[2+2+2] Cocyclization Using [$Mo(CO)_6$-*p*-ClPhOH]

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Since Mortreux reported that $Mo(CO)_6$-*p*-ClPhOH was an effective catalyst for alkyne metathesis, the reaction has attracted considerable attention, and great interest has been devoted to the reaction mechanism.[1] Schrock reported that the active species of this catalytic system was carbyne complex.[2] In this reaction, *p*-ClPhOH is required, but the role of *p*-ClPhOH is still unclear.[3] Recently, we reported a novel synthesis of disubstituted alkynes via cross-alkyne metathesis.[4] In this article,[4b] we showed that alkyne having an *o*-hydroxyphenyl group provided trimerization product, whereas alkynes having *m*- or *p*-hydroxyphenyl group gave cross-alkyne methathesis products. From these results, we considered that molybudenacyclopentadiene should be formed in this reaction and that alkyne metathesis would proceed via the same molybdenacyclopentadiene.

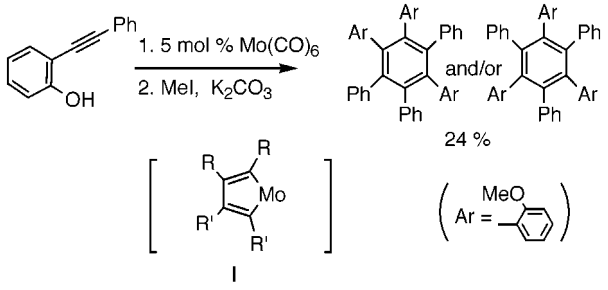
Because the trimerization product might have been produced via molybdenapentadiene **I**, dialkyne in a tether would react with alkyne via molybdenacycle **II** to afford [2+2+2] cocyclization compounds as shown in Scheme 3. Based on this idea, we report here [2+2+2] cocyclization from dialkyne and alkyne in the presence of $Mo(CO)_6$-*p*-ClPhOH.[5]

When a toluene solution of dialkyne **1** (1 mmol) and diethylacetylene (2 equiv) was refluxed in the presence of $Mo(CO)_6$ (35 mol %) and *p*-ClC_6H_4OH (100 mol %) for 3.5 h, the [2+2+2] cocyclization product **3a** (R = Et, R′ = Et) was produced in 10% yield along with 8% of **4** (Table 1, run 1).

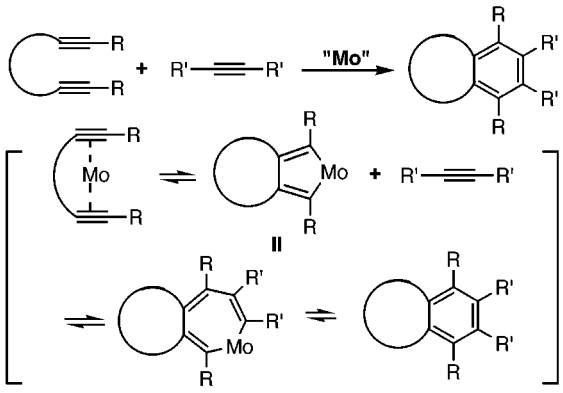
Byproduct **4** should be produced from molybdenacyclopentadiene **II**, and it accounted for the reaction mechanism via molybdenacycle **II**. To increase the yield of product **3**, the effects of the amount of diethylacetylene were examined. The reaction with a large amount of diethylacetylene provided **3** in good yield (runs 2, 3, and 4). When 15 equiv of diethylacetylene was used in the reaction, **3a** was obtained in 44% yield (run 4).[6] Furthermore, even 20 mol % catalyst was effective enough to provide 43% of cyclized product **3a** (run 5). Cyclization using alkyne with aromatic rings also succeeded despite large steric hindrance. The reaction of **1** with 15 equiv of diphenylacetylene produced **3b** (R = Ph, R′ = Ph) in 43% yield (run 6). The reaction with phenylpropyne afforded **3c** (R = Ph, R′ = Me) in 49% yield as a sole product, and no **3b** or **3d** (R = Me, R′ = Me) was obtained (run 7).

