A Convenient Domino Synthesis of 4,9-Diphenyl-2,3-dihydro-1*H*-benzo[*f*]isoindole Derivatives

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Received 26 May 2010; revised 21 June 2010

Abstract: The first examples are described of a convenient domino synthesis of 4,9-diphenyl-2,3-dihydro-1*H*-benzo[*f*]isoindole derivatives in a single operation by means of palladium-catalyzed cyclization of simple 1,6-diynes with aryl halides.

Key words: domino reactions, cyclizations, heterocycles, polycycles, isoindoles

Condensed heterocyclic compounds are playing increasingly important roles as synthetic building blocks, pharmacophores, and electroluminescent materials.[1] The intramolecular palladium-catalyzed hydroarylation of alkynes was reported by Fujiwara in 2000.[2] Soon after, other transition-metal- and Lewis acid catalyzed domino versions of this transformation emerged, and the method quickly became a powerful tool for the construction of indoles and other heterocycles.[3] These processes are usually clean, because they generate less waste by minimizing the need for isolation of intermediates in multistep syntheses of complex molecular targets.[4] The design of new catalytic and regioselective syntheses of π-conjugated fused rings is therefore a continuing challenge that lies at the forefront of synthetic chemistry.[5] Recently, an effective rhodium-catalyzed asymmetric cyclopentannulation has been developed that leads to a formal synthesis of indolyl rings,[6] and a gold(I)-catalyzed 5-endo-dig cyclization has been used in an enantioselective synthesis of cephalostatin 1.[7] A promising route has been developed for activation of C–H bonds in the preparation of indolines and tetrahydroisoquinolines.[8] The palladium-catalyzed, chemoselective, intramolecular, sp^3 C–H activation of the methyl group in 2-bromo-*N*-(2-methylaryl)-pyrroles can afford 9*H*-pyrrolo[1,2-*a*]indoles;[9] these molecules have interesting biological and pharmaceutical properties, and much effort has been made toward their synthesis.[10] The intramolecular nature of these transformations means that the regio- and stereoselectivities are often excellent, permitting the single compound to be obtained after several bond-forming reactions.

Here, we report the first examples of convenient palladium-catalyzed domino reactions[11] of 4-methyl-*N*,*N*-bis(3-phenylprop-2-yn-1-yl)benzenesulfonamide (**1**), *N*,*N*-bis(3-phenylprop-2-yn-1-yl)benzenesulfonamide (**2**), 4-methyl-*N*,*N*-bis[3-(4-methylphenyl)prop-2-yn-1-yl]benzenesulfonamide (**3**), *N*,*N*-bis[3-(4-chlorophenyl)prop-2-yn-1-yl]-4-methylbenzenesulfonamide (**4**), and *N*,*N*-bis[3-(4-methoxyphenyl)prop-2-yn-1-yl]-4-methylbenzenesulfonamide (**5**) with various aryl halides, which provide a direct, efficient, and economic method for the construction of polycyclic heterocycles through both C–C bond coupling and C–H bond activation processes.

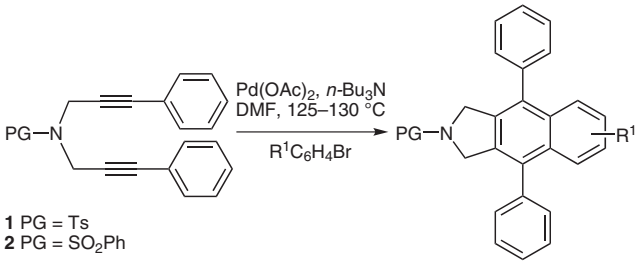
In a typical procedure, a mixture of 4-methyl-*N*,*N*-bis(3-phenylprop-2-yn-1-yl)benzenesulfonamide (**a**), ethyl 4-bromobenzoate, tributylamine, and palladium(II) acetate was heated in *N*,*N*-dimethylformamide under argon overnight. Standard workup procedures afforded the coupled products in excellent regioselectivities for most 1,6-diynes (Table 1). The temperature was crucial for this reaction, and no domino reaction occurred at below 95 °C. In contrast, higher reaction temperatures (above 130 °C) led to the decomposition of the product **1a** (Table 1), as indicated by thin-layer chromatography. Among the catalysts tested [chitosan-Pd, tetrakis(triphenylphosphine)palladium, palladium(II) chloride/triphenylphosphine, palladium(II) acetate/triphenylphosphine, and bis(dibenzylideneacetone)palladium/triphenylphosphine], palladium(II) acetate/triphenylphosphine was the most effective. *N*,*N*-Dimethylformamide was found to be a better solvent than *N*,*N*-dimethylacetamide, toluene, or dioxane for this reaction. Tributylamine was more effective than any of inorganic base tested (potassium or cesium carbonate). The following standard reaction conditions were therefore selected for subsequent studies. The diyne (1 equiv) was treated with the aryl halide (1.2 equiv) in the presence of palladium(II) acetate (2 mol%), triphenylphosphine (4 mol%), and tributylamine (2 equiv) in *N*,*N*-dimethylformamide at 125 °C.

