3-oxazinan-2-ones. Apart from their medicinal properties 1,3-oxazinan-2-ones and their analogs were also used as aminopropylation agents [6], as intermediates for the preparation of amino alcohols [7] and as a precursor in the synthesis of phosphinic ligands for the asymmetric catalysis [8,9]. In spite of huge potential, there have been very few synthetic methods reported for their preparation and condensation of amino alkylnaphthols with phosgene or carbonyl diimidazoles in presence of triethyl amine have been the most widely used methods for their preparation.

To our best knowledge, there are only few methods available for the synthesis of substituted 1-aryl-1,2-dihydro-naphtho[1,2-e][1,3]oxazin-3-ones, which involves either toxic chemicals [10–12] or long reaction time [13]. Hence, there is still scope for the development of milder, safer, economical, and more efficient synthetic protocols for the preparation of aromatic-fused oxazinones. The development of mild, low cost, high performance acid catalyst, and replacement of toxic volatile organic solvents as reaction media with nontoxic solvents or reaction under solvent free conditions have been the area of active research in recent years. Inspired by catalytic applications of molecular iodine [14–16], boric acid [17–20], sulfamic acid [21], $FeCl_3$ [22], $SnCl_2$ [23], $InCl_3$ [24], and $Yb(OTf)_3$ [25] in the organic transformation, we decided to compare catalytic potential of these Lewis acids in the synthesis of aromatic-fused 1-aryl-1,2-dihydro-naphtho[1,2-e][1,3]oxazin-3-one derivatives and study their mechanism. Development of a reaction that uses catalytic amounts of mild, nontoxic and readily available reagent should greatly contribute to the creation of environmentally benign processes.

RESULT AND DISCUSSION

Because of several advantages of molecular iodine as a catalyst, initially reaction was performed by the condensation

Scheme 1. Direction of product formation in iodine catalyzed MCRs.



of β-naphthols, benzaldehyde and urea in presence of 10 mol % iodine on a preheated hot plate at 140–150°C, and the reaction yielded 80% desired product within 5 min. To establish the role of catalytic amount of iodine, reaction was carried out in presence of variable amount of iodine, and it was observed that reaction can be carried out even in presence of 5 mol % of iodine without compromising yield and reaction time. To reduce the temperature of reaction, we carried out same reaction using higher catalytic amount of iodine at 90–100°C but interestingly, we observed the formation of 14-phenyl-14*H*-dibenzo[*a,j*]xanthene (**2d**) [25] (Scheme 1) in low yield. Plausible mechanism for the formation of compounds **1** and **2** is shown in Scheme 2. It is evident by mechanism that there are two different key intermediates **3** and **4** that lead the reaction to yield in different products at different temperature. Formation of **4** over **3** at relatively low temperature is reasonable because at lower temperature formation of **3** is kinetically not favored. To further confirm the presence of compound **3** as an intermediate in this reaction, we synthesized compound **3** by literature method [26] (Scheme 3). To confirm the involvement of intermediate **3**, in this transformation, reaction of compound **3** with one mole of β-naphthol in presence of catalytic amount of iodine at 140–150°C was carried out, and desired compound was isolated in very good yield (Scheme 3). This confirms the involvement of intermediate 3, which is formed only at 140–150°C and Lewis acid catalyst is required for this reaction.

Encouraged by this observation, series of benzaldehydes having electron withdrawing, electron donating and neutral functionality at different position of the aromatic ring were reacted with β-naphthols, and urea in presence of catalytic amount of iodine. In all of the cases, product was isolated in 50–95% yield and reaction completes within 5 min (Table 1). When temperature was dropped to 90–100°C, formation of aryl-14*H*-dibenzo(a,j)xanthenes was observed and this class of compounds have been prepared *via* two component condensation of β-naphthol and substituted benzaldehydes at 90–100°C. Existence of intermediate **5** has been reported in this multicomponent synthesis [25]. In the present investigation we believe, urea does not react with substituted benzaldehydes at 90–100°C due to the fact that lone pair electrons of nitrogen are not available for nucleophilic attack at this temperature due to resonance stabilized structure of urea [27], rather substituted benzaldehydes reacts with β-naphthol which leads to the formation of intermediate **5** via **4** and subsequently leads the formation of compound **2** [25]. This study confirmed the involvement of two different intermediates (**3** and **4**) in this reaction resulting in the formation of two different products at different temperature. Compound **2** was isolated in very good yield when two mole of β-naphthol was used instead of one mole.2

