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Ring closing and opening reactions leading to aza-polycyclic aromatic compounds

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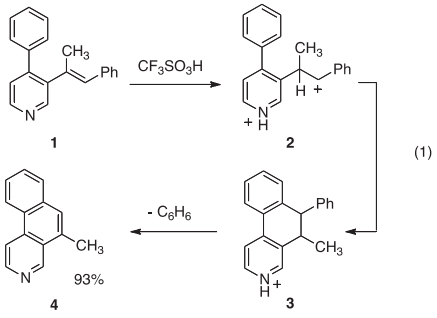
ABSTRACT

A series of functionalized aza-polycyclic aromatic compounds were prepared by a superacid-promoted ring closing and opening reaction cascade. A reaction mechanism is proposed, which involves reactive dicationic intermediates. A key step in the conversions involves *ipso* protonation of an aryl group and elimination of an alkyl phenyl group.

1. Introduction

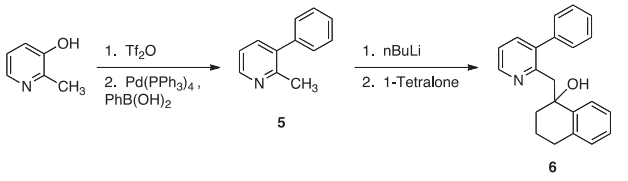
Aza-polycyclic aromatic compounds are substances useful in material science applications as they can have interesting optical properties.[1] The substances can also have a variety of biological activities.[2] As such, the aza-polycyclic aromatic compounds have been the frequent targets of synthetic methods.[3] Our own superacid-promoted reactions gave aza-polycyclic aromatic compounds by reactions involving dicationic intermediates (Eq. 1).[4] For example, compound **1** is diprotonated to give ion **2** which cyclizes to compound **4**. The reaction involves cyclization of **2** followed by an unusual benzene elimination step. This reaction step is envisioned to occur by an *ipso*-protonation of the phenyl group on intermediate **3**. While interesting from a mechanistic standpoint, the benzene elimination step only serves as a means of generating the new π-bond. Other than the aromatic ring, no functionality is produced on the arene by this reaction step. In this manuscript, we describe chemistry leading to aza-polycyclic aromatic compounds with additional aryl-alkyl groups. The products are formed by a ring-closure, ring-opening cascade.



(1)

2. Results

We reasoned that indane and tetraline-type substituent groups could lead to phenethyl-substituted aza-polycyclic aromatic compounds by a ring-closure and ring-opening sequence. To test this hypothesis, a series of heterocyclic alcohols were prepared and reacted with superacidic CF_3SO_3H (Table 1). The alcohol substrates were prepared from the reactions of organolithium reagents with tetralones and other ketones (Eq. 2).



(2)

For example, 3-hydroxy-2-methylpyridine is converted to compound **5** by standard Suzuki coupling methods.[5] Methyl deprotonation is readily accomplished with 2-methylpyridines (2-

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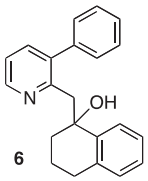
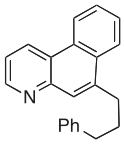
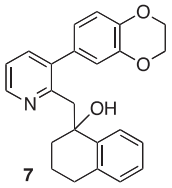
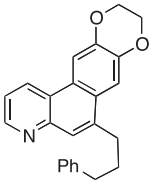
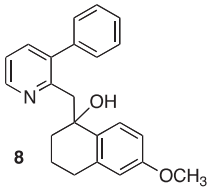
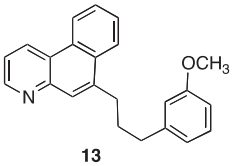
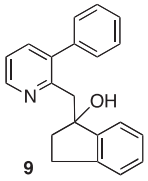
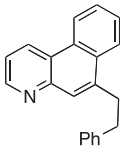
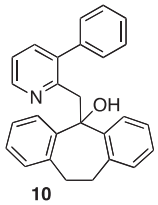
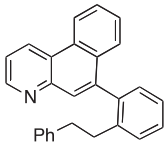
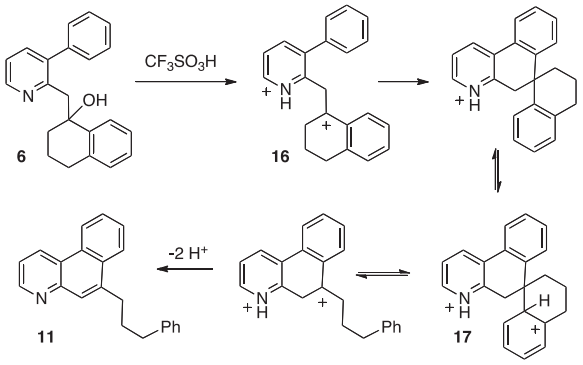
Table 1
Benzo[*f*]quinolone products from the reactions of alcohols **6**–**10**