Illustrative examples of the scope of the reaction are shown in Table 1. Various diynes and substituted aryl halides are compatible with this palladium-catalyzed domino reaction. A range of 4,9-diphenyl-2,3-dihydro-1*H*-benzo[*f*]isoindole derivatives were readily isolated in good-to-excellent yields, except in the case of **1i**, when aryl halides with a variety of substituents was used. Suitable substituents included ethoxycarbonyl, methoxycarbonyl, chloro, sulfonyl, oxo, naphthyl, cyano, or formacyl groups. When substrate **1** or **2** was treated with an aryl halide, such as ethyl 4-bromobenzoate, methyl 4-bromobenzoate, or 1-bromonaphthalene, the reaction afforded 3,3-

SYNTHESIS 2010, No. 20, pp 3467–3473
Advanced online publication: 30.07.2010
DOI: 10.1055/s-0030-1257908; Art ID: F09310SS

disubstituted 4,9-diphenyl-2,3-dihydro-1*H*-benzo[*f*]isoindoles **1a**, **1e**, **1h**, and **2c**, respectively, in yields of more than 84%. The yield of compound **1e** was the highest (87%; entry 5). Yields from substrate **1** were close to those of substrate **2**. These results demonstrate the application of bromo compounds for the direct C–H functionalization of molecular scaffolds in the synthesis of the benzo[*f*]isoindoline core.

Table 1 Palladium-Catalyzed One-Pot Cascade Reaction

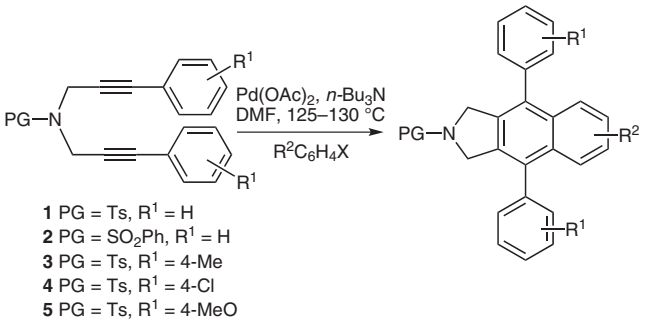


Entry	Substrate[a]	$R^2C_6H_4Br$	Product	Yield (%)[b]
1	**1**	4-$EtO_2CC_6H_4Br$	**1a**	85
2	**1**	4-AcC_6H_4Br	**1b**	73
3	**1**	4-NCC_6H_4Br	**1c**	60
4	**1**	4-ClC_6H_4Br	**1d**	62
5	**1**	1-bromonaphthalene	**1e**	87
6	**1**	4-$MeO_2SC_6H_4Br$	**1f**	82
7	**1**	4-$OHCC_6H_4Br$	**1g**	62
8	**1**	4-$MeO_2CC_6H_4Br$	**1h**	84
9	**1**	4-MeC_6H_4Br	**1i**	45
10	**2**	4-$EtO_2CC_6H_4Br$	**2a**	83
11	**2**	4-AcC_6H_4Br	**2b**	74
12	**2**	1-bromonaphthalene	**2c**	85
13	**2**	4-$OHCC_6H_4Br$	**2d**	60
14	**2**	4-$MeO_2CC_6H_4Br$	**2e**	82

[a] General conditions: **1** or **2** (1.0 equiv), ArBr (1.2 equiv), $Pd(OAc)_2$ (2 mol%), Ph_3P (4 mol%), Bu_3N (2 equiv), DMF (10 mL), 125–130 °C.
[b] Isolated yield after flash column chromatography.

To examine the effect of the diyne on the C–H functionalization, and to study the generality of the method in preparing more-highly functionalized benzo[f]isoindoline derivatives, the reactions of various 1,6-diynes (**3**, **4**, and **5**) with various aryl halides were studied (Table 2). The results indicate that various benzo[*f*]isoindoline could be constructed in a one-pot process in good yields by reaction of the corresponding diynes with aryl halides (Table 2, entries 1–8). However, the reaction of substrates having strongly electron-donating methoxy groups (**5**) with 1-bromonaphthalene gave the corresponding tetracyclic compound **5a** (58%) in a relatively low yield (entry 8), showing the existence of electronic effects on the domino reaction. The reaction of substrate **2** with aryl iodides gave much lower yields than did aryl bromides (entries 9–11). Interestingly, when aryl halides containing C–Cl or C–F bonds on the benzene ring in addition to C–Br reacted, the C–Br bond coupled selectively with the 1,6-diyne (entries 6, 10–12).

Table 2 The Palladium-Catalyzed Reaction for the Formation of π-Conjugated Fused Heterocycles



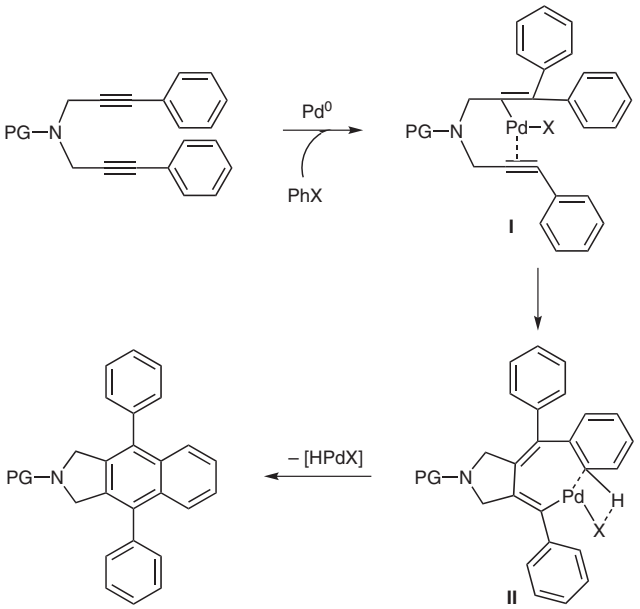
Entry	Substrate[a]	$R^2C_6H_4X$	Product	Yield (%)[b]
1	**3**	4-$EtO_2CC_6H_4Br$	**3a**	75
2	**3**	1-bromonaphthalene	**3b**	76
3	**3**	4-$MeO_2CC_6H_4Br$	**3c**	69
4	**4**	4-$EtO_2CC_6H_4Br$	**4a**	80
5	**4**	1-bromonaphthalene	**4b**	79
6	**4**	4-ClC_6H_4Br	**4c**	70
7	**4**	4-$MeO_2CC_6H_4Br$	**4d**	72
8	**5**	1-bromonaphthalene	**5a**	58
9	**1**	PhI	**1j**	55
10	**1**	4-FC_6H_4I	**1k**	56
11	**2**	4-FC_6H_4I	**2f**	57
12	**2**	4-FC_6H_4Br	**2f**	69