Scheme 1

Scheme 2



Scheme 3



Next, intramolecular [2+2+2] cocyclizations in the presence of $Mo(CO)_6$-*p*-ClPhOH were investigated (Table 2). The reaction of **5** (n = 1) was accomplished in 2 h to afford **6** (n = 1) in 44% yield (run 1). In **5** (n = 2), the cyclized product was obtained in 37% yield (run 2).

Thus, [2+2+2] cocyclization took place in the presence of Mortrex's catalyst [$Mo(CO)_6$-*p*-ClPhOH]. Byproduct **4** indicated that these reactions should proceed via molybdenacyclopentadiene **II**. Further studies on the mechanism of the alkyne metathesis are in progress.

Experimental Section

General. All the manipulations were performed under Ar unless otherwise mentioned. Anhydrous solvents were obtained by distillation from benzophenone ketyl (diethyl ether, tetrahydrofuran), calcium hydride (CH_2Cl_2), or LAH (toluene).

Preparation of *N,N*-Di-2-butynyl-*p*-toluenesulfonamide (1). To a solution of *p*-toluenesulfonamide (3.04 g, 17.8 mmol) in dimethylformamide (DMF) (50 mL) was added NaH (60%

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(2) McCullough L. G.; Schrock, R. R. *J. Am. Chem. Soc.* **1984**, *106*, 4067.

(3) Recently, Bunz reported that a Schrock-type carbyne [$(ArO)_3$-Mo*f*CR] would be accessed by in situ oxidation of $Mo(CO)_6$ in the presence of PhOH. Koppenburg, L.; Song. D.; Burtz, U. H. F. *J. Am. Chem. Soc.* **1998**, *120*, 7973.

(4) (a) Kaneta, N.; Hirai, T.; Mori, M. *Chem. Lett.* **1995**, 627. (b) Kaneta, N.; Hikichi, K.; Asaka, S.; Uemura, M.; Mori, M. *Chem. Lett.* **1995**, 1055.

(5) The synthesis of phenol from metal-carbynes and diyenes was reported by Katz. Sivavec T. M.; Katz T. J. *Tetrahedron Lett.* **1985**, *26*, 2159.

(6) When the reaction was carried out without *p*-ClPhOH, **3a** and **4** were obtained in 6% and 5% yields, respectively, and 19% of **1** was recovered.

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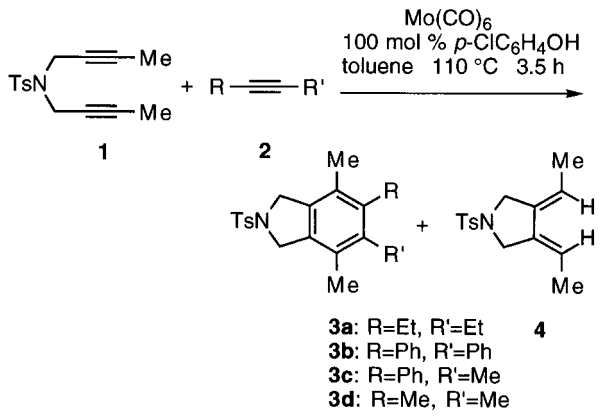
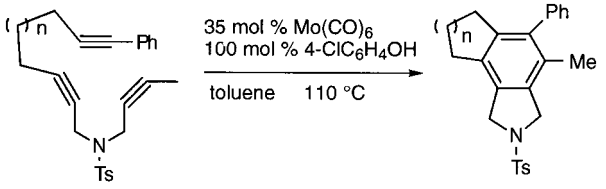
Table 1. [2+2+2] Cocyclization of Dialkyne and Alkyne



run	R	R'	eq.	$Mo(CO)_6$ mol %		**3** (%)	**4** (%)
1	Et	Et	2	35	**3a**	10	8
2	Et	Et	5	35	**3a**	25	6
3	Et	Et	8	35	**3a**	34	3
4	Et	Et	15	35	**3a**	44	5
5	Et	Et	15	20	**3a**	43	7
6	Ph	Ph	15	35	**3b**	43	5
7	Ph	Me	15	35	**3c**	49[a]	3

[a] The reaction time was 100 min.

