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Hydrogen Peroxide Oxidation of Naphthalene Derivatives Catalyzed by Poly(bis-1,2-diphenylene) Diselenide

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Oxidation of 1- and 2-substituted naphthalenes (**1**) with 30% hydrogen peroxide in the presence of poly(bis-1,2-diphenylene) diselenide (PPDS) has been investigated. Depending on the substrate used *trans*-2-carboxycinnamic acid (**2**), and its isomer, (1-oxo-1,3-dihydroisobenzofuran-1-yl)acetic acid (**3**) or 2-naphthoic acid (**4b**) was a major product. Oxidation of hydroxynaphthalenes **1b** and **1c** is a convenient way to obtain *trans*-2-carboxy cinnamic acid (**2**) in almost quantitative yield. The mechanism of the reaction is postulated.

Key words: cinnamic acids, diselenides, hydrogen peroxide, hydroxynaphthalenes, oxidation

Oxidation of the organic compounds with hydrogen peroxide is a current problem of synthetic organic chemistry, because this oxidant is easily available, cheap, ecologically friendly and useful for a large-scale synthesis [1–4]. Unfortunately its activity toward many organic substrates is too low. For this purpose an oxygen-transfer agent, making oxidation more effective, must be used. Selenium compounds such as selenium(IV) oxide, areneseleninic acids, diaryl diselenides and benzisoselenazol-3(2H)-ones heve been reported during last three decades as efficient hydrogen peroxide catalysts [5–10].

Although several works on the oxidation of aromatic compounds with these reagents have been published recently, there are only one older work reporting oxidative transformations of the aromatic ring [5,11]. Our attention was focused on the cleavage of aromatic ring in naphthalene resulting in formation of 2-carboxycinnamic acid (**2**). The problem remains still open, since different authors having no spectroscopic evidence described the acid **2** as isomer *cis* or *trans* alternatively [12,13]. The reaction has a practical value, because ring substituted derivatives of cinnamic acid could found an application, like to industrially produced cinnamic acid [14]. Although it has been reported [11,15,16] that hydrogen peroxide oxidation of naphthalene or 2-hydroxynaphthalene without catalyst or in the presence of ammonium molybdate gave acid **2**, our attempts to repeat the reaction were unsatisfactory, since complex mixtures of several compounds were produced, making of desired

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product isolation troublesome. It raises the question about the results of hydrogen peroxide oxidation of different 1- or/and 2-substituted naphthalenes in the presence of poly(bis-1,2-diphenylene) diselenide (PPDS). We found that this compound was an effective catalyst for preparative oxidation of azomethine compounds (oximes, azines and tosylhydrazones) into arenecarboxylic acids [7].

RESULTS AND DISCUSSION

In this paper we report the results of our studies on the oxidation of mono- and 1,2-disubstituted naphthalenes with hydroxy, methoxy, formyl, acetyl and azomethine groups. An oxidant was 30% hydrogen peroxide and the catalyst (used in 5% mol) was PPDS. The reaction was carried out in tetrahydrofuran under reflux for 20 h. The reaction proceeded according to Scheme 1. Depending on the substrate used, *trans*-2-carboxycinnamic acid (**2**) and/or its isomer (1-oxo-1,3-dihydroisobenzofuran-1-yl)acetic acid (**3**) were formed as the major products accompanied with naphthoic acid **4**. In some cases, from azomethine derivatives the parent carbonyl compounds were formed. The results are presented in Scheme 1 and Table 1.