[a] General conditions: **1**–**5** (1.0 equiv), ArX (X = Br; I) (1.2 equiv), $Pd(OAc)_2$ (2 mol%), Ph_3P (4 mol%), nBu_3N (2 equiv), DMF 10 mL, 125–130 °C.
[b] Isolated yield after flash column chromatography.

All the new benzo[*f*]isoindoline polycycles were fully characterized by various spectroscopic techniques (^1H NMR, ^{13}C NMR, UV, and IR), and by high-resolution mass spectrometry.

On the basis of the above observations, a plausible mechanism is proposed for the selective formation polycyclic heterocycles (Scheme 1). Coordination and insertion of the arylpalladium(II) halide in the diyne substrate produces the intermediate **I** ('Heck-type' product), which then reacts with the C=C double bond through a carbopalladation reaction and σ-bond metathesis onto the aryl

group via the intermediate **II**. This is followed by proton abstraction[12] by the base to give the benzo[*f*]isoindoline polycyclic product.



Scheme 1 Proposed mechanism for the domino reaction

To summarize, we have developed an efficient method for the synthesis of 3,3-disubstituted 4,9-diphenyl-2,3-dihydro-1*H*-benzo[*f*]isoindoline derivatives in good yields and excellent regioselectivities under mild conditions by cyclization of a diyne with an aryl halides in the presence of a palladium(II) acetate based catalytic system. This method provides direct, easy, and economic construction of fused π-conjugated polycyclic heterocycles through multistep C–C bond formation and C–H activation of the benzene ring. In addition to the synthetic utility of the reaction, its ability to combine direct arylation into this class of tandem processes raises confidence in the viability of other domino processes involving alkenylpalladium(II) reactive intermediates. Further investigation to understand this catalytic transformation, evaluation of the process with a broader scope of substrates, synthesis of more-complex π-system heterocycles, and the development of functional organic materials are in progress.

All melting points were determined on a Gallenkamp melting-point apparatus and are uncorrected. IR spectra were recorded on a Nicolet 5DX spectrophotometer. The ^{1}H NMR spectra were recorded in $CDCl_3$ using TMS as an internal reference on a Bruker Avance 300 NMR spectrometer operating at 300 MHz. The ^{13}C NMR spectra were determined using TMS as an internal reference with a Bruker Avance 300 spectrometer operating at 75.5 MHz. HRMS spectra were recorded on an Agilent 6200 LC/MS TOF spectrometer in EI mode (70 eV). Flash column chromatography was performed on silica gel (230–240 mesh). All the organic solvents were dried over appropriate drying agents and distilled before use. All the chemicals used in this study were sourced commercially.

Ethyl 4,9-Diphenyl-2-tosyl-2,3-dihydro-1*H*-benzo[*f*]isoindole-6-carboxylate (1a); Typical Procedure

4-Methyl-*N*,*N*-bis(3-phenylprop-2-yn-1-yl)benzenesulfonamide (**1**; 1.0 equiv), 4-Br$C_6H_4CO_2$Et (1.2 equiv), $Pd(OAc)_2$ (2 mol%), and Ph_3P (4 mol%) were added to the degassed solution of Bu_3N (2 equiv) in DMF (5 mL). The mixture was stirred at rt for 30 min, heated at 125 °C for 20 h, and then cooled. The reaction was quenched with H_2O, and the mixture was extracted with EtOAc (20 mL). The combined organic layers were washed sequentially with 5% aq HCl (5 mL), 5% aq Na_2CO_3 (5 mL), and brine (3 × 5 mL), then dried ($MgSO_4$), and concentrated. The residue was subjected to flash chromatography [silica gel, PE–EtOAc (6:1)] to give a white solid; mp 250–251 °C; R_f = 0.48 (PE–EtOAc, 6:1).

FT-IR (KBr): 2978, 1716, 1344, 1287, 1255, 1096, 606 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.39 (s, 1 H), 7.92 (d, *J* = 8.5 Hz, 1 H), 7.70–7.67 (m, 3 H), 7.63–7.45 (m, 6 H), 7.31–7.29 (m, 6 H), 4.56 (s, 4 H), 4.32 (q, *J* = 6.9 Hz, 2 H), 2.40 (s, 3 H), 1.32 (t, *J* = 6.9 Hz, 3 H).

^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 166.6, 143.7, 137.1, 136.8, 135.4, 134.4, 133.8, 131.6, 129.8, 129.3, 128.9, 128.8, 128.1, 127.8, 127.6, 126.1, 125.2, 61.1, 53.6, 53.5, 21.5, 14.2.

HRMS (EI): *m/z* calcd for $C_{34}H_{29}NO_4S$: 547.1817; found: 547.1887.

UV/Vis (MeCN): λ_{max} = 252 nm.

1-(4,9-Diphenyl-2-tosyl-2,3-dihydro-1*H*-benzo[*f*]isoindol-6-yl)ethanone (1b)

White solid; mp 245–246 °C; R_f = 0.46 (PE–EtOAc, 6:1).