Starting material	Product	Yield[a] (%)
 **6**	 **11**	98
 **7**	 **12**	99
 **8**	 **13**	96
 **9**	 **14**	89
 **10**	 **15**	99

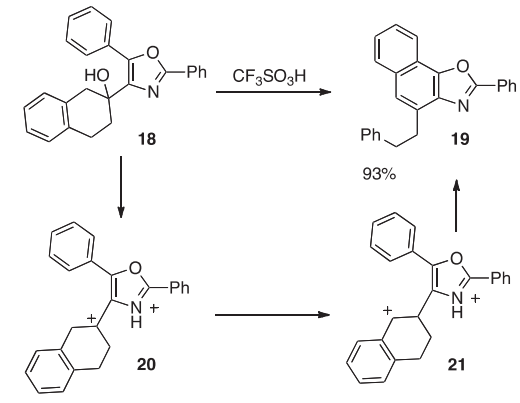
[a] Isolated yield.

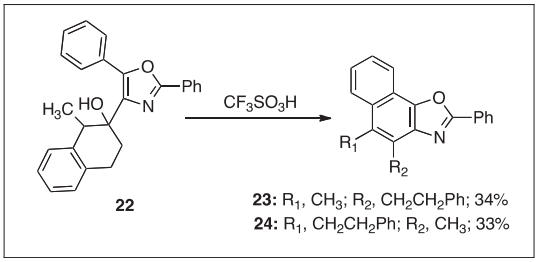
picolines),[6] so compound **5** provides substrate **6** from 1-tetralone. When compound **6** is reacted in superacid, the benzo[*f*]quinolone derivative (**11**) is formed in quantitative yield (Table 1). As expected, the tetraline ring system provided the 3-phenylpropyl substituent group. With the 1,4-benzodioxan derivative, cyclization occurs at the 5-position to give product **12**.[7] Product **12** is likewise formed in nearly quantitative yield with the 3-phenylpropyl substituent group. Utilization of a methoxy-substituted tetralone gives alcohol **8** and reaction with superacid yields the benzo[*f*]quinolone derivative (**13**) having the 3-(4-methoxyphenyl)propyl group. Reaction of the indan-derivative (**9**) forms the phenethyl-substituted benzo[*f*]quinolone (**14**) in good yield. Reaction of compound **10** leads to formation of a 2-phenethylphenyl-substituted benzo[*f*]quinolone (**15**).

The conversions to aza-polycyclic aromatic compounds are thought to occur by a series of reaction steps involving dicationic intermediates (Scheme 1). Initial ionization of the pyridyl and hydroxyl groups leads to dication **16**, which forms the spirocyclic intermediate. The superacidic media leads to protonation of arene carbons[8] so dication **17** is formed. This leads to ring opening and product formation. Previously, we described chemistry leading to aza-polycyclic aromatic compounds and a key step involved charge migration.[4a] We have found that substrates may undergo charge migration, ring closure, and then ring opening reaction steps (Scheme 2). Thus, compound **18** was prepared from 2,5-diphenyloxazole and 2-tetralone. Reaction of substrate **18** with CF_3SO_3H gave the phenethyl-substituted product **19** in good yield. Initial ionization gives the 1,3-dication (**20**) and charge migration provides the 1,4-dication (**21**). Previous studies have shown that such isomerizations occur by deprotonation–reprotonation steps,[4a] rather than by direct hydride shift. The charge migration is driven by charge–charge repulsive effects and formation of the stabilized benzylic carbocation. Ring-closure and ring-opening steps then lead to product **19**. Thus, the entire transformation occurs in 11 reaction steps (protonation–deprotonation steps included). Interestingly, compound **22** provides the expected product **23** but a rearranged product (**24**) is also formed in an almost equal amount. The scrambling of the methyl and phenethyl groups is somewhat unexpected but it may occur by arene protonation–alkyl shift–deprotonation mechanisms.[9] This may also account for product **24**.