Scheme 1

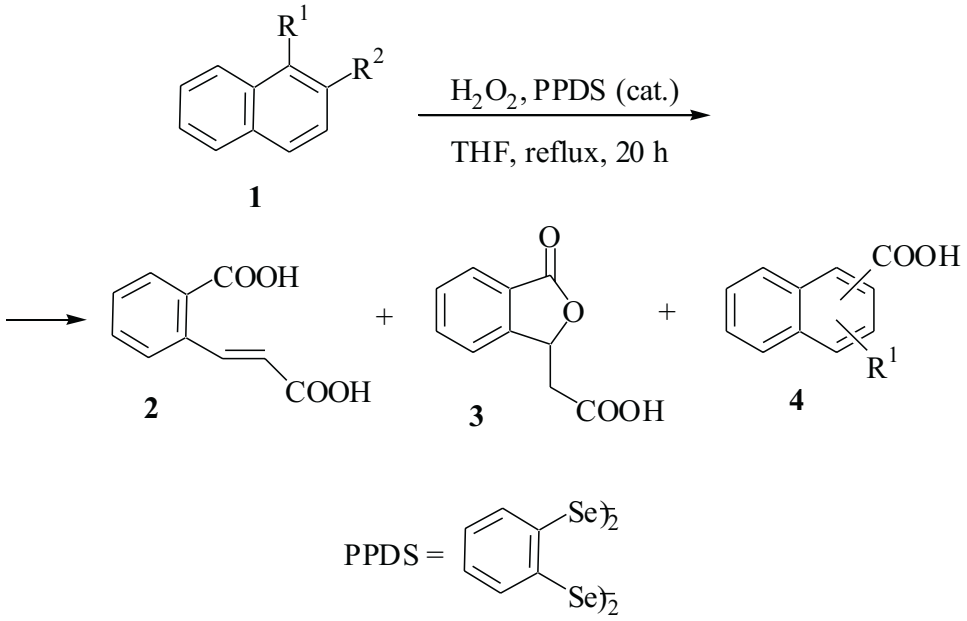


Table 1. Results of the oxidation of naphthalene derivatives by H_2O_2–PPDS system.

	Substrate		Products, yield [%][a]			
1	R^1	R^2	**1**	**2**	**3**	**4**
a	H	H	97	–	–	–
b	OH	H	–	98	–	–
c	H	OH	–	98	–	–
d	1,2-Naphthoquinone		–	97[b]	–	–
			–	60[b,c]	–	–
e	OMe	H	80	20	–	–
f	H	OMe	40[d]	(23)	(35)	–
g	CHO	H	–	50	(20)	(30)[e]
h	CH=NOH	H	–	48	(32)	(20)[e]
i	CH=NN=CH-(1-naphthyl)	H	–	50	(25)	(25)[e]
j	CH=NNHTS	H	–	44	(36)	(7)[e]
k	$CH=NNHCONH_2$	H	92	–	–	–
l	Ac	H	(72)[f]	5.6	(1.5)	(5.3)[e]
m	$CH_3C=NNHTs$	H	–[g]	21[d]	(32)	(33)[e]
n	CHO	OH	–	57	(38)	–
o	CH=NNHTs	OH	–	54	(44)	–
p	H	CHO	–	–	–	98[h]
q	H	CH=NNHTs	–	–	–	98[h]
r	OH	CHO	–	66	(33)	–
s	OH	CH=NNHTs	–	63	(37)	–
t	H	Ac	69	4.5	(1.5)	(11)[h]
u	H	$CH_3C=NNHTs$	–[d,j]	–	(29)	(49)[h]
v	OMe	CHO	–	32	48	(17)[i]
w	OMe	CH=NNHTs	–	(10)	81	(8.1)[i]
x	NO	OH	–	(45)	(20)	–

