

## Chapter 2

# Iron-Catalyzed Naphthalene Synthesis from Alkyne and Grignard Reagent

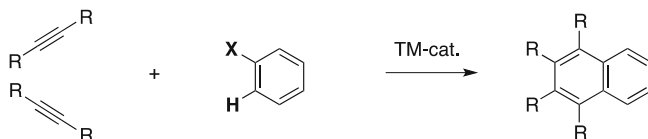
### 2.1 Introduction

Linear fused acenes are important structures for organic semiconductors [1]. Naphthalene is the simplest acene and the [2+2+2] annulation reaction of an aromatic ring with two alkynes via C–H bond activation is an efficient method for synthesizing naphthalenes or other acene structures (Scheme 2.1). This strategy was originally developed using stoichiometric amounts of chromium by the Zeiss group in 1959 [2, 3]. The Sakakibara and Heck groups also developed a palladium-catalyzed reaction of iodobenzene and alkyne [4–7]. Recently, the Satoh and Miura groups reported several iridium [8, 9]—and rhodium [10–14]—catalyzed reactions. However, these reactions typically require harsh conditions and there have been no examples of iron-catalyzed reactions.

As described in the previous chapter, the author previously found that direct functionalization of an aromatic C–H bond can proceed using an iron catalyst and an oxidant [15–20]. Furthermore, it is known that an iron catalyst promotes the carbometalation reaction of alkynes [21–24]. Using these results, iron-catalyzed alkyne annulation via C–H bond activation was envisioned (Scheme 2.2). In this chapter, iron-catalyzed naphthalene synthesis is investigated as the first trial of iron-catalyzed fused aromatic synthesis via C–H bond activation.

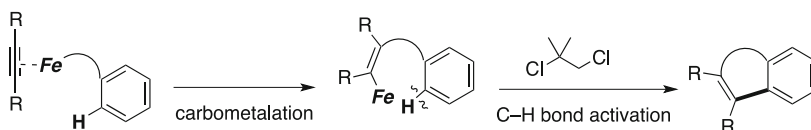
### 2.2 Initial Finding of Naphthalene Formation

The initial discovery of iron-catalyzed alkyne annulation occurred during the study of the iron-catalyzed reaction of alkyne and phenyl Grignard reagent. The reaction of diphenylacetylene with phenylmagnesium bromide proceeded in the presence of iron(III) acetylacetonate and bipyridine-type ligand with organic dichloride oxidant to give 1,2,3,4-tetraphenylnaphthalene as a product (Eq. 2.1). Usually, alkyne carbometalation reactions proceed under these conditions [21–24]. However, in the presence of an oxidant, naphthalene became the major product instead of the



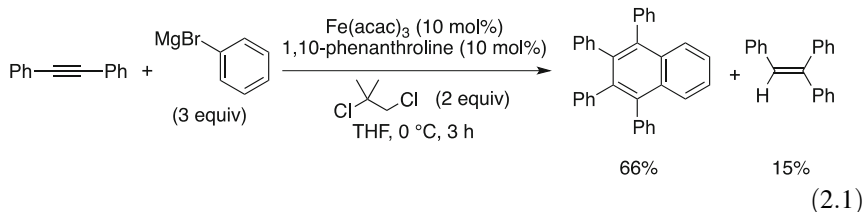
- Cr mediated Zeiss *J. Am. Chem. Soc.* **1959**  
(X = CrR<sub>2</sub>) Whitesides, Ehmman, *J. Am. Chem. Soc.* **1970**
- Pd cat. Sakakibara, *Chem. Lett.* **1986** (X = I); Heck *Organometallics* **1987** (X = I);  
Sato, Miura *J. Org. Chem.* **2003** (X = I); Wu, *Chem. —Eur. J.* **2008** (X = H)
- Ir cat. Sato, Miura *J. Am. Chem. Soc.* **2002** (X = COCl),  
*J. Org. Chem* **2007** (X = COOH),
- Rh cat. Sato, Miura *J. Org. Chem* **2008** (X = CR<sub>2</sub>OH),  
*Angew. Chem., Int. Ed.* **2008** (X = H), *Org. Lett.* **2009** (X = B(OH)<sub>2</sub>)