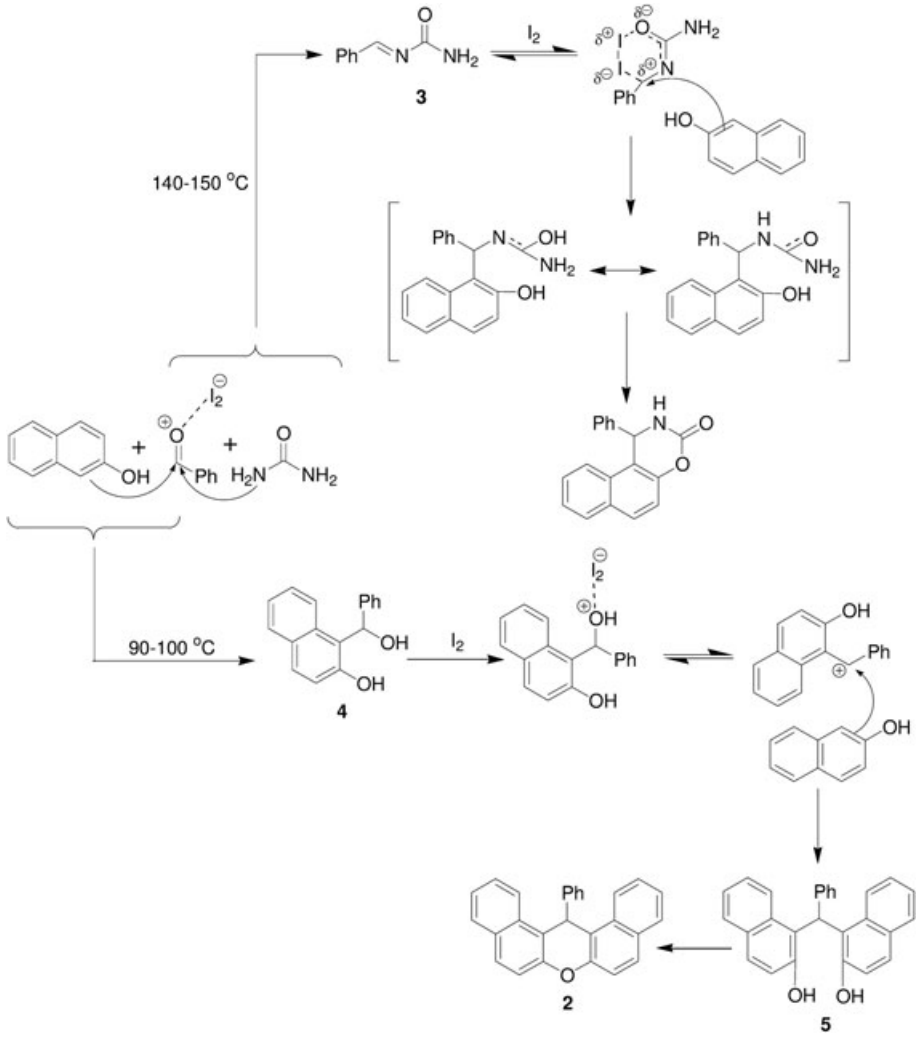
Next, we turned our attention to compare catalytic activity of range of Lewis acids, and reaction was carried out with selected benzaldehydes. Different Lewis acids such as boric acid, P_2O_5, sulfamic acid, $FeCl_3$, $SnCl_2$, $InCl_3$, $Pb(OAc)_4$ and $Yb(OTf)_3$ were explored and it was found that iodine, P_2O_5 and $Yb(OTf)_3$ are the best catalyst in term of yield of the desired product. It is important to mention here that this is the best reaction condition for the synthesis of 1,2-dihydro-1-aryl-naphtho[1,2-e]oxazine-3-ones in term of yield and reaction time. Under the identical reaction conditions, thiourea instead of urea did not yield any product while use of aliphatic aldehydes gave poor yield of the products, possibly due to lower boiling points of aliphatic aldehydes.