Table 2. Intramolecular [2+2+2] Cocyclization



run	*n*	time (h)	**6** (%)
1	1	2.0	44
2	2	2.5	37

dispersion, 1.58 g, 37.8 mmol) at 0 °C. After 25 min at room temperature, a solution of 1-methansulfoxy-2-butyne (5.79 g, 39.1 mmol) in DMF (70 mL) was added. The reaction mixture was stirred for 1 h and quenched by the addition of aq. NH_4Cl. The water layer was extracted with ether. The combined ether extracts were washed with brine, dried over Na_2SO_4, concentrated, and chromatographed (hexane:AcOEt = 4:1) to give **1** (4.17 g, 85%). **1**: IR (KBr) 1346, 1332, 1162, 660 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$, TMS) δ 7.73−7.70 (m, 2 H), 7.30−7.27 (m, 2 H), 4.08 (q, J = 2.3 Hz, 4 H), 2.42 (s, 3 H), 1.65 (t, J = 2.3 Hz, 6H); ^{13}C NMR (270 MHz, $CDCl_3$) d 143.4 (C), 135.5 (C), 129.1 (CH), 127.9 (CH), 81.5 (C), 71.5 (C), 36.6 (CH_2), 21.4 (CH_3), 3.3 (CH_3); EI-MS *m*/*z* (%); 275(M^+, 1.5), 260 (35.3), 155 (24.6), 139 (16.7), 120 (87.2), 91 (100.0); EI−HRMS calcd for $C_{15}H_{17}O_2NS$ 275.0981, found 275.0973.

Preparation of *N*-2-Butynyl-*N*-(8-phenyl-2,7-octadiynyl)-*p*-toluenesulfonamide (5) (*n* = 1) and *N*-2-Butynyl-*N*-(9-phenyl-2,8-nonadiynyl)-*p*-toluenesulfonamide (5) (*n* = 2). *N*-*tert*-Butoxycarbonyl-*N*-(8-phenyl-2,7-octadiynyl)-*p*-toluenesulfonamide: To a solution of *N*-*tert*-butoxycarbonyl-*N*-*p*-toluenesulfonamide (1.06 g, 3.89 mmol), PPh_3(1.02 g, 3.9 mmol), and 8-pheny-2,7-octadiyn-1-ol (762 mg, 3.85 mmol) in tetrahydrofuran (THF) (9 mL) was added DEAD (0.71 mL, 3.91 mmol) at 0 °C. After 2 h, the reaction was quenched by the addition of aq. NH_4Cl. The water layer was extracted with AcOEt. The organic extracts were washed with brine, dried over Na_2SO_4, concentrated, and chromatographed (hexane:AcOEt = 6:1) to give *N*-*tert*-butoxycarbonyl-*N*-(8-phenyl-2,7-octadiynyl)-*p*-toluenesulfonamide (1.69 g, 97%): IR (neat) 2228, 1732, 1598, 1490, 1362, 1154, 758, 674 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$, TMS) δ 7.93 (d, J = 8.2 Hz, 2 H), 7.40−7.37 (m, 2 H), 7.31−7.26 (m, 5 H), 4.62 (t, J = 2.0 Hz, 2 H), 2.50 (t, J = 6.9 Hz, 2 H), 2.40 (tt, J = 6.9, 2.0 Hz, 2 H), 2.37 (s, 3 H), 1.80 (tt, J = 6.9, 6.9 Hz, 2 H), 1.35 (s, 9 H); EI-MS *m*/*z* (%); 451 (M^+, 0.2), 350 (0.7), 155 (16.7), 115 (15.7), 91 (51.9), 57 (78.1), 41 (100.0); EI−HRMS calcd for $C_{26}H_{29}O_4NS$ 451.1819, found 451.1807.