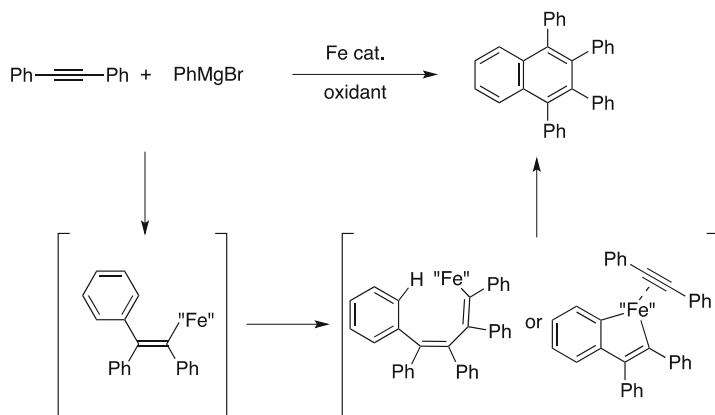
**Scheme 2.1** [2+2+2] annulation of alkyne via aromatic C–H bond activation



**Scheme 2.2** Strategy of iron-catalyzed alkyne annulation via C–H bond activation

carbometallation product. This naphthalene formation was attributed to the sequential insertion of two alkyne molecules followed by C–H bond activation induced by neighboring organoiron species or C–H bond activation of vinyliron intermediate followed by alkyne insertion (Scheme 2.3).





**Scheme 2.3** Iron-catalyzed naphthalene formation

## 2.3 Investigation of the Reaction Conditions

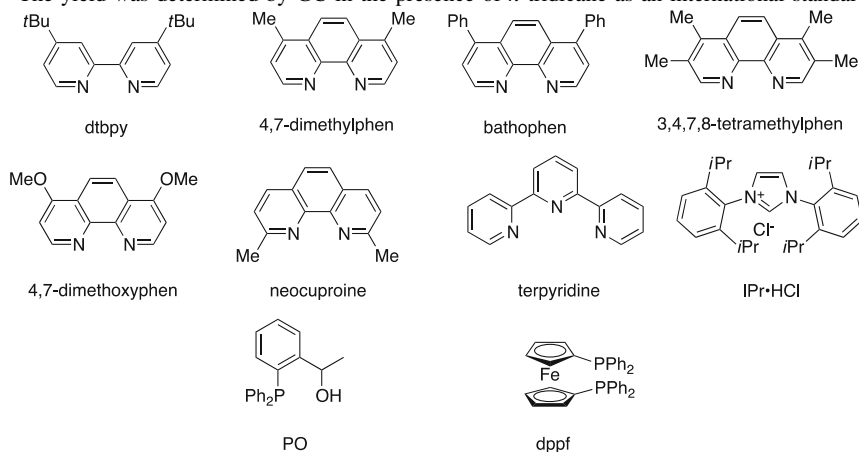
First, the effect of the ligand for this annulation reaction was investigated. The results are summarized in Table 2.1. With no ligand, the reaction only afforded the naphthalene product in 1 % yield and the major product was the product formed by carbometalation of the alkyne (**2**, entry 1). In contrast, the reaction afforded the annulation product **1** when bipyridine-type ligands were used (entries 2–8). Among the bipyridine-type ligands, 1,10-phenanthroline afforded the best result to give naphthalene **1** in 66 % yield (entry 4). This reaction is very sensitive to the substituents on the ligand: for example, phenyl or methyl substitution dramatically decreased the yield (entries 5–8). Substitution to the next position of the nitrogen atom totally suppressed the desired reaction, perhaps because of steric hindrance (entry 9). A tridentate pyridine ligand such as terpyridine also suppressed the reaction. *N*-Heterocyclic carbene-type ligands are often used in iron-catalyzed carbometalation reactions [23] or cross-coupling reactions [25, 26]; however, a carbene ligand did not affect the reaction (entry 11). Phosphine ligands such as hydroxyphosphine [27] or dppf were also examined, but these ligands had no effect on this reaction (entries 12 and 13).

After finding that phenanthroline was the best ligand, the effect of the amounts of reagents was investigated (Table 2.2). Without dihalide, naphthalene formation did not occur: only 2 % of desired product **1** was obtained and 72 % of the alkyne was recovered after the reaction (entry 5). In contrast, in the presence of dichloride, the conversion of alkyne increased and the naphthalene product was obtained in 68 % yield by using 3 equivalents of dichloride (entry 8). These results clearly suggest that the oxidant is necessary for this reaction and promotes catalytic turnover. Regarding the catalyst loading, 10 mol% of iron gave the best result and less catalyst or more decreased the reactivity (entries 10 and 11). The ratio of iron, alkyne, and Grignard reagent appears to be important for this reaction.