FT-IR (KBr): 2859, 1686, 1599, 1344, 1240, 1157, 1096, 706, 596 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.24 (s, 1 H), 7.89 (d, *J* = 8.5 Hz, 1 H), 7.70–7.68 (m, 4 H), 7.59–7.50 (m, 5 H), 7.32–7.26 (m, 6 H), 4.56 (s, 4 H), 2.49 (s, 3 H), 2.39 (s, 3 H).

^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 198.0, 143.8, 137.0, 136.7, 135.7, 135.3, 134.5, 134.4, 134.0, 133.9, 131.7, 129.8, 129.4, 129.0, 128.4, 128.2, 128.0, 127.6, 126.5, 123.9, 53.6, 53.5, 26.5, 21.5.

HRMS (EI): *m/z* calcd for $C_{33}H_{27}NO_3S$: 517.1712; found: 517.1779.

UV/Vis (MeCN): λ_{max} = 265 nm.

4,9-Diphenyl-2-tosyl-2,3-dihydro-1*H*-benzo[*f*]isoindole-6-carbonitrile (1c)

White solid; mp 258–259 °C; R_f = 0.46 (PE–EtOAc, 6:1).

FT-IR (KBr): 2860, 2226, 1599, 1491, 1344, 1157, 1096, 667 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.99 (s, 1 H), 7.79 (d, *J* = 7.3 Hz, 2 H), 7.71–7.68 (m, 5 H), 7.49–7.46 (m, 4 H), 7.29–7.27 (m, 5 H), 4.56 (s, 4 H), 2.41 (s, 3 H).

^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 143.5, 136.4, 132.8, 132.7, 132.2, 131.5, 129.8, 129.2, 129.1, 128.9, 128.7, 128.6, 128.5, 127.9, 127.6, 127.2, 126.3, 119.1, 118.3, 112.4, 109.4, 53.5, 53.4, 21.5.

HRMS (EI): *m/z* calcd for $C_{32}H_{24}N_2O_2S$: 500.1558; found: 500.1619.

UV/Vis (MeCN): λ_{max} = 250 nm.

6-Chloro-4,9-diphenyl-2-tosyl-2,3-dihydro-1*H*-benzo[*f*]isoindole (1d)

White solid; mp 235–236 °C; R_f = 0.46 (PE–EtOAc, 6:1).

FT-IR (KBr): 2858, 1599, 1494, 1344, 1157, 1096, 605 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.77 (d, *J* = 7.7 Hz, 2 H), 7.54–7.29 (m, 6 H), 7.59–7.43 (m, 3 H), 7.32–7.28 (m, 6 H), 4.53 (s, 4 H), 2.40 (s, 3 H).

^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 143.7, 137.1, 136.9, 134.2, 133.2, 132.2, 130.6, 129.8, 129.3, 129.0, 128.9, 128.8, 128.1, 127.9, 127.6, 126.7, 125.9, 125.5, 124.7, 120.7, 54.3, 53.8, 53.6, 53.1, 21.5.

HRMS (EI): *m/z* calcd for $C_{31}H_{24}ClNO_2S$: 509.1216; found: 509.1275.

UV/Vis (MeCN): λ_{max} = 248 nm.

7,11-Diphenyl-9-tosyl-9,10-dihydro-8*H*-naphtho[1,2-*f*]isoindole (1e)

White solid; mp 268–269 °C; R_f = 0.46 (PE–EtOAc, 6:1).

FT-IR (KBr): 3053, 2846, 1597, 1493, 1443, 1348, 1159, 1093, 705, 699 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.77 (d, *J* = 7.6 Hz, 1 H), 7.69–7.66 (m, 3 H), 7.64–7.38 (m, 6 H), 7.31–7.26 (m, 2 H), 7.31–7.26 (m, 6 H), 7.09–7.04 (m, 2 H), 4.58 (s, 2 H), 4.51 (s, 2 H), 2.40 (s, 3 H).

^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 143.6, 142.1, 138.3, 135.4, 133.2, 133.1, 131.9, 129.9, 129.8, 129.5, 128.9, 128.3, 127.9, 127.8, 127.6, 127.5, 126.2, 125.3, 124.5, 54.7, 54.1, 21.5.

HRMS (EI): *m/z* calcd for $C_{35}H_{27}NO_2S$: 525.1762; found: 525.1819.

UV/Vis (MeCN): λ_{max} = 271 nm.

6-Mesyl-4,9-diphenyl-2-tosyl-2,3-dihydro-1*H*-benzo[*f*]isoindole (1f)

White solid; mp 279–280 °C; R_f = 0.46 (PE–EtOAc, 6:1).

FT-IR (KBr): 3055, 2926, 1599, 1495, 1342, 1038, 1157, 756, 705 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.29 (s, 1 H), 7.80–7.78 (m, 2 H), 7.70–7.67 (m, 2 H), 7.57–7.53 (m, 4 H), 7.36–7.30 (m, 8 H), 4.57 (s, 4 H), 3.01 (s, 3 H), 2.41 (s, 3 H).

^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 143.9, 137.6, 136.7, 136.5, 135.2, 134.4, 131.5, 129.9, 129.3, 129.1, 128.9, 128.7, 127.8, 127.6, 126.8, 126.3, 122.2, 53.4, 53.5, 44.6, 44.3, 21.5.

HRMS (EI): *m/z* calcd for $C_{32}H_{27}NO_4S_2$: 553.1381; found: 553.1466.

UV/Vis (MeCN): λ_{max} = 249 nm.

4,9-Diphenyl-2-tosyl-2,3-dihydro-1*H*-benzo[*f*]isoindole-6-carbaldehyde (1g)

White solid; mp 252–253 °C; R_f = 0.42 (PE–EtOAc, 6:1).