EXPERIMENTAL

All of the chemicals used in the synthesis were purchased from Sigma-Aldrich and were used as such. Progress of reaction was monitored by pre-coated Merck silica gel TLC $60F_{254}$ and the spots were detected either by UV or by charring in iodine. Solvents were distilled before use. Melting points were determined on a melting point apparatus and are uncorrected. IR (KBr) spectra were recorded using Perkin-Elmer model 2000 FT-IR spectrophotometer and the values are expressed as ν_{max} cm^{-1}. Mass spectral data were recorded on a Jeol (Japan) JMS-DX303 and micromass LCT, Mass Spectrometer/Data system. The ^1H NMR spectra were recorded on Bruker Spectrospin spectrometer at 300 MHz, Jeol Spectrospin spectrometer at 400 MHz and ^{13}C NMR 75.5 MHz, and 100 MHz respectively using TMS as an internal standard. The chemical shift values are recorded on δ scale and the coupling constants (J) are in Hz.

Experimental procedure. A mixture of benzaldehyde (6.02 mmol), β-naphthol (6.02 mmol), and urea (9.03 mmol) were taken into a 50-mL round bottom flask and stirred at 140–150°C until the reaction mixture became homogeneous, to this

Scheme 2. Proposed mechanism.



reaction mixture iodine (0.3 mmol) was added quickly and the progress of reaction was monitored by thin layer chromatography and reaction takes 5 min to complete. After completion, reaction mixture was allowed to cool at room temperature and quenched by cold water. The aqueous layer was extracted with chloroform, and reaction mixture was dried over anhydrous Na_2SO_4. The crude product was purified by crystallization to yield the pure compound and characterized by spectroscopic techniques.

The products **1a**, **1c**, **1d**, **1g,** and **1i** were previously reported [10] and the spectral data of these products were in agreement with the data reported in literature. The spectral data for new compounds are described below.

1-(3-Nitro-phenyl)-1,2-dihydro-naphtho[1,2-e][1,3]oxazin-3-one (1b). Yield: 70%; mp: 260–262°C; IR (Nujol, cm^{-1}): 3238, 3136, 1760, 1696, 1532, 1345, 1224, 1177; ^{1}H NMR (300 MHz, DMSO-d_6): 6.45 (d, J = 2.7 Hz, 1H, Ph*CH*), 7.39–7.51 (m, 3H), 7.60–7.66 (m, 2H), 7.94–8.14 (m, 4H), 8.26 (d, J = 6.6 Hz, 1H), 9.00 (s, 1H); ^{13}C NMR (75.5 MHz, DMSO-d_6): 52.77, 112.88, 116.89, 121.88, 122.96, 123.07, 125.25, 127.61, 128.69, 130.40, 130.72, 133.44, 144.66, 147.68, 147.93, 149.02; ESI-HRMS calculated for $C_{18}H_{12}N_2O_4$: 320.0797. Found: 320.0796 (M^+).

1-(4-Fluoro-phenyl)-1,2-dihydro-naphtho[1,2-e][1,3]oxazin-3-one (1e). Yield: 87%; mp: 200°C; IR (KBr, cm^{-1}): 3220, 3134, 2958, 1751, 1603, 1508, 1398, 1224, 743; ^{1}H NMR (300 MHz, DMSO-d_6): 6.21 (d, J = 2.4 Hz, 1H, Ph*CH*), 7.10–7.16 (m, 2H), 7.30–7.49 (m, 4H), 7.76 (d, J = 8.1 Hz, 1H), 7.92–7.99 (m, 2H), 8.28 (s, 1H), 8.83 (s, 1H); ^{13}C NMR (75.5 MHz, DMSO-d_6): 52.94, 113.78, 115.56, 115.85, 116.83, 123.01, 125.06, 127.36, 128.60, 129.01, 129.12, 130.29, 130.38, 139.07, 147.37, 149.20, 159.93, 163.17; ESI-HRMS calculated for $C_{18}H_{12}FNO_2$: 293.0852. Found: 293.0849 (M^+).

Scheme 3. Involvement of intermediate (**3**) in the formation of **1**.