**Table 2.1** Effect of ligand on the iron-catalyzed naphthalene synthesis.

Entry	Ligand	Yield <sup>a</sup> (%)			Ph-Ph (equiv)
		1	2	Recovery	
1	None	1	21	63	0.23
2	2,2'-Bipyridine	12	19	55	0.31
3	dtbpy	23	20	39	0.32
4	phen	66	15	2	0.91
5	4,7-dimethylphen	56	19	6	0.55
6	bathopen	22	12	52	1.24
7	3,4,7,8-tetramethylphen	8	21	53	0.24
8	4,7-dimethoxyphen	3	21	63	0.23
9	neocuproine	0	6	90	0.43
10	terpyride	0	3	43	0.60
11	iPr·HCl	1	21	60	0.23
12	PO	1	24	59	0.24
13	dppf	2	24	61	0.24

<sup>a</sup> The yield was determined by GC in the presence of *n*-tridicane as an international standard



The screening of the amount of reagent clearly suggests the importance of the oxidant. Thus, the reaction was examined using other types of oxidant (Table 2.3). Simple 1,2-dichloroethane promoted the reaction and gave **1** in 36 % yield (entry 1). Other primary or secondary vicinal dichlorides such as 1,2-dichloropropane, 2,3-dichlorobutane, or 1,2-dichlorocyclohexane also promoted the reaction but not as efficiently as a tertiary dichloride, 1,2-dichloro-2-methylpropane (entry 5). The dichloride is considered to act as an oxidant by electron transfer to the iron center followed by  $\beta$ -elimination. The effect of the two methyls of the dichloride is not

**Table 2.2** Effect of the amount of reagents on the iron-catalyzed naphthalene synthesis.

$\text{Ph}-\text{C}\equiv\text{C}-\text{Ph} + \text{PhMgBr} \xrightarrow[\text{THF, 0 } ^\circ\text{C, 3 h}]{\begin{matrix} \text{Fe(acac)}_3 \text{ (z mol\%)} \\ \text{phen (z mol\%)} \\ \text{Cl-C(CH}_3)_2\text{-Cl (y equiv)} \end{matrix}} \text{Product 1} + \text{Product 2}$							
				<b>1</b>		<b>2</b>	
Entry	PhMgBr (x equiv)	Oxidant (y equiv)	Catalyst (Z mol %)	Yield (%) <sup>a</sup>			Ph-Ph (equiv)
				1	2	Recovery	
1	1	2	10	15	4	66	0.27
2	2	2	10	48	7	27	0.56
3	3	2	10	57	14	10	0.84
4	5	2	10	38	8	20	0.88
5	3	0	10	2	9	72	0.15
6	3	1	10	23	20	39	0.32
7	3	2	10	57	14	10	0.84
<b>8</b>	<b>3</b>	<b>3</b>	<b>10</b>	<b>68</b>	<b>10</b>	<b>6</b>	<b>0.89</b>
9	3	5	10	62	8	7	0.90
10	3	3	5	39	11	38	0.52
11	3	3	20	46	13	15	0.94

<sup>a</sup> The yield was determined by GC in the presence of *n*-tridicane as an international standard

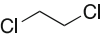
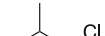
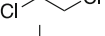
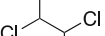

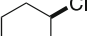
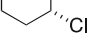

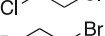
clear, but they might stabilize the radical intermediate generated in the electron transfer step or increase the rate of  $\beta$ -elimination by steric hindrance. Bromide or iodide instead of chloride did not work in this reaction because of the fast homodimerization of the Grignard reagent (entries 6 and 7) [28, 29]. Other types of oxidant such as diphenylsulfone or NMO did not promote this reaction (entries 8 and 9).