FT-IR (KBr): 3055, 2858, 1697, 1597, 1493, 1344, 1157, 1096, 704, 605 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 9.96 (s, 1 H), 8.11 (s, 1 H), 7.86–7.79 (m, 4 H), 7.70–7.58 (m, 6 H), 7.34–7.30 (m, 6 H), 4.57 (s, 4 H), 2.41 (s, 3 H).

^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 192.2, 143.8, 136.9, 136.6, 136.5, 135.3, 134.4, 133.9, 132.6, 129.8, 129.4, 129.3, 129.1, 129.0, 128.9, 128.5, 128.3, 127.6, 127.1, 122.6, 53.7, 53.6, 53.5, 21.5.

HRMS (EI): *m/z* calcd for $C_{32}H_{25}NO_3S$: 503.1555; found: 503.1610.

UV/Vis (MeCN): λ_{max} = 268 nm.

Methyl 4,9-Diphenyl-2-tosyl-2,3-dihydro-1*H*-benzo[*f*]isoindole-6-carboxylate (1h)

White solid; mp 247–248 °C; R_f = 0.39 (PE–EtOAc, 6:1).

FT-IR (KBr): 2949, 1720, 1599, 1440, 1344, 1285, 1252, 1159, 1096, 704, 605 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.38 (s, 1 H), 7.92 (d, *J* = 8.7 Hz, 1 H), 7.70–7.64 (m, 3 H), 7.57–7.51 (m, 6 H), 7.32–7.26 (m, 6 H), 4.56 (s, 4 H), 3.87 (s, 3 H), 2.40 (s, 3 H).

^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 167.1, 143.8, 137.1, 136.9, 135.5, 135.2, 134.5, 133.9, 131.6, 129.8, 129.4, 129.0, 128.9, 128.8, 128.3, 128.2, 127.7, 127.4, 126.2, 125.3, 53.6, 53.5, 52.2, 21.5.

HRMS (EI): *m/z* calcd for $C_{33}H_{27}NO_4S$: 533.1661; found: 533.1733.

UV/Vis (MeCN): λ_{max} = 253 nm.

6-Methyl-4,9-diphenyl-2-tosyl-2,3-dihydro-1*H*-benzo[*f*]isoindole (1i)

White solid; mp 266–267 °C; R_f = 0.43 (PE–EtOAc, 6:1).

FT-IR (KBr): 2858, 1597, 1493, 1344, 1157, 1096, 752, 704 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.69 (d, *J* = 7.4 Hz, 2 H), 7.63–7.58 (m, 5 H), 7.42–7.38 (m, 4 H), 7.22–7.18 (m, 5 H), 7.19 (d, *J* = 7.6 Hz, 1 H), 4.53 (s, 4 H), 2.40 (s, 3 H), 2.36 (s, 3 H).

^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 143.6, 137.8, 135.6, 133.5, 132.9, 132.8, 132.5, 131.9, 130.6, 129.8, 128.8, 128.7, 128.1, 127.9, 127.8, 127.6, 125.9, 125.8, 124.8, 53.6, 53.5, 21.7, 21.5.

HRMS (EI): *m/z* calcd for $C_{32}H_{27}NO_2S$: 489.1762; found: 489.1837.

UV/Vis (MeCN): λ_{max} = 239 nm.

4,9-Diphenyl-2-tosyl-2,3-dihydro-1*H*-benzo[*f*]isoindole (1j)

White solid; mp 270–271 °C; R_f = 0.39 (PE–EtOAc, 6:1).

FT-IR (KBr): 2857, 1597, 1494, 1344, 1159, 1093, 704, 678 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.69 (d, *J* = 7.5 Hz, 2 H), 7.64–7.61 (m, 3 H), 7.55–7.52 (m, 6 H), 7.33–7.28 (m, 7 H), 4.56 (s, 4 H), 2.40 (s, 3 H).

^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 143.7, 137.7, 133.7, 133.5, 132.8, 132.3, 129.8, 129.5, 128.8, 127.9, 127.6, 125.9, 53.6, 21.5.

HRMS (EI): *m/z* calcd for $C_{31}H_{25}NO_2S$: 475.1606; found: 475.1672.

UV/Vis (MeCN): λ_{max} = 243 nm.

6-Fluoro-4,9-diphenyl-2-tosyl-2,3-dihydro-1*H*-benzo[*f*]isoindole (1k)

White solid; mp 263–264 °C; R_f = 0.39 (PE–EtOAc, 6:1).

FT-IR (KBr): 2858, 1597, 1492, 1444, 1342, 1157, 705, 611 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.69 (d, *J* = 7.5 Hz, 1 H), 7.64–7.52 (m, 2 H), 7.64–7.52 (m, 6 H), 7.34–7.29 (m, 7 H), 7.21–7.12 (m, 1 H), 4.54 (s, 4 H), 2.40 (s, 3 H).

^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 162.4, 143.7, 137.4, 137.2, 134.1, 133.4, 129.8, 129.5, 129.4, 129.3, 128.9, 128.6, 128.4, 128.2, 128.1, 127.9, 127.6, 125.8, 120.7, 116.1, 115.8, 109.6, 109.3, 53.6, 53.5, 53.1, 21.5.

HRMS (EI): *m/z* calcd for $C_{31}H_{24}FNO_2S$: 493.1512; found: 493.1584.

UV/Vis (MeCN): λ_{max} = 244 nm.

Ethyl 4,9-Diphenyl-2-(phenylsulfonyl)-2,3-dihydro-1*H*-benzo[*f*]isoindole-6-carboxylate (2a)

White solid; mp 227–228 °C; R_f = 0.42 (PE–EtOAc, 6:1).