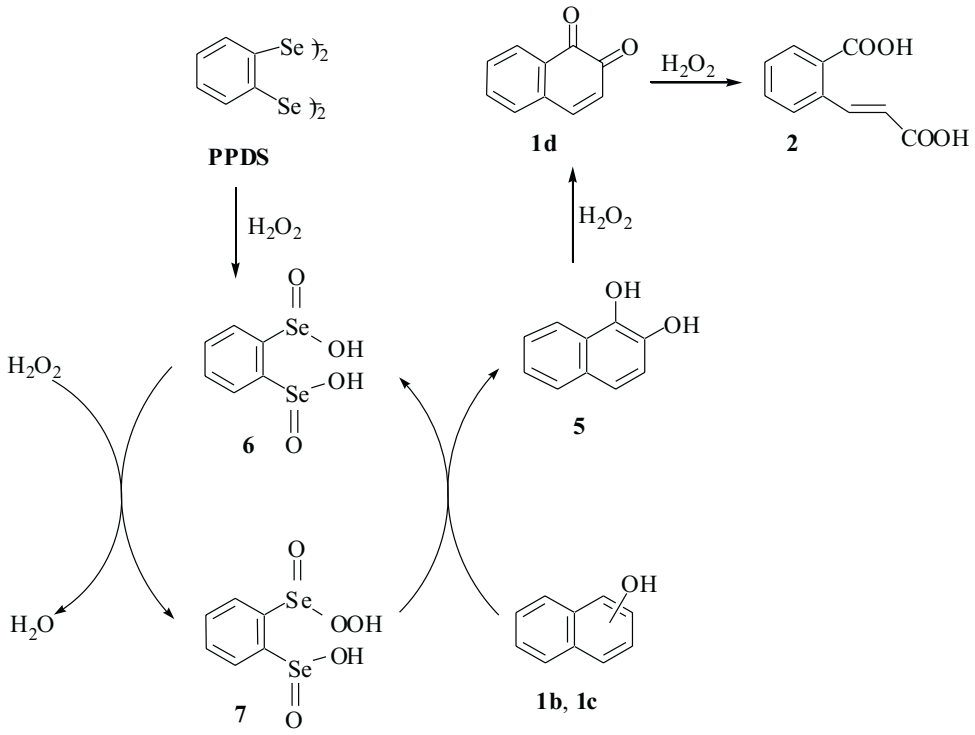
[a] Preparative yield. Data in parentheses are referred to yield determined by ^{1}H NMR and/or by GC. [b] Reaction carried out in room temperature. [c] Without catalyst. [d] After 70 h. [e] 1-Naphthoic acid (**4a**). [f] Accompanied with 1-naphthyl acetate (15% yield). [g] 1-Acetonaphthalene was formed in 15% yield. [h] 2-Naphthoic acid (**4b**). [i] 1-Methoxy-2-naphthoic acid (**4c**). [j] 2-Acetonaphthone was formed in 20% yield.

It has been found, that aromatic ring of unsubstituted naphthalene remains resistant toward oxidation, both in the absence and presence of the catalyst. Even when aromatic system is activated by the presence of hydroxyl group, such as in the 1- and 2-hydroxynaphthalenes, (**1b**, **1c**) the reaction was not observed when hydrogen peroxide was used without catalyst. On the contrary, when catalyst was added, the reaction proceeds smoothly and both naphthols were almost quantitatively converted into acid **2**. This makes the role of catalyst evident. Moreover, although in all experiments the substrate was used in 5 mmol amount, the oxidation of **1b** and **1c** carried out in 0.1 molar scale gave similar results. Since the isolation of acid **2** is easy and the yield of the product is substantially higher than that reported earlier [11,15,16], the reaction has a practical value as a simple and efficient method for preparation of *trans*-2-carboxycinnamic acid (**2**) from cheap hydroxynaphthalenes.

It seems to be possible that the reaction proceeds according to Scheme 2. The first steps are hydroxylation of the aromatic ring and oxidation of intermediate 1,2-dihydroxynaphthalene (**5**) to 1,2-napthoquinone (**1d**) which finally is converted to acid **2**. Additional experiments have shown, that the quinone **1d** is susceptible for oxidation by hydrogen peroxide, even in the mild reaction conditions without the catalyst.

Replacement of hydroxy group in naphthols by methoxy group made the methoxynapthalenes (**1e**, **1f**) less reactive. The acid **2** was formed only in low yields, while the substantial amounts of the substrate remained unreacted. It is consistent with a general rule, that quinones are formed substantially easier from phenols than from their ethers [17]. The role of the catalyst can be explained according to the mechanism postulated earlier for other diaryl diselenides [17,18]. In our case PPDS oxidized with hydrogen peroxide produces areneseleninic acid **6**, which is subsequently oxidized to peroxyseleninic acid **7** being the active intermediate.

Scheme 2