In the previous study of iron-catalyzed C–H bond activation reactions, a solvent effect was sometimes observed as a critical factor [19, 20]. Therefore, the effect of the cosolvent for this reaction was investigated (Table 2.4). In the previous study, aromatic cosolvents stabilized organoiron species efficiently. However, in this reaction, aromatic cosolvents such as benzene and toluene slightly suppressed the conversion (entries 2 and 3). The coordination of the aromatic cosolvent probably competes with the alkyne coordination to iron. The reaction was sluggish with nonpolar cosolvents such as hexane (entry 4). The slightly polar cosolvent dichloromethane reacted with the Grignard reagent in the presence of iron and totally suppressed the desired reaction (entry 5). The reaction in the diethylether gave **1** in slightly lower yield than the reaction in THF (entry 6). Regarding the solvent, pure THF was the best choice for this reaction (entry 1).

According to the results of the effect of reagent amount (Table 2.2) and the previous study of the iron-catalyzed C–H bond activation reaction with Grignard reagents [19, 20], control of the concentration of the Grignard reagent is quite important to suppress the undesired homodimerization of the Grignard reagent.

**Table 2.3** Effect of oxidant on the iron-catalyzed naphthalene synthesis.

$\text{Ph}-\text{C}\equiv\text{C}-\text{Ph} + \text{PhMgBr} \xrightarrow[\text{THF, 0 } ^\circ\text{C, 1 h}]{\text{Fe(acac)}_3 \text{ (10 mol\%)} \atop \text{1,10-phenanthroline (10 mol\%)} \atop \text{oxidant (3 equiv)}} \text{1} + \text{2}$

Entry	Oxidant	Yield (%) <sup>a</sup>			Ph-Ph (equiv)
		1	2	Recovery	
1		36	12	25	0.61
2		45	18	24	0.67
3		36	17	33	0.64
4		45	17	28	0.58
5		68	10	6	0.89
6		1	0	83	0.99
7		0	0	97	1.17
8		0	14	78	0.21
9		0	0	89	0.70

<sup>a</sup> The yield was determined by GC in the presence of *n*-tridicane as an international standard

**Table 2.4** Effect of cosolvent on the iron-catalyzed naphthalene synthesis.

$\text{Ph}-\text{C}\equiv\text{C}-\text{Ph} + \text{PhMgBr} \xrightarrow[\text{THF/cosolvent (1:1)}]{\text{Fe(acac)}_3 \text{ (10 mol\%)} \atop \text{1,10-phenanthroline (10 mol\%)} \atop \text{1,1-dichloro-2,2-dimethylpropane (3 equiv)}} \text{1} + \text{2}$

Entry	Cosolvent	Yield (%) <sup>a</sup>			Ph-Ph (equiv)
		1	2	Recovery	
1	None	68	10	6	0.80
2	Benzene	49	19	15	0.72
3	Toluene	40	19	67	0.70
4	Hexane	13	17	52	0.43
5	CH <sub>2</sub> Cl <sub>2</sub>	0	2	52	0.75
6	Et <sub>2</sub> O	47	20	15	0.80

<sup>a</sup> The yield were determined by GC using *n*-tridicane as an internal standard