FT-IR (KBr): 3059, 2974, 1717, 1444, 1340, 1285, 1245, 1157, 1096, 752, 615 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.40 (s, 1 H), 7.92 (d, *J* = 8.4 Hz, 1 H), 7.80 (d, *J* = 6.2 Hz, 2 H), 7.68–7.64 (m, 1 H), 7.59–7.50 (m, 8 H), 7.34–7.28 (m, 5 H), 4.59 (s, 4 H), 4.32 (q, *J* = 6.3 Hz, 2 H), 1.33 (t, *J* = 6.2 Hz, 3 H).

^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 166.6, 137.1, 136.8, 135.3, 134.4, 133.8, 132.9, 131.6, 129.4, 129.3, 129.2, 128.9, 128.8, 128.3, 128.2, 127.8, 127.5, 126.3, 125.3, 61.1, 53.7, 53.6, 14.2.

HRMS (EI): *m/z* calcd for $C_{33}H_{27}NO_4S$: 533.1661; found: 533.1734.

UV/Vis (MeCN): λ_{max} = 251 nm.

1-[4,9-Diphenyl-2-(phenylsulfonyl)-2,3-dihydro-1*H*-benzo[*f*]isoindol-6-yl]ethanone (2b)

White solid; mp 203–204 °C; R_f = 0.40 (PE–EtOAc, 6:1).

FT-IR (KBr): 3057, 2860, 1684, 1599, 1444, 1346, 1155, 1097, 723, 621 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.25 (s, 1 H), 7.89 (d, *J* = 8.6 Hz, 1 H), 7.80 (d, *J* = 6.8 Hz, 1 H), 7.68 (d, *J* = 7.6 Hz, 2 H), 7.69–7.66 (m, 1 H), 7.57–7.51 (m, 8 H), 7.35–7.29 (m, 4 H), 4.59 (s, 4 H), 2.50 (s, 3 H).

^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 197.9, 136.9, 136.8, 135.6, 134.4, 133.9, 132.9, 129.4, 129.3, 129.2, 129.0, 128.9, 128.4, 128.2, 127.9, 127.5, 126.5, 123.9, 53.6, 53.5, 26.5.

HRMS (EI): *m/z* calcd for $C_{32}H_{25}NO_3S$: 503.1555; found: 503.1626.

UV/Vis (MeCN): λ_{max} = 265 nm.

7,11-Diphenyl-9-(phenylsulfonyl)-9,10-dihydro-8*H*-naphtho[1,2-*f*]isoindole (2c)

White solid; mp 239–240 °C; R_f = 0.41 (PE–EtOAc, 6:1).

FT-IR (KBr): 3057, 2851, 1597, 1445, 1348, 1312, 1163, 1097, 721, 619 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.79–7.72 (m, 5 H), 7.79–7.08 (m, 9 H), 7.57–7.50 (m, 5 H), 7.09–7.02 (m, 2 H), 4.57 (d, *J* = 19.2 Hz, 4 H).

^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 142.1, 138.3, 136.8, 135.3, 133.2, 132.8, 131.9, 130.5, 129.9, 129.5, 129.2, 128.9, 128.4, 128.0 127.9, 127.6, 127.4, 126.2, 125.3, 124.5, 54.7, 54.1.

HRMS (EI): *m/z* calcd for $C_{34}H_{25}NO_2S$: 511.1606; found: 511.1680.

UV/Vis (MeCN): λ_{max} = 270 nm.

4,9-Diphenyl-2-(phenylsulfonyl)-2,3-dihydro-1*H*-benzo[*f*]isoindole-6-carbaldehyde (2d)

White solid; mp 215–216 °C; R_f = 0.39 (PE–EtOAc, 6:1).

FT-IR (KBr): 3057, 2965, 1682, 1622, 1445, 1346, 1163, 1097, 721, 613 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 9.49 (s, 1 H), 7.83–7.80 (m, 4 H), 7.61–7.54 (m, 8 H), 7.36–7.30 (m, 6 H), 4.59 (s, 4 H).

^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 195.3, 149.1, 144.7, 137.1, 132.9, 132.3, 129.4, 129.3, 129.2, 129.1, 129.0, 128.9, 128.3, 128.2, 127.9, 127.5, 127.2, 126.8, 126.5, 122.7, 53.7, 53.6.

HRMS (EI): *m/z* calcd for $C_{31}H_{23}NO_3S$: 489.1379; found: 489.1468.

UV/Vis (MeCN): λ_{max} = 293 nm.

Methyl 4,9-Diphenyl-2-(phenylsulfonyl)-2,3-dihydro-1*H*-benzo[*f*]isoindole-6-carboxylate (2e)

White solid; mp 215–216 °C; R_f = 0.38 (PE–EtOAc, 6:1).

FT-IR (KBr): 2949, 1720, 1444, 1346, 1287, 1252, 1155, 1097, 752, 619 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.37 (s, 1 H), 7.92 (d, *J* = 8.5 Hz, 1 H), 7.80 (d, *J* = 7.0 Hz, 2 H), 7.70–7.67 (m, 4 H), 7.58–7.47 (m, 5 H), 7.31–7.27 (m, 5 H), 4.58 (s, 4 H), 3.86 (s, 3 H).

^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 167.1, 137.1, 136.8, 135.4, 133.8, 132.9, 131.6, 129.4, 129.3, 129.0, 128.9, 128.8, 128.3, 128.2, 127.5, 126.3, 125.3, 53.7, 53.6, 52.2.

HRMS (EI): *m/z* calcd for $C_{32}H_{25}NO_4S$: 519.1504; found: 519.1574.