Scheme 1.

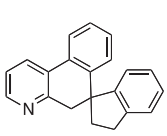




Scheme 2.

Although compound **9** does provide the desired phenethyl-substituted arene (**14**), we have observed that ring-opening reactions with five-membered rings are very sluggish. Compound **9**

produces the spirocyclic intermediate (**25**) quantitatively but formation of the final product (**14**) requires an extended reaction period. When compound **26** is reacted in CF_3SO_3H, product **27** is formed but no ring-opened product could be isolated (even with extended forcing conditions).



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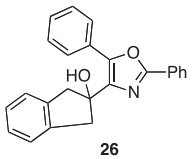

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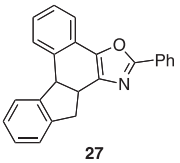

27

3. Conclusion

In summary, we have observed the formation of functionalized aza-polycyclic aromatic compounds from cyclization/ring-opening reaction cascades. These reactions produce arenes with phenethyl and 3-(aryl)propyl groups. The proposed mechanism involves a series of dicationic intermediates.

4. Experimental section

4.1. Materials and methods

Unless otherwise indicated, all reagents and chemical were obtained from commercial suppliers. The trifluoromethanesulfonic acid was distilled from an argon atmosphere prior to its use. All reactions were done in oven-dried glassware with an inert atmosphere (Ar). High-resolution mass spectral analyses were done by an off-site analytical laboratory, while low-resolution mass spectra were obtained directly from a gas chromatography instrument equipped with a mass-selective detector.

4.2. General synthetic method to prepare alcohol substrates (6–10, 20, 22)

2-Methyl-3-phenylpyridine or 2,5-diphenyloxazole (2.3 mmol) is dissolved in anhydrous Et_2O (10 mL) and the solution is cooled to −78 °C. *n*-BuLi solution (2.5 mmol) is then added and the mixture is stirred for 2 h. The ketone substrate (2.5 mmol, dissolved in 10 mL of Et_2O) is added and the mixture is stirred 16 h, during which time the solution warms to 25 °C. The mixture is then quenched with H_2O, diluted with Et_2O, washed with brine, and dried over Na_2SO_4. Concentration in vacuo provides a product, which is further purified by flash chromatography (hexane/ether).

*4.2.1. 1-((3-Phenylpyridin-2-yl)methyl)-1,2,3,4-tetrahydronaphthalen-1-ol (**6**).* ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm): 1.29–1.38 (m, 1H), 1.67–1.83 (m, 3H), 2.5–2.6 (m, 1H), 2.7–2.8 (m, 1H), 3.19 (dd, *J*=37, 15 Hz, 2H), 6.98–7.04 (m, 3H), 7.11–7.14 (m, 2H), 7.28–7.36 (m, 4H), 7.46–7.49 (m, 1H), 7.57 (dd, *J*=7.7, 1.8 Hz, 1H), 8.6 (dd, *J*=4.9, 1.8 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm): 20.0, 29.3, 36.3, 43.8, 73.3, 121.4, 125.9, 126.7, 127.0, 127.6, 128.39, 128.44, 129.0, 136.4, 138.2, 138.5, 138.9, 142.7, 147.1, 157.2. Low-resolution mass spectrum (EI): 297 (M−17), 296, 168, 167, 128. High-resolution mass spectrum (EI), calcd for $C_{22}H_{19}N$ (M−18) 297.15175, found 297.15028.