***N*-(8-Phenyl-2,7-octadiynyl)-*p*-toluenesulfonamide:** To a solution of *N*-*tert*-butoxycarbonyl-*N*-(8-phenyl-2,7-octadiynyl)-*p*-toluenesulfonamide (1.63 g, 3.62 mmol) in CH_2Cl_2 (9 mL), was added trifluoroacetic acid (TFA) (1.4 mL, 18.4 mmol) at 0 °C. After 4 h, the reaction was quenched by the addition of aq. $NaHCO_3$. The water layer was extracted with AcOEt. The organic extracts were washed with brine, dried over Na_2SO_4, concentrated, and chromatographed (hexane:AcOEt = 9:2) to give *N*-(8-phenyl-2,7-octadiynyl)-*p*-toluenesulfonamide (1.14 g, 90%): IR (KBr) 3050, 2224, 1596, 1492, 1344, 1154, 758, 690 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$, TMS) δ 7.77 (d, J = 8.6 Hz, 2 H), 7.40−7.36 (m, 2 H), 7.33−7.26 (m, 5 H), 4.55 (t, J = 5.9 Hz, 1 H), 3.82 (dt, J = 5.9, 2.0 Hz, 2 H), 2.41 (s, 3 H), 2.36 (t, J = 6.9 Hz, 2 H), 2.16 (tt, J = 6.9, 2.0 Hz, 2 H), 1.60 (tt, J = 6.9, 6.9 Hz, 2 H); EI-MS *m*/*z* (%); 351 (M^+, 2.4), 350 (8.3), 196 (65.2), 115 (38.7), 91 (100.0); EI−HRMS calcd for $C_{21}H_{21}O_2NS$ 351.1294, found 351.1279.

***N*-2-Butynyl-*N*-(8-phenyl-2,7-octadiynyl)-*p*-toluenesulfonamide (5) (*n* = 1):** To a solution of *p*-toluenesulfonamide (1.06 g, 3.03 mmol) in DMF (7 mL), was added NaH (60% dispersion, 138 mg, 3.64 mmol) at 0 °C. After 40 min at room temprature, a solution of 1-methansulfoxy-2-butyne (586 mg, 3.84 mmol) in DMF (13 mL) was added. The reaction mixture was stirred for 1.5 h and quenched by the addition of aq. NH_4Cl at 0 °C. The water layer was extracted with ether. The combined ether extracts were washed with brine, dried over Na_2SO_4, concentrated, and chromatographed (hexane:AcOEt = 50:1−20:1) to give **5** (*n* = 1) (1.03 g, 84%). **5** (*n* = 1): IR (neat) 2228, 1598, 1490, 1350, 1164, 758, 658 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$, TMS) δ 7.74−7.70 (m, 2 H), 7.40−7.36 (m, 2 H), 7.30−7.26 (m, 5 H), 4.13 (t, J = 2.0 Hz, 2 H), 4.08 (q, J = 2.3 Hz, 2 H), 2.40 (s, 3 H), 2.39 (t, J = 6.9 Hz, 2 H), 2.20 (tt, J = 6.9, 2.0 Hz, 2 H), 1.67 (t, J = 2.3 Hz, 3 H), 1.63 (tt, J = 6.9, 6.9 Hz, 2 H); EI-MS *m*/*z* (%); 403 (M^+, 1.3), 248 (38.9), 221 (11.1), 167 (12.7), 155 (13.3), 129 (10.5), 115 (37.7), 91 (100.0), 77 (18.8); EI−HRMS calcd for $C_{25}H_{25}O_2NS$ 403.1608, found 403.1616.