UV/Vis (MeCN): λ_{max} = 253 nm.

6-Fluoro-4,9-diphenyl-2-(phenylsulfonyl)-2,3-dihydro-1*H*-benzo[*f*]isoindole (2f)

White solid; mp 245–246 °C; R_f = 0.39 (PE–EtOAc, 6:1).

FT-IR (KBr): 3059, 2860, 1627, 1445, 1344, 1161, 1097, 721, 621 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.80 (d, *J* = 6.7 Hz, 2 H), 7.64–7.60 (m, 4 H), 7.54–7.52 (m, 6 H), 7.32–7.29 (m, 5 H), 7.12 (t, *J* = 8.3 Hz, 1 H), 4.57 (s, 4 H).

^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 162.5, 137.4, 137.1, 134.1, 132.9, 129.5, 129.3, 129.2, 129.1, 129.0, 128.9, 128.8, 128.6, 128.4, 128.2, 128.1, 127.9, 127.5, 125.9, 116.2, 115.6, 109.6, 109.3, 53.6, 53.5.

HRMS (EI): *m/z* calcd for $C_{30}H_{22}FNO_2S$: 479.1365; found: 479.1427.

UV/Vis (MeCN): λ_{max} = 240 nm.

Ethyl 4,9-Bis(4-tolyl)-2-tosyl-2,3-dihydro-1*H*-benzo[*f*]isoindole-6-carboxylate (3a)

White solid; mp 252–253 °C; R_f = 0.39 (PE–EtOAc, 6:1).

FT-IR (KBr): 2978, 2920, 1717, 1516, 1344, 1285, 1250, 1157, 1096, 675, 607 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.42 (s, 1 H), 7.89 (d, *J* = 7.8 Hz, 1 H), 7.68 (d, *J* = 7.1 Hz, 3 H), 7.36–7.34 (m, 4 H), 7.31–7.29 (m, 2 H), 7.21–7.19 (m, 4 H), 4.56 (s, 4 H), 4.33(q, *J* = 6.9 Hz, 2 H), 2.50 (s, 6 H), 2.40 (s, 3 H), 1.33 (t, *J* = 6.75 Hz, 3 H).

^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 166.7, 143.7, 137.9, 137.8, 135.5, 134.6, 134.2, 133.9, 133.6, 133.5, 131.7, 129.8, 129.6, 129.3, 129.2, 128.9, 127.6, 126.2, 125.0, 61.0, 53.7, 53.6, 21.5, 21.3, 14.3.

HRMS (EI): *m/z* calcd for $C_{36}H_{33}NO_4S$: 575.2130; found: 575.2201.

UV/Vis (MeCN): λ_{max} = 251 nm.

7,11-Bis(4-methylphenyl)-9-tosyl-9,10-dihydro-8*H*-naphtho[1,2-*f*]isoindole (3b)

White solid; mp 266–267 °C; R_f = 0.49 (PE–EtOAc, 6:1).

FT-IR (KBr): 3026, 2864, 1512, 1443, 1331, 1153, 1097, 752, 669 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.75–7.67 (m, 5 H), 7.57–7.54 (m, 2 H), 7.37–7.29 (m, 6 H), 7.21–7.10 (m, 5 H), 4.57 (s, 2 H), 4.51 (s, 2 H), 2.53 (s, 3 H), 2.51 (s, 3 H), 2.40 (s, 3 H).

^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 139.1, 137.6, 137.4, 135.5, 133.3, 133.0, 130.6, 129.8, 129.5, 128.3, 128.1, 127.5, 127.4, 126.1, 125.2, 124.6, 54.8, 54.2, 21.5, 21.4, 21.3.

HRMS (EI): *m/z* calcd for $C_{37}H_{31}NO_2S$: 553.2075; found: 553.2134.

UV/Vis (MeCN): λ_{max} = 268 nm.

Methyl 4,9-Bis(4-methylphenyl)-2-tolyl-2,3-dihydro-1*H*-benzo[*f*]isoindole-6-carboxylate (3c)

White solid; mp 260–261 °C; R_f = 0.45 (PE–EtOAc, 6:1).

FT-IR (KBr): 3024, 2863, 1718, 1516, 1443, 1345, 1285, 1252, 1157, 1096, 813, 675 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.42 (s, 1 H), 7.90 (m, 1 H), 7.70 (m, 3 H), 7.36–7.22 (m, 6 H), 7.24–7.20 (m, 4 H), 4.56 (s, 4 H), 3.87 (s, 3 H), 2.52 (s, 6 H), 2.40 (s, 3 H).

^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 167.2, 143.8, 137.9, 137.8, 135.5, 134.6, 134.1, 133.9, 133.8, 133.6, 131.7, 129.8, 129.7, 129.6, 129.3, 128.9, 127.6, 127.3, 126.3, 125.1, 53.7, 53.6, 52.3, 21.5, 21.4, 21.3.

HRMS (EI): *m/z* calcd for $C_{35}H_{31}NO_4S$: 561.1974; found: 561.2043.

UV/Vis (MeCN): λ_{max} = 249 nm.

Ethyl 4,9-Bis(4-chlorophenyl)-2-tosyl-2,3-dihydro-1*H*-benzo[*f*]isoindole-6-carboxylate (4a)
White solid; mp 262–263 °C; R_f = 0.45 (PE–EtOAc, 6:1).

FT-IR (KBr): 2978, 1717, 1493, 1344, 1286, 1248, 1157, 1094, 825, 605 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.34 (s, 1 H), 7.95 (d, *J* = 7.9 Hz, 1 H), 7.70 (d, *J* = 7.5 Hz, 2 H), 7.63–7.27 (m, 5 H), 7.32–7.26 (m, 6 H), 4.52 (s, 4 H), 4.34 (q, *J* = 6.9 Hz, 2 H), 2.41 (s, 3 H), 1.35 (t, *J* = 6.9 Hz, 3 H).