*4.2.2. 1-((3-(2,3-Dihydrobenzo[b][1,4]dioxin-6-yl)pyridin-2-yl)methyl)-1,2,3,4-tetrahydronaphthalen-1-ol (**7**).* ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm): 1.20–1.52 (m, 1H), 1.67–1.82 (m, 3H), 2.56–2.84 (m, 2H), 3.21 (dd, *J*=31.2, 14.9 Hz, 2H), 4.27 (s, 4H), 6.50 (dd, *J*=8.2, 2.07 Hz, 1H), 6.58 (d, *J*=2.04 Hz, 1H), 6.83 (d, *J*=21.6 Hz, 1H), 7.01–7.04 (m, 1H), 7.11–7.20 (m, 2H), 7.23–7.27 (m, 1H), 7.41 (br s, 1H), 7.51–7.55 (m, 2H), 8.56 (dd, *J*=4.8, 1.78 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm): 20.0, 29.4, 36.2, 43.7, 64.3, 73.3, 117.2, 117.9, 121.3, 122.1, 126.0, 126.7, 127.1, 128.5, 132.2, 136.5, 138.0, 138.2, 142.7, 143.24, 143.26, 146.8, 157.4.

*4.2.3. 6-Methoxy-1-((3-phenylpyridin-2-yl)methyl)-1,2,3,4-tetrahydronaphthalen-1-ol (**8**).* ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm): 1.28–1.36 (m, 1H), 1.64–1.79 (m, 3H), 2.49–2.78 (m, 2H), 3.18 (dd, *J*=40.1, 14.9 Hz, 2H), 3.76 (s, 3H), 6.52 (s, 1H), 6.72 (dd, *J*=8.6, 2.5 Hz, 1H), 7.05–7.08 (m, 2H), 7.27–7.42 (m, 6H), 7.57 (dd, *J*=7.7, 1.7 Hz, 1H), 8.52–8.60 (m, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm): 20.1, 29.7, 36.4, 44.0, 55.2, 73.0, 112.2, 112.7, 121.4, 127.6, 128.3, 128.4, 129.0, 135.3, 137.9, 138.2, 138.5, 139.0, 147.1, 157.2, 158.2. Low-resolution mass spectrum (EI): 327 (M−18), 326, 312, 282, 168. High-resolution mass spectrum (EI), calcd for $C_{23}H_{21}NO$ (M−18) 327.16232, found 327.16115.

*4.2.4. 1-((3-Phenylpyridin-2-yl)methyl)-2,3-dihydro-1H-inden-1-ol (**9**).* ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm): 2.01–2.22 (m, 2H), 2.47–2.57 (m, 1H), 2.91 (ddd, *J*=15.9, 8.40, 2.91 Hz, 1H), 3.1 (dd, *J*=39.3, 14.7 Hz, 2H), 6.75–6.78 (m, 2H), 6.84 (d, *J*=7.5 Hz, 1H), 7.04–7.10 (m, 1H), 7.17–7.19 (m, 2H), 7.24–7.32 (m, 4H), 7.35 (br s, 1H), 7.74 (dd, *J*=7.7, 1.8 Hz, 1H), 8.61 (dd, *J*=4.9, 1.8 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm): 29.0, 40.7, 41.8, 83.4, 121.5, 123.1, 124.7, 126.3, 127.5, 127.7, 128.3, 128.8, 138.0, 138.3, 138.8, 142.2, 147.0, 147.8, 157.1. Low-resolution mass spectrum (EI): 283 (M−18), 282, 267, 167, 154. High-resolution mass spectrum (EI), calcd for $C_{21}H_{17}N$ (M−18) 283.13610, found 283.13673.

*4.2.5. 1-((3-Phenylpyridin-2-yl)methyl)-dibenzosuberol (**10**).* ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm): 2.58–2.82 (m, 4H), 3.74 (s, 2H), 6.84–6.87 (m, 2H), 6.96–6.99 (m, 2H), 7.09–7.18(m, 5H), 7.34–7.43 (m, 4H), 7.96–7.99 (m, 2H), 8.54 (dd, *J*=4.8, 1.7 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm): 34.4, 46.4, 78.8, 121.5, 125.9, 126.9, 127.7, 128.0, 128.2, 129.5, 130.1, 137.9, 138.2, 138.6, 138.8, 144.1, 146.6, 156.2. Low-resolution mass spectrum (EI): 359 (M−18), 358, 208, 180, 169, 168. High-resolution mass spectrum (EI), calcd for $C_{27}H_{21}N$ (M−18) 359.16740, found 359.16550.