N-2-Butynyl-*N*-(9-phenyl-2,8-nonadiynyl)-*p*-toluenesulfonamide (**5**) (*n* = 2) was prepared as discribed for **5** (*n* = 2). **5** (*n* = 2): IR (neat) 2232, 1598, 1490, 1350, 1162, 756, 658 cm^{-1}; ^1H NMR (270 MHz, $CDCl_3$, TMS) δ 7.71 (d, J = 8.6 Hz, 2 H), 7.41−7.37 (m, 2 H), 7.29−7.26 (m, 5 H), 4.12 (t, J = 2.0 Hz, 2 H), 4.07 (q, J = 2.6 Hz, 2 H), 2.41 (s, 3 H), 2.38 (t, J = 6.6 Hz, 2 H), 2.08 (tt, J = 6.6, 2.0 Hz, 2 H), 1.65 (t, J = 2.6 Hz, 3 H), 1.60−1.53 (m, 4 H); EI-MS *m*/*z* (%); 417 (M^+, 1.3), 155 (14.3), 129 (10.7), 115 (41.4), 91 (100.0), 89 (10.2), 77 (16.3); EI−HRMS calcd for $C_{26}H_{27}O_2NS$ 417.1764, found 417.1763.

General Procedure (Table 1, run 4). To a mixture of **1** (275 mg, 1 mmol), $Mo(CO)_6$ (92.7 mg, 35 mmol, 35 mol %), and *p*-chlorophenol (129 mg, 1 mmol) in degassed toluene (10 mL) was added 3-hexyne (1.7 mL, 15 mmol). The whole reaction mixture was refluxed for 210 min. After addition of ether, the organic layer was washed with 10% NaOH and brine, dried over Na_2SO_4, concentrated, and chromatographed (hexane:AcOEt = 30:1−15:1) to give 5,6-diethyl-4,7-dimethyl-2-*p*-toluenesulfonylisoindoline (**3a**) (158.4 mg, 44% yield) and 3(*E*),4(*E*)-diethylidene-1-*p*-toluenesulfonylpyrrolidine (**4**) (14.5 mg, 5% yield). **3a:** mp 175.5−176.5 °C (2-propanol). IR (Nujol) 2360, 2344, 1458, 1344, 1160 cm^{-1}. ^1H NMR (270 MHz, $CDCl_3$, TMS) δ 7.78 (d, J = 7.9 Hz, 2 H), 7.30 (d, J = 7.9 Hz, 2 H), 4.57 (s, 4 H), 2.62 (q, J = 7.3 Hz, 4 H), 2.40 (s, 3 H), 2.12 (s, 6 H), 1.08 (t, J = 7.3 Hz, 6 H). ^{13}C NMR (400 MHZ, $CDCl_3$) d 143.4 (C), 140.0 (C), 133.8 (C), 132.8 (C), 129.7 (CH), 127.8 (C), 127.5 (CH), 54.1 (CH_2), 22.1 (CH_2), 21.4 (CH_3), 15.5 (CH_3), 14.6 (CH_3). EI-MS *m*/*z* (%); 357 (M^+, 9.6), 342 (2.5), 202 (51.8), 201 (100.0), 186 (36.1), 172 (17.8), 158 (8.1), 91 (26.7). EI−HRMS calcd for $C_{21}H_{27}O_2NS$ 357.1764, found 357.1785. Anal. Calcd for $C_{21}H_{27}O_2NS$: C, 70.55; H, 7.61; N, 3.92; S, 8.97. Found: C, 70.38; H, 7.56; N, 3.89; S, 9.01. **4:** mp 120−121 °C (hexane-benzene). IR (Nujol) 2360, 2342, 1670, 1456, 1340, 1164, 1098, 820 cm^{-1}. ^1H NMR (270 MHz, $CDCl_3$, TMS) d 7.74 (d, J = 8.3 Hz, 2 H), 7.33 (d, J = 8.3 Hz, 2 H), 5.73 (q, J = 6.6 Hz, 2 H), 3.94 (s, 4 H), 2.43 (s, 3 H), 1.63 (d, J = 6.6 Hz, 6 H). ^{13}C NMR (400 MHZ, $CDCl_3$) d 143.6 (C), 134.4 (C), 133.1 (C), 129.7 (CH), 127.8 (CH), 114.0 (CH), 51.0 (CH_2), 21.5