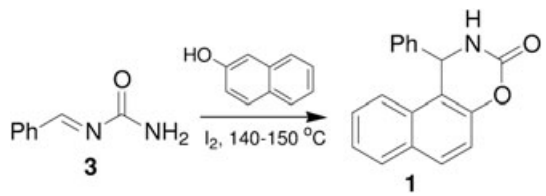
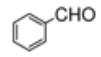
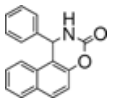
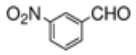
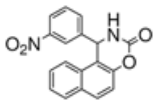
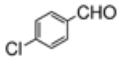
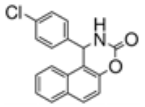
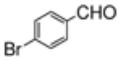
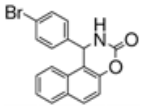
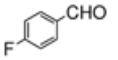
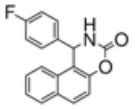
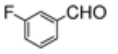
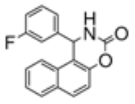
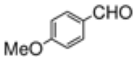
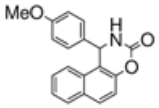
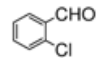
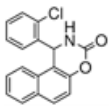
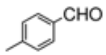
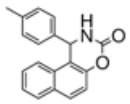
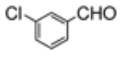
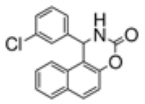
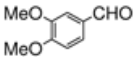
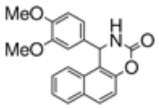
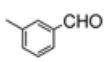
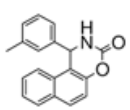


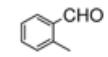
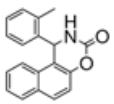
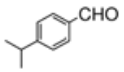
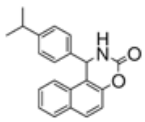
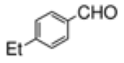
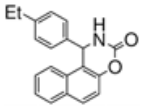
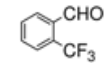
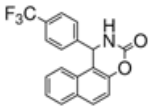
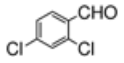
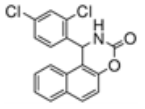
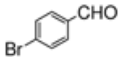
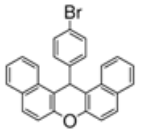
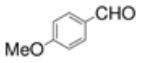
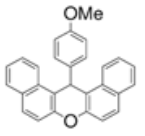
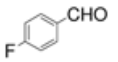
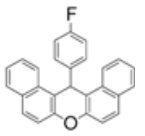
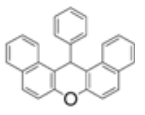
Table 1

One-pot iodine-catalyzed synthesis of 1-aryl-1,2-dihydro-naphtho[1,2-e][1,3]oxazin-3-ones.

Entry	Benzaldehyde	Product	Reaction Temp	Yield (%)	Mp (°C)
1j			140–150	80	220–221[10]
1b			140–150	70	260–262
1c			140–150	93	203–208[10]
1d			140–150	74	220–222[10]
1e			140–150	87	200
1f			140–150	95	250–253
1g			140–150	94	188–190[10]
1h			140–150	89	246–248
1i			140–150	88	170[10]
1j			140–150	66	227–230
1k			140–150	82	200
1l			140–150	86	220–223

(*Continued*)

Table 1
(Continued)

Entry	Benzaldehyde	Product	Reaction Temp	Yield (%)	Mp (°C)
1m			140–150	92	256
1n			140–150	95	241–243
1o			140–150	86	210–213
1p			140–150	93	260–261
1q			140–150	50	250–253
2a			90–100	69	294–296[28,29]
2b			90–100	77	201–204[29,30]
2c			90–100	84	237–240[29,30]
2d			90–100	69	226–228[29,31]

1-(3-Fluoro-phenyl)-1,2-dihydro-naphtho[1,2-e][1,3] oxazin-3-one (1f). Yield: 95%; mp: 250–253°C; IR (Nujol, cm^{-1}): 3134, 1730, 1224, 796; ^1H NMR (300 MHz, DMSO-d_6): 6.23 (d, J = 2.1 Hz, 1H, Ph*CH*), 7.03–7.49 (m, 7H), 7.79 (d, J = 7.8 Hz, 1H), 7.93–8.00 (m, 2H), 8.90 (s, 1H); ^{13}C NMR (75.5 MHz, DMSO-d_6): 53.12, 113.38, 113.83, 114.12, 114.72, 116.85, 122.82, 123.00, 125.13, 127.46, 128.62, 128.80, 130.37, 131.04, 145.42, 147.50, 149.18, 160.51, 163.75; ESI-HRMS calculated for $C_{18}H_{12}FNO_2$: 293.0852. Found: 293.0850 (M^+).