**Table 2.5** Effect of addition rate of Grignard reagent on the iron-catalyzed naphthalene synthesis

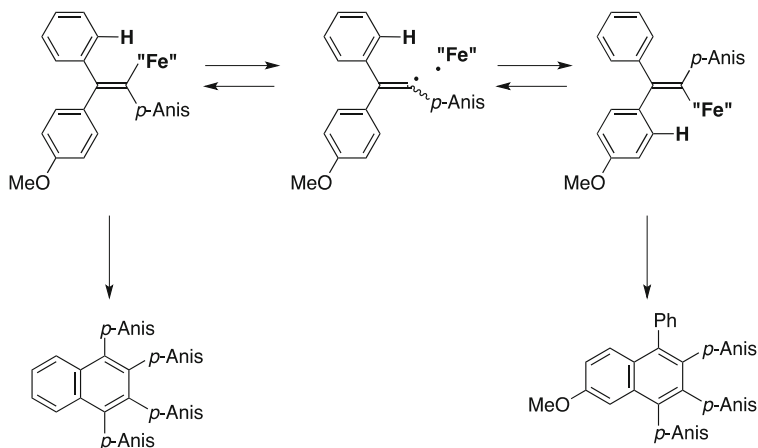
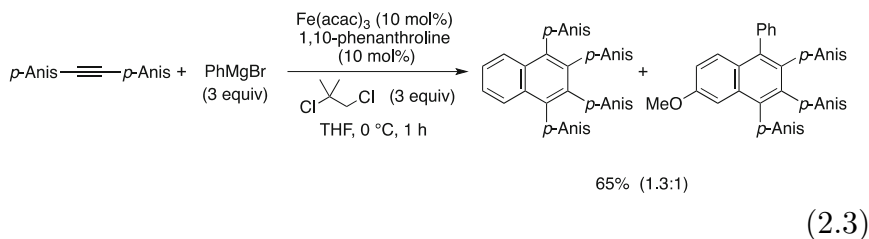
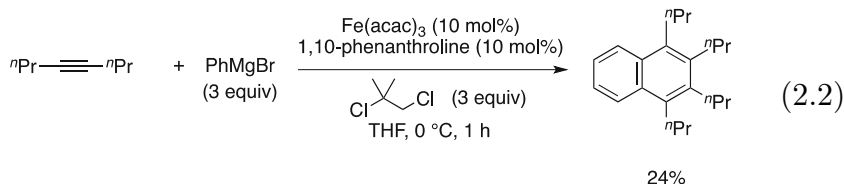
$\text{Ph}\text{---}\text{C}\equiv\text{C}\text{---}\text{Ph} + \text{PhMgBr} \xrightarrow[\text{THF, 0 } ^\circ\text{C, 3 h}]{\begin{array}{l} \text{Fe(acac)}_3 \text{ (10 mol\%)} \\ \text{1,10-phenanthroline (10 mol\%)} \\ \text{Cl-C(CH}_3)_2\text{-Cl (3 equiv)} \end{array}} \text{1} + \text{2}$ <p style="text-align: center;">(3 equiv) addition time</p>					
Entry	Addition time <sup>a</sup>	Yield (%) <sup>b</sup>			Ph–Ph (equiv)
		1	2	Recovery	
1	1 h	75	7	2	0.92
2	30 s	71	9	5	0.88
3	<2 s	44	22	16	0.81
4	0 <sup>c</sup>	2	25	57	0.38

<sup>a</sup> All reaction used a 15 mm Schlenk tube and stirred at 700 rpm<sup>b</sup> The yield were determined by GC using *n*-tridicane as an international standard<sup>c</sup> Catalyst was added after Grignard addition

Thus, the slow addition of the Grignard reagent was investigated (Table 2.5). The reaction afforded **1** in good yield when the Grignard reagent was added slowly to the reaction mixture (entry 1). On the contrary, fast addition decreased the yield of **1** (entry 3). In particular, the reaction did not proceed when the catalyst was added after addition of the Grignard reagent (entry 4). A high concentration of Grignard reagent may form iron-ate complexes [30, 31] and reduce reactivity.

## 2.4 Scope and Limitations

With these optimized conditions, the reaction of various alkynes and Grignard reagents was investigated. As shown in Eq. 2.2, the reaction of 4-octyne and phenylmagnesium bromide proceeded to give tetraalkylnaphthalene in 24 % yield. Although the reaction was sluggish compared with the diphenylacetylene case, this reaction is also applicable to alkyl alkynes. The reaction of di-*p*-methoxyphenylacetylene and phenylmagnesium bromide gave the naphthalene product as a two-isomer mixture (Eq. 2.3). This isomer formation was attributed to the *cis/trans* isomerization of a vinyliron intermediate. Without isomerization, annulation proceeded at the C–H bond of phenylmagnesium bromide to form the naphthalene product. After isomerization, C–H bond activation occurred on the aryl group of the alkyne (Scheme 2.4). Similar isomerization has been sometimes observed in palladium-catalyzed naphthalene synthesis [6].

**Scheme 2.4** Isomerization of the vinyliron intermediate

## 2.5 Summary

In this chapter, an iron-catalyzed naphthalene synthesis via C–H bond activation was developed. The reaction of internal alkynes and phenyl Grignard reagent proceeded in the presence of a catalytic amount of iron(III) acetylacetonate and 1,10-phenanthroline, and 1,2-dichloroalkane as an oxidant to give a 1,2,3,4-tetrasubstituted naphthalene. Although controlling the reaction selectivity is slightly difficult because of the isomerization of alkenyliron species, this result



demonstrates that iron catalysis can be applied to the alkyne annulation reaction via C–H bond activation.