(CH_3), 14.6 (CH_3). EI-MS *m*/*z* (%); 277 (M^+, 22.6), 262 (18.8), 214 (78.3), 199 (67.0), 155 (17.4), 121 (100.0), 106 (53.4), 91 (64.2). EI−HRMS calcd for $C_{15}H_{19}O_2NS$ 277.1138, found 277.1134. Anal. calcd for $C_{15}H_{19}O_2NS$: C, 64.95; H, 6.90; N, 5.05; S, 11.56. Found: C, 65.03; H, 6.87; N, 4.96; S, 11.36.

5,6-Dimethyl-4,7-diphenyl-2-*p*-toluenesulfonylisoindoline (3b): mp 199−200 °C (ether). IR (Nujol) 2360, 2344, 1456, 1348, 1164, 668 cm^{-1}. ^1H NMR (270 MHz, $CDCl_3$, TMS) δ 7.84 (d, J = 8.3 Hz, 2 H), 7.36 (d, J = 8.3 Hz, 2 H), 7.14−7.05 (m, 6 H), 6.87−6.83 (m, 4 H), 4.69 (s, 4 H), 2.44 (s, 3 H), 1.91 (s, 6 H). ^{13}C NMR (400 MHZ, $CDCl_3$) d 143.6 (C), 141.6 (C), 140.0 (C), 134.3 (C), 134.2 (C), 130.1 (CH), 129.9 (CH), 128.0 (C), 127.7 (CH), 127.5 (CH), 126.2 (CH), 54.1 (CH_2), 21.5 (CH_3), 17.1 (CH_3). EI-MS *m*/*z* (%); 453 (M^+, 12.1), 298 (51.3), 297 (100.0), 282 (6.4), 269 (5.8), 155 (3.4), 91 (16.9). EI−HRMS calcd for $C_{29}H_{27}O_2$ NS 453.1764, found 453.1766. Anal. calcd for $C_{29}H_{27}O_2$ NS: C, 76.79; H, 6.00; N, 3.09; S, 7.07. Found: C, 76.69; H, 6.09; N, 2.96; S, 6.98.

4,5,7-Trimethyl-6-phenyl-2-*p*-toluenesulfonylisoindoline (3c): mp 176−177 °C (ether). IR (Nujol) 2360, 2346, 1456, 1348, 1166, 668 cm^{-1}. ^1H NMR (270 MHz, $CDCl_3$, TMS) δ 7.82−7.79 (m, 2 H), 7.43−7.31 (m, 5 H), 7.05−7.02 (m, 2 H), 4.65 (s, 2 H), 4.61 (s, 2 H), 2.42 (s, 3 H), 2.13 (s, 3 H), 1.88 (s, 3 H), 1.81 (s, 3 H). ^{13}C NMR (270 MHZ, $CDCl_3$) d 144.0 (C), 142.4 (C), 141.4 (C), 135.0 (C), 134.4 (C), 132.7 (C), 130.3 (CH), 129.6 (CH), 128.9 (CH), 128.8 (C), 128.4 (C), 128.1 (CH), 127.2 (CH), 54.5 (CH_2), 54.4 (CH_2), 21.9 (CH_3), 17.6 (CH_3), 17.5 (CH_3), 16.7 (CH_3). EI-MS *m*/*z* (%); 391 (M^+, 7.4), 236 (55.6), 235 (100.0), 220 (18.4), 179 (12.0), 155 (13.2), 91 (62.7). EI−HRMS calcd for $C_{24}H_{25}O_2$-NS 391.1608, found 391.1622. Anal. calcd for $C_{24}H_{25}O_2$ NS: C, 73.62; H, 6.44; N, 3.58; S, 8.19. Found: C, 73.73; H, 6.54; N, 3.49; S, 8.26.