1-(2-Chloro-phenyl)-1,2-dihydro-naphtho[1,2-e][1,3] oxazin-3-one (1h). Yield: 89%; mp: 246–248°C; IR (Nujol, cm^{-1}): 3233, 3139, 1723, 1226, 1123, 761, 747; ^1H NMR (300 MHz, DMSO-d_6): 6.42 (d, J = 2.4 Hz, 1H, Ph*CH*), 7.09–7.50 (m, 8H), 7.85–7.94 (m, 2H), 8.72 (s, 1H); ESI-HRMS calculated for $C_{18}H_{12}ClNO_2$: 309.0557. Found: 309.0551 (M^+), 311.3196 (M^++2).

1-(3-Chloro-phenyl)-1,2-dihydro-naphtho[1,2-e][1,3]oxazin-3-one (1j). Yield: 66%; mp: 227–230°C; IR (KBr, cm^{-1}): 3144, 2957, 1752, 1224, 1179; ^1H NMR (300 MHz, DMSO-d_6): 6.23 (s, 1H, Ph*CH*), 7.14–7.52 (m, 7H), 7.74–781 (m, 1H), 7.93–8.01(m, 2H),

Table 2

Effect of different Lewis acid catalysts on yield.

Catalyst	Yield (%)			
	Entry **1a**	Entry **1b**	Entry **1c**	Entry **1g**
Iodine	80	70	93	82
Boric acid	59	48	31	41
Sulphamic acid	36	31	41	38
$SnCl_2$	30	32	35	39
$FeCl_3$	34	25	32	30
$InCl_2$	40	44	38	41
$Yb(OTf)_3$	71	68	79	72
P_2O_5	82	75	89	86
$Pb(OAc)_4$	46	49	42	39

8.87(s, 1H); ESI-HRMS calculated for $C_{18}H_{12}ClNO_2$: 309.0557. Found: 309.0554 (M^+), 311.0548 (M^++2).

1-(3,4-Dimethoxy-phenyl)-1,2-dihydro-naphtho[1,2-e][1,3] oxazin-3-one (1k):. Yield: 82%; mp: 200°C; IR (Nujol, cm^{-1}): 3149, 3033, 2924, 2854, 1736, 1516, 1221; ^1H NMR (300 MHz, DMSO-d_6): 3.65 (s, 3H, O*CH*$_3$), 3.69 (s, 3H, O*CH*$_3$), 6.10 (d, J = 2.7 Hz, 1H, Ph*CH*), 6.58 (d, J = 8.4 Hz, 1H), 6.80 (d, J = 8.4 Hz, 1H), 7.04 (s, 1H), 7.33–7.49 (m, 3H), 7.78 (d, J = 8.1 Hz, 1H), 7.91–7.98 (m, 2H), 8.73 (s, 1H); ^{13}C NMR (75.5 MHz, DMSO-d_6): 53.45, 55.41, 55.45, 111.07, 111.88, 114.03, 116.77, 118.74, 123.20, 124.99, 127.25, 128.53, 128.96, 130.08, 130.33, 135.22, 147.35, 148.38, 148.70, 149.32; ESI-HRMS calculated for $C_{20}H_{17}NO_2$: 335.3533. Found: 335.3530 (M^+).

1-m-Tolyl-1,2-dihydro-naphtho[1,2-e][1,3]oxazin-3-one (1l). Yield: 86%; mp: 220–223°C; IR (KBr, cm^{-1}): 3141, 2923, 2854, 1751, 1222, 1181, 993; ^1H NMR (300 MHz, DMSO-d_6): 2.23 (s, 3H, Ph*CH*$_3$), 6.13 (d, J = 3 Hz, 1H, Ph*CH*), 7.05–7.23 (m, 4H), 7.36–7.51 (m, 3H), 7.79 (d, J = 8.1 Hz, 1H), 7.93–8.00 (m, 2H), 8.81 (s, 1H); ESI-HRMS calculated for $C_{19}H_{15}NO_2$: 289.1103. Found: 289.1118 (M^+).

1-o-Tolyl-1,2-dihydro-naphtho[1,2-e][1,3]oxazin-3-one (1m). Yield: 92%; mp: 256°C; IR (Nujol, cm^{-1}): 3231, 3140, 2924, 1725, 1377, 1224, 1187, 823; ^1H NMR (300 MHz, DMSO-d_6): 2.65 (s, 3H, Ph*CH*$_3$), 6.33 (d, J = 2.4 Hz, 1H, Ph*CH*), 6.75 (d, J = 7.8 Hz, 1H), 7.02–7.14 (m, 2H), 7.20 (d, J = 7.2 Hz, 1H), 7.35–7.43 (m, 4H), 7.91–8.00 (m, 2H), 8.70 (s, 1H); ESI-HRMS calculated for $C_{19}H_{15}NO_2$: 289.1103. Found: 289.1109 (M^+).