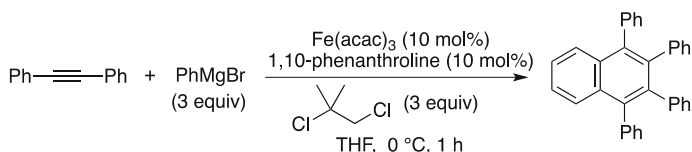
## 2.6 Experimental Part

### General

All the reactions dealing with air- or moisture-sensitive compounds were carried out in a dry reaction vessel under a positive pressure of argon or nitrogen. Air- and moisture-sensitive liquids and solutions were transferred via syringe or Teflon cannula. Analytical thin-layer chromatography was performed using glass plates pre-coated with 0.25 mm 230–400 mesh silica gel impregnated with a fluorescent indicator (254 nm). Thin layer chromatography plates were visualized by exposure to ultraviolet light (UV) and/or by iodine vapors, or immersion in an acidic staining solution of *p*-anisaldehyde followed by heating on a hot plate. Organic solutions were concentrated by rotary evaporation at *ca.* 15 Torr (evacuated with a diaphragm pump). Flash column chromatography was performed as described by Still et al. [32] employing Kanto Silica gel 60 (spherical, neutral, 140–325 mesh) or Wako activated alumina (*ca.* 300 mesh).

The melting points of solid materials were determined on a Mel-Temp II capillary melting-point apparatus and were uncorrected. Proton nuclear magnetic resonance ( $^1\text{H}$  NMR) and carbon nuclear magnetic resonance ( $^{13}\text{C}$  NMR) spectra were recorded with JEOL ECA-500 (500 MHz) NMR spectrometer. Chemical data for protons are reported in parts per million (ppm,  $\delta$  scale) downfield from tetramethylsilane and are referenced to the residual proton in the NMR solvent ( $\text{CDCl}_3$ ;  $\delta = 7.26$ ). Carbon nuclear magnetic resonance spectra ( $^{13}\text{C}$  NMR) were recorded at 125 MHz. Chemical data for carbons are reported in parts per million (ppm,  $\delta$  scale) downfield from tetramethylsilane and are referenced to the carbon resonance of the solvent ( $\text{CDCl}_3$ ;  $\delta = 77.0$ ). The data is presented as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet and/or multiplet resonances, br = broad), coupling constant in Hertz (Hz), and integration. Mass spectra (GC MS) are taken at SHIMADZU Parvum 2 gas chromatograph mass spectrometer.

Commercial reagents were purchased from Tokyo Kasei Co., Aldrich Inc., and other commercial suppliers and were used without further purification, unless mentioned. The Grignard reagents were prepared from the corresponding bromide and magnesium turnings in anhydrous diethyl ether or tetrahydrofuran (THF), and were titrated prior to use.  $\text{Fe}(\text{acac})_3$  (99.9 + %) was purchased from Aldrich Inc. and used as received. Anhydrous THF and diethyl ether were purchased from Kanto Chemical Co. and purified by a solvent purification system (GlassContour) equipped with columns of activated alumina and copper catalyst prior to use [33]. The water content of the solvent was confirmed by a Karl-Fischer moisture titrator to be less than 20 ppm.

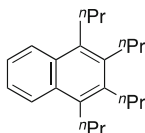
**Typical procedure: synthesis of 1,2,3,4-tetraphenyl naphthalene.**

In a flame-dried Schlenk tube were placed diphenylacetylene (75 mg, 0.42 mmol), 1,2-dichloro-2-methylpropane (150  $\mu$ L, 1.3 mmol) and 1.0 mL of THF. To this solution,  $\text{Fe}(\text{acac})_3$  and 1,10-phenanthroline in THF (0.8 mL, 0.5 M, 0.04 mmol) were added, followed by dropwise addition of phenylmagnesium bromide in THF (1.7 mL, 0.67 M, 1.3 mmol) over 1 h at 0  $^{\circ}\text{C}$ . The reaction mixture was stirred for an additional 1 h, then 1 mL of 1 M HCl (aq) and 5 mL of toluene were added. The resulting organic layer was separated and extracted with toluene (5 mL  $\times$  3). The combined organic layers were dried over sodium sulfate, and the volatiles were removed *in vacuo*. The remaining solid was purified by silica gel column chromatography (eluent: hexane/toluene = 20/1). The title compound was obtained as colorless solid (combined 155 mg, 71 % yield). The compound data were in good accordance with the literature.<sup>ivb</sup>

$^1\text{H}$ -NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.66–7.62 (m, 2H), 7.41–7.37 (m, 2H), 7.26–7.17 (m, 10H), 6.87–6.81 (m, 10H).