General Procedure (Table 2, run 1). A mixture of **5** (n = 1) (455.9 mg, 1.13 mmol), $Mo(CO)_6$ (104.8 mg, 0.40 mmol, 35 mol %) and *p*-chlorophenol (145.7 mg, 1.13 mmol) in degassed toluene (11 mL) was refluxed for 120 min. After addition of ether, the organic layer was washed with 10% NaOH and brine, dried over Na_2SO_4, concentrated, and chromatographed (hexane: AcOEt = 30:1−15:1) to give **6** (n = 1) (201.7 mg, 44% yield).

4-Methyl-5-phenyl-2-*p*-toluenesulfonyl-6,7-cyclopentenoisoindoline (6) (n = 1): mp 180−181 °C (AcOEt). IR (KBr) 3052, 1598, 1458, 1344, 1164, 672 cm^{-1}. ^1H NMR (270 MHz, $CDCl_3$, TMS) δ 7.81 (d, J = 7.9 Hz, 2 H), 7.41−7.28 (m, 5 H), 7.13−7.10 (m, 2 H), 4.60 (s, 4 H), 2.78 (t, J = 7.3 Hz, 2 H), 2.57 (t, J = 7.3 Hz, 2 H), 2.41 (s, 3 H), 1.98 (tt, J = 7.3, 7.3 Hz, 2 H), 1.94 (s, 3 H). ^{13}C NMR (125 MHZ, $CDCl_3$) d 143.5 (C), 143.4 (C), 139.8 (C), 138.0 (C), 135.3 (C), 133.9 (C), 133.7 (C), 130.5 (C), 129.8 (CH), 128.9 (CH), 128.2 (CH), 128.0 (C), 127.6 (CH), 126.8 (CH), 53.6 (CH_2), 53.3 (CH_2), 32.5 (CH_2), 31.1 (CH_2), 25.2 (CH_2), 21.4 (CH_3), 16.4 (CH_3). EI-MS *m*/*z* (%); 403 (M^+, 10.4), 248 (53.9), 247 (100.0), 232 (4.4), 91 (20.0). EI−HRMS calcd for $C_{25}H_{25}O_2NS$ 403.1608, found 403.1579. Anal. Calcd for C_{25}-$H_{25}O_2NS$: C, 74.41; H, 6.24; N, 3.4

4-Methyl-5-phenyl-2-*p*-toluenesulfonyl-6,7-cyclohexenoisoindoline (6) (n = 2): IR (KBr) 3056, 1598, 1458, 1348, 1166, 670 cm^{-1}. ^1H NMR (270 MHz, $CDCl_3$, TMS) δ 7.80 (d, J = 8.2 Hz, 2 H), 7.43−7.28 (m, 5 H), 7.05−7.01 (m, 2 H), 4.60 (s, 2 H), 4.59 (s, 2 H), 2.55 (t, J = 5.9 Hz, 2 H), 2.42 (s, 3 H), 2.25 (t, J = 5.9 Hz, 2 H), 1.81 (s, 3 H), 1.77−1.68 (m, 2 H), 1.66−1.56 (m, 2 H). ^{13}C NMR (270 MHZ, $CDCl_3$) d 143.5 (C), 141.6 (C), 140.2 (C), 135.0 (C), 133.9 (C), 133.6 (C);132.2 (C), 129.8 (CH), 129.2 (C), 129.0 (CH), 128.5 (CH), 127.5 (CH), 126.7 (CH), 53.8 (CH_2), 53.5 (CH_2), 28.6 (CH_2), 26.7 (CH_2), 23.1 (CH_2), 22.3 (CH_2), 21.4 (CH_3), 16.7 (CH_3). EI-MS *m*/*z* (%); 417 (M^+, 11.7), 262 (58.7), 261 (100.0), 246 (11.1), 91 (20.6). EI−HRMS calcd for $C_{26}H_{27}O_2$ NS 417.1764, found 417.1763.

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