4.3. General synthetic method to prepare functionalized arenes (11–15, 19, 23, 24, 27)

The alcohol precursor (2 mmol) is dissolved in 2 mL of $CHCl_3$, and 4 mL of CF_3SO_3H is added. After stirring overnight, the mixture is poured over ice and the solution is made basic with 10 M NaOH. The solution is then extracted with $CHCl_3$ (3×30 mL) and the combined organic extracts are washed with H_2O (1×) and brine (2×). The resulting solution is then dried with anhydrous Na_2SO_4. If necessary, the product is purified by chromatography (hexane/ether).

*4.3.1. 6-(3-Phenylpropyl)benzo[f]quinolone (**11**).* ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm): 2.00–2.11 (m, 2H), 2.72 (t, *J*=7.5 Hz, 2H), 3.09 (t, *J*=15.7 Hz, 2H), 7.10–7.23 (m, 5H), 7.64–7.69 (m, 3H), 7.93–7.97 (m, 1H), 8.11 (s, 1H), 8.55–8.58 (m, 1H), 8.84 (dd, *J*=5.0, 1.3 Hz, 1H), 9.13 (d, *J*=8.3 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm): 31.5, 33.0, 35.8, 120.9, 121.5, 123.3, 125.1, 125.9, 126.0, 128.2, 128.5, 128.5, 128.7, 128.8, 131.1, 135.5, 141.5, 143.0, 144.1, 146.3. Low-resolution mass spectrum (EI): 297 (M^+), 204, 193, 91. High-resolution mass spectrum (EI), $C_{22}H_{19}N$ calcd 297.15175, found 297.15220.

*4.3.2. 6-(3-Phenylpropyl)-2,3-dihydrobenzo[b][1,4]dioxin[2,3-f]quinoline (**12**).* ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm): 2.06 (m, 2H), 2.76 (t, *J*=7.4 Hz, 2H), 2.99 (t, *J*=7.5 Hz, 2H), 4.41 (s, 4H), 7.16–7.30 (m, 5H), 7.38 (s, 1H), 7.97 (d, *J*=8.37 Hz, 3H), 8.89 (s, 1H), 9.23 (d, *J*=7.5 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm): 31.1, 33.3, 35.7, 64.6, 110.6,

112.0, 115.1, 121.0, 123.0, 126.1, 126.3, 126.6, 128.4, 128.4, 138.8, 139.1, 139.9, 141.2, 146.0, 146.3, 149.0. Low-resolution mass spectrum (EI): 355 (M^+), 340, 326, 270, 254. High-resolution mass spectrum (EI), $C_{24}H_{21}NO_2$ calcd 355.15723, found 355.15859.

*4.3.3. 6-(3-(3-Methoxyphenyl)propyl)benzo[f]quinolone (**13**).* ^{1}H NMR (300 MHz, $CDCl_3$) δ (ppm): 2.00–2.11 (m, 2H), 2.72 (t, J=7.44 Hz, 2H), 3.01 (t, J=7.5 Hz, 2H), 3.72 (s, 3H), 6.66–6.70 (m, 2H), 6.76 (d, J=7.6 Hz, 1H), 7.15 (t, J=7.8 Hz, 1H), 7.68–7.79 (m, 2H), 7.91 (dd, J=8.4, 5.3 Hz, 1H), 7.96–7.99(m, 1H), 8.18(s, 1H), 8.62–8.66(m, 1H), 9.01 (dd, J=223.0, 8.78 Hz, 1H), 8.93 (dd, J=5.2, 1 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm): 31.3, 33.0, 35.8, 55.1, 111.4, 114.1, 118.7, 120.9, 121.3, 123.6, 125.2, 126.4, 128.0, 128.9, 129.4, 129.5, 130.9, 138.1, 140.6, 142.0, 142.9, 148.2, 159.6. Low-resolution mass spectrum (EI): 327 (M^+), 326, 298, 206, 193, 122. High-resolution mass spectrum (EI), $C_{23}H_{21}NO$ calcd 327.16232, found 327.16126.