1-(4-Iso-propyl-phenyl)-1,2-dihydro-naphtho[1,2-e][1,3] oxazin-3-one (1n). Yield: 95%; mp: 241–243°C; IR (Nujol, cm^{-1}): 3135, 2924, 2854, 1730, 1221, 1172, 1110; ^1H NMR (300 MHz, DMSO-d_6): 1.10 (d, J = 4.2 Hz, 6H, PhCH(*CH*$_3$)$_2$), 2.74–2.80 (m, 1H, Ph*CH*(CH$_3$)$_2$), 6.11 (d, J = 2.7 Hz, 1H, Ph*CH*), 7.15–7.22 (m, 4H), 7.34–7.47 (m, 3H), 7.79 (d, J = 8.1 Hz, 1H), 7.91–7.98 (m, 2H), 8.76 (s, 1H); ESI-HRMS calculated for $C_{21}H_{19}NO_2$: 317.1416. Found: 317.1419 (M^+).

1-(4-Ethyl-phenyl)-1,2-dihydro-naphtho[1,2-e][1,3]oxazin-3-one (1o). Yield: 86%; mp: 210–213°C; IR (KBr, cm^{-1}): 3256, 3147, 2964, 2928, 1734, 1516, 1222, 810; ^1H NMR (300 MHz, DMSO-d_6): 1.09 (t, J = 7.5 Hz, 3H, PhCH$_2$*CH*$_3$), 2.50 (q, J = 7.5 Hz, 2H, Ph*CH*$_2$CH$_3$), 6.14 (d, J = 2.7 Hz, 1H, Ph*CH*), 7.13 (d, J = 8.1 Hz, 2H), 7.20 (d, J = 8.1 Hz, 2H), 7.36–7.50 (m, 3H), 7.80 (d, J = 8.1 Hz, 1H), 7.92–7.99 (m, 2H), 8.81 (s, 1H); ESI-HRMS calculated for $C_{20}H_{17}NO_2$: 303.1259. Found: 303.1252 (M^+).

1-(2-Trifluoromethyl-phenyl)-1,2-dihydro-naphtho[1,2-e] [1,3]oxazin-3-one (1p). Yield: 93%; mp: 260–261°C; IR (KBr, cm^{-1}): 3226, 3140, 1723, 1394, 1224, 1118, 754; ^1H NMR (300 MHz, DMSO-d_6): 6.33 (s, 1H, Ph*CH*), 6.77 (d, J = 7.5 Hz, 1H), 7.04 (t, J = 7.2 Hz, 1H), 7.13 (t, J = 7.2 Hz, 1H), 7.24 (d, J = 7.5 Hz, 1H), 7.35–7.42 (m, 4H), 7.91–8.00 (m, 2H), 8.70 (s, 1H); ^{13}C NMR (75.5 MHz, DMSO-d_6): 56.04, 118.90, 121.95, 127.92, 130.25, 132.11, 132.27, 132.66, 133.23, 133.88, 134.07, 135.48, 135.66, 136.22, 140.42, 145.76, 153.08, 154.24; ESI-HRMS calculated for $C_{19}H_{15}NO_2$: 343.0820. Found: 343.0819 (M^+).

1-(2,4-Dichloro-phenyl)-1,2-dihydro-naphtho[1,2-e][1,3] oxazin- 3-one (1q). Yield: 50%; mp: 250–253°C; IR (KBr, cm^{-1}): 3255, 3246, 3143, 1737, 1392, 1226, 839, 742; ^1H NMR (300 MHz, DMSO-d_6): 6.48 (s, 1H, Ph*CH*), 7.18–7.48 (m, 6H), 7.66 (s, 1H), 7.92–8.01 (m, 2H), 8.89 (s, 1H); ESI-HRMS calculated for $C_{18}H_{11}Cl_2NO_2$: 343.0167. Found: 343.0168 (M^+), 345.0174 (M^++2), 347.0171 (M^++4).

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