^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 166.4, 143.9, 135.5, 135.4, 135.1, 134.5, 134.4, 134.3, 134.0, 131.5, 130.7, 130.6, 129.9, 129.3, 128.5, 128.2, 127.6, 125.9, 125.6, 124.7, 61.2, 53.4, 53.3, 21.5, 18.2.

HRMS (EI): *m/z* calcd for $C_{34}H_{27}Cl_2NO_4S$: 615.1038; found: 615.1103.

UV/Vis (MeCN): λ_{max} = 250 nm.

7,11-Bis(4-chlorophenyl)-9-tosyl-9,10-dihydro-8*H*-naphtho[1,2-*f*]isoindole (4b)
White solid; mp 255–256 °C; R_f = 0.46 (PE–EtOAc, 6:1).

FT-IR (KBr): 3049, 1597, 1491, 1350, 1094, 817, 671 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.80 (d, *J* = 7.7 Hz, 1 H), 7.69 (d, *J* = 7.9 Hz, 1 H), 7.64–7.60 (m, 5 H), 7.56–7.48 (m, 3 H), 7.32–7.26 (m, 6 H), 7.17–7.12 (m, 2 H), 4.54 (s, 2 H), 4.48 (s, 2 H), 2.41 (s, 3 H).

^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 140.4, 136.5, 135.4, 134.2, 133.9, 133.4, 133.1, 130.8, 130.2, 129.9, 129.8, 129.2, 128.6, 128.0, 127.8, 127.6, 126.4, 125.6, 124.0, 54.5, 53.9, 21.5.

HRMS (EI): *m/z* calcd for $C_{35}H_{25}Cl_2NO_2S$: 593.0983; found: 593.1049.

UV/Vis (MeCN): λ_{max} = 274 nm.

6-Chloro-4,9-bis(4-chlorophenyl)-2-tosyl-2,3-dihydro-1*H*-benzo[*f*]isoindole (4c)
White solid; mp 276–277 °C; R_f = 0.45 (PE–EtOAc, 6:1).

FT-IR (KBr): 3030, 2860, 1597, 1493, 1344, 1157, 1094, 1014, 821, 669 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.69 (d, *J* = 6.5 Hz, 2 H), 7.56–7.52 (m, 6 H), 7.33–7.30 (m, 3 H), 7.29–7.25 (m, 4 H), 4.50 (s, 4 H), 2.41 (s, 3 H).

^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 143.9, 135.4, 135.2, 134.5, 134.4, 134.3, 133.4, 132.6, 130.8, 130.7, 129.9, 129.4, 129.3, 129.2, 127.6, 127.4, 127.1, 124.5, 53.4, 53.3, 21.5.

HRMS (EI): *m/z* calcd for $C_{31}H_{22}Cl_3NO_2S$: 577.0437; found: 577.0497.

UV/Vis (MeCN): λ_{max} = 245 nm.

Methyl 4,9-Bis(4-chlorophenyl)-2-tosyl-2,3-dihydro-1*H*-benzo[*f*]isoindole-6-carboxylate (4d)
White solid; mp 275–276 °C; R_f = 0.45 (PE–EtOAc, 6:1).

FT-IR (KBr): 2953, 1718, 1493, 1344, 1259, 1157, 1093, 821, 675 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.32 (s, 1 H), 7.96 (s, 1 H), 7.53 (d, *J* = 7.5 Hz, 2 H), 7.64–7.53 (m, 4 H), 7.32–7.25 (m, 7 H), 4.52 (s, 4 H), 3.89 (s, 3 H), 2.41 (s, 3 H).

^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 166.80, 143.9, 135.6, 135.2, 135.0, 134.4, 134.3, 133.2, 131.4, 130.7, 129.8, 129.3, 129.2, 128.4, 127.7, 127.5, 126.0, 125.6, 53.4, 53.3, 52.3, 21.5.

HRMS (EI): *m/z* calcd for $C_{33}H_{25}Cl_2NO_4S$: 601.0881; found: 601.0934.

UV/Vis (MeCN): λ_{max} = 251 nm.

7,11-Bis(4-methoxyphenyl)-9-tosyl-9,10-dihydro-8*H*-naphtho[1,2-*f*]isoindole (5a)
White solid; mp 245–246 °C; R_f = 0.45 (PE–EtOAc, 6:1).

FT-IR (KBr): 2833, 1608, 1514, 1460, 1335, 1244, 1163, 1097, 1029, 831, 667 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.78–7.68 (m, 4 H), 7.60–7.56 (m, 2 H), 7.43–7.38 (m, 2 H), 7.25–7.19 (m, 5 H), 7.14–7.08 (m, 5 H), 4.59 (s, 2 H), 4.52 (s, 2 H), 3.95 (s, 6 H), 2.40 (s, 3 H).

^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 159.2, 159.1, 143.6, 135.8, 134.3, 134.2, 133.9, 133.5, 133.1, 132.3, 130.7, 130.4, 129.8, 129.4, 128.4, 127.9, 127.6, 127.4, 126.1, 125.3, 124.5, 115.3, 114.3, 55.4, 55.3, 54.7, 54.2, 21.5.

HRMS (EI): *m/z* calcd for $C_{37}H_{31}NO_4S$: 585.1974; found: 585.2043.

UV/Vis (MeCN): λ_{max} = 236 nm.

Acknowledgment

The authors thank the National Science Foundation of China (20872002) for financial support.

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