*4.3.4. 6-Phenethylbenzo[f]quinoline (**14**).* ^{1}H NMR (300 MHz, CDCl) δ (ppm): 3.16–3.22 (m, 2H), 3.46–3.52 (m, 2H), 7.21–7.29 (m, 1H), 7.31–7.35 (m, 4H), 7.52–7.56 (m, 1H), 7.71–7.75 (m, 2H), 7.9 (s, 1H), 8.20–8.24 (m, 1H), 8.69–8.72 (m, 1H), 8.93–8.96 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm): 35.3, 36.2, 120.9, 123.2, 124.4, 124.7, 126.2, 126.8, 127.2, 127.4, 128.4, 128.5, 130.1, 130.4, 130.9, 140.5, 141.7, 148.1, 149.8. Low-resolution mass spectrum (EI): 283 (M^+), 192, 191, 166, 91. High-resolution mass spectrum (EI), $C_{21}H_{17}N$ calcd 283.13610, found 283.13524.

*4.3.5. 6-(2-Phenethylphenyl)benzo[f]quinoline (**15**).* ^{1}H NMR (300 MHz, CDCl) δ (ppm): 2.67–2.85 (m, 4H), 6.82–6.85 (m, 2H), 7.08–7.11 (m, 3H), 7.35–7.49 (m, 4H), 7.54–7.63 (m, 3H), 7.69–7.75 (m, 1H), 7.75 (s, 1H), 8.73 (d, J=8.3 Hz, 1H), 9.01–9.05 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm): 35.5, 37.5, 121.4, 122.9, 125.2, 125.8, 126.1, 127.1, 127.3, 127.4, 128.2, 128.2, 128.3, 128.7, 129.3, 129.8, 130.5, 130.6, 131.6, 139.2, 140.3, 141.6, 142.1, 147.7, 150.1. Low-resolution mass spectrum (EI): 359 (M^+), 268, 267, 254, 91. High-resolution mass spectrum (EI), $C_{27}H_{21}N$ calcd 359.16740, found 359.16616.

*4.3.6. 2-Phenyl-4-(2-phenylethyl)naphtho[2,1-d]oxazole (**19**).* Yellow solid, mp: 145–147 °C. ^{1}H NMR ($CDCl_3$, 500 MHz) δ (ppm): 3.27–3.20 (t, J=8.1 Hz, 2H), 3.49–3.52 (t, J=8.1 Hz, 2H), 7.24–7.26 (m, 1H), 7.32–7.36 (m, 4H), 7.52–7.63 (m, 6H), 7.91 (d, J=8.1 Hz, 1H), 8.30 (d, J=8.1 Hz, 1H), 8.40–8.41 (m, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ (ppm): 33.6, 36.3, 119.3, 120.1, 123.4, 125.6, 126.0, 127.4, 127.8, 128.2, 128.4, 128.6, 128.9, 131.0, 131.8, 132.8, 138.7, 142.1, 143.6, 146.3, 162.0. Low-resolution mass spectrum (EI): 349 (M^+), 259, 258, 127, 91. High-resolution mass spectrum (EI), $C_{25}H_{19}NO$ calcd 349.14667, found 349.14608.

*4.3.7. 5-Methyl-2-phenyl-4-(2-phenylethyl)naphtho[2,1-d]oxazole (**23**).* Yellow solid, mp: 165–167 °C. ^{1}H NMR ($CDCl_3$, 500 MHz) δ (ppm): 2.65 (s, 3H), 3.09–3.12 (m, 2H), 3.58–3.61 (m, 2H), 7.26–7.27 (m, 1H), 7.33–7.37 (m, 4H), 7.57–7.64 (m, 5H), 8.14 (d, J=8.4 Hz, 1H), 8.34 (d, J=8.7 Hz, 1H), 8.39–8.41 (m, 2H). NOE enhancement observed between δ 2.65 (CH_3) and δ 8.14 (CH). ^{13}C NMR ($CDCl_3$, 125 MHz) δ (ppm): 14.4, 30.8, 36.6, 119.5, 120.6, 125.2, 125.5, 125.6, 125.9, 127.4, 128.3, 128.6, 128.8, 130.4, 130.8, 131.3, 138.7, 142.2, 144.9, 161.7. Low-resolution mass spectrum (EI): 363 (M^+), 272, 141, 115, 91. High-resolution mass spectrum (EI), $C_{26}H_{21}NO$ calcd 363.16232, found 363.16305.