$^{13}\text{C}$ -NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  140.5, 139.5, 138.8, 138.4, 132.0, 131.3, 127.5, 126.9, 126.5, 126.4, 125.9, 125.3.

**1,2,3,4-tetrapropynaphthalene (Eq. 2.2):** obtained in 24 % yield as a colorless oil. The compound data were in good accordance with the literature.<sup>iva</sup>



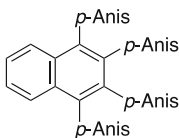
$^1\text{H}$ -NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.00–7.97 (m, 2H), 7.40–7.38 (m, 2H), 3.02–2.99 (m, 4H), 2.75–2.71 (m, 4H), 1.72–1.63 (m, 4H), 1.61–1.53 (m, 4H), 1.13–1.08 (m, 12H).

$^{13}\text{C}$ -NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  136.9, 134.1, 131.1, 124.5, 124.4, 32.6, 31.3, 24.9, 24.5, 15.1, 14.9.

GC MS (EI)  $m/z$  (relative intensity): 296 ( $\text{M}^+$ , 100), 267 (43), 225 (80), 195 (20), 183 (93), 165 (26).

**1,2,3,4-tetrakis(4-methoxyphenyl)naphthalene and 6-methoxy-2,3,4-tris(4-methoxyphenyl)-1-phenylnaphthalene (Eq. 2.3):** obtained in 65 % yield (of isomers (1.3:1 mixture of isomers by NMR) as a colorless solid. The analytical data were obtained from a pure fraction of silica gel column ( $\text{CH}_2\text{Cl}_2$ /toluene = 2/8).

**1,2,3,4-tetrakis(4-methoxyphenyl)naphthalene:** major isomer. The compound data were in good accordance with the literature.<sup>ivb</sup>



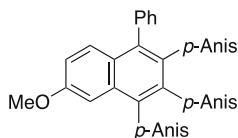
<sup>1</sup>H-NMR (500 MHz, CDCl<sub>3</sub>): δ 7.66–7.62 (m, 2H), 7.38–7.34 (m, 2H), 7.11–7.08 (m, 4H), 6.81–6.78 (m, 4H), 6.73–6.70 (m, 4H), 6.44–6.41 (m, 4H), 3.79 (s, 6H), 3.63 (s, 6H).

<sup>13</sup>C-NMR (125 MHz, CDCl<sub>3</sub>): δ 157.9, 156.8, 139.1, 138.1, 133.3, 132.33, 132.26, 132.23, 132.1, 126.9, 125.6, 113.0, 112.1, 55.1, 54.9.

GC MS (EI) *m/z* (relative intensity): 553 (42), 552 (M<sup>+</sup>, 100), 281 (8), 207 (11), 108 (6), 77 (4).

**6-methoxy-2,3,4-tris(4-methoxyphenyl)-1-phenylnaphthalene:** minor isomer.

Mp: 256.1–258.5 °C.



<sup>1</sup>H-NMR (500 MHz, CDCl<sub>3</sub>): δ 7.51 (d, *J* = 9.2 Hz, 1H), 7.24–7.22 (m, 2H), 7.19–7.16 (m, 3H), 7.11–7.09 (m, 2H), 7.03 (dd, *J* = 2.6, 9.2 Hz, 1H), 6.96 (d, *J* = 2.6 Hz, 1H), 6.80–6.78 (m, 2H), 6.72–6.69 (m, 4H), 6.44–6.39 (m, 4H), 3.79 (s, 3H), 3.70 (s, 3H), 3.63 (s, 3H), 3.61 (s, 3H).

<sup>13</sup>C-NMR (125 MHz, CDCl<sub>3</sub>): δ 157.8, 157.3, 156.8 (2C), 140.0, 139.6, 138.4, 137.1, 136.7, 133.5, 133.4, 133.2, 132.4 (2C), 132.25, 132.20 (2C), 132.17 (2C), 131.2 (2C), 128.6, 127.5 (2C), 127.4, 126.2, 117.7, 113.1, (2C), 112.1 (2C), 112.0 (2C), 105.6, 55.15, 55.07, 54.86, 54.84.

GC MS (EI) *m/z* (relative intensity): 553 (42), 552 (M<sup>+</sup>, 100), 281 (7), 207 (9), 108 (6), 77 (6).

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