*4.3.8. 4-Methyl-2-phenyl-5-(2-phenylethyl)naphtho[2,1-d]oxazole (**24**).* Yellow solid, mp: 126–128 °C. ^{1}H NMR ($CDCl_3$, 500 MHz) δ (ppm): 2.76 (s, 3H), 3.00–3.03 (m, 2H), 3.47–3.50 (m, 2H), 7.31–7.35 (m, 3H), 7.38–7.41 (m, 2H), 7.58–7.64 (m, 5H), 8.22–8.23 (m, 1H), 8.34–8.36 (m, 1H), 8.38–8.40 (m, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ (ppm): 13.8, 30.9, 36.5, 119.6, 120.9, 124.6, 125.4, 125.7, 126.2, 126.8, 127.3, 127.8, 128.4, 128.6, 128.9, 130.3, 130.9, 131.9, 139.1, 142.0, 143.6, 144.8, 161.8. Low-resolution mass spectrum (EI): 363 (M^+), 272, 141, 115, 91. High-resolution mass spectrum (EI), $C_{26}H_{21}NO$ calcd 363.16232, found 363.16340.

*4.3.9. 2′,3′-Dihydro-5H-spiro[benzo[f]quinoline-6,1′-indene] (**25**).* ^{1}H NMR (300 MHz, CDCl) δ (ppm): 1.93–2.27 (m, 2H), 2.85–3.07 (m, 2H), 3.36 (dd, J=122.5, 15.6 Hz, 2H), 6.81 (dd, J=7.7, 1.0 Hz, 1H), 7.19–7.37 (m, 7H), 7.81 (d, J=7.0 Hz, 1H), 8.08 (dd, J=7.9, 2.3 Hz, 1H), 8.50 (dd, J=4.8, 1.4 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ (ppm): 29.9, 39.3, 43.9, 52.7, 122.5, 124.1, 124.3, 124.8, 126.6, 126.8, 127.1, 127.4, 128.7, 129.7, 130.5, 132.0, 143.7, 144.7, 147.8, 148.1, 156.1. Low-resolution mass spectrum (EI): 283 (M^+), 282, 268, 254, 154. High-resolution mass spectrum (EI), $C_{21}H_{17}N$ calcd 283.13610, found 283.13579.

*4.3.10. Compound **27**.* Yellow solid, mp: 225–227 °C. ^{1}H NMR ($CDCl_3$, 500 MHz) δ (ppm): 3.51–3.56(q, J_1=15.7 Hz, J_2=7.2 Hz, 1H), 3.78 (d, J=15.7 Hz, 1H), 4.15–4.18 (t, J=7.4 Hz, 1H), 4.62 (d, J=7.5 Hz, 1H), 6.94 (d, J=7.5 Hz, 1H), 7.09–7.12 (t, J=7.5 Hz, 1H), 7.16–7.19 (t, J=7.3 Hz, 1H), 7.29–7.51 (m, 7H), 7.57–7.58 (m, 1H), 8.08–8.10 (m, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz) δ (ppm): 120.4, 123.9, 124.7, 125.1, 126.3, 126.4, 127.2, 127.3, 127.5, 128.7, 129.4, 130.2, 135.0, 138.1, 141.6, 144.0, 145.7, 161.0. Low-resolution mass spectrum (EI): 355 (M^+), 231, 202, 166. High-resolution mass spectrum (EI), $C_{24}H_{17}NO$ calcd 335.13102, found 335.13152.

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Supplementary data

Characterization data for compounds **6**–**10**, ^{1}H and ^{13}C NMR spectra for compounds **11**–**15**, **23**–**25**, and **27**. Supplementary data associated with this article can be found in the online version, at doi:10.1016/j.tet.2012.02.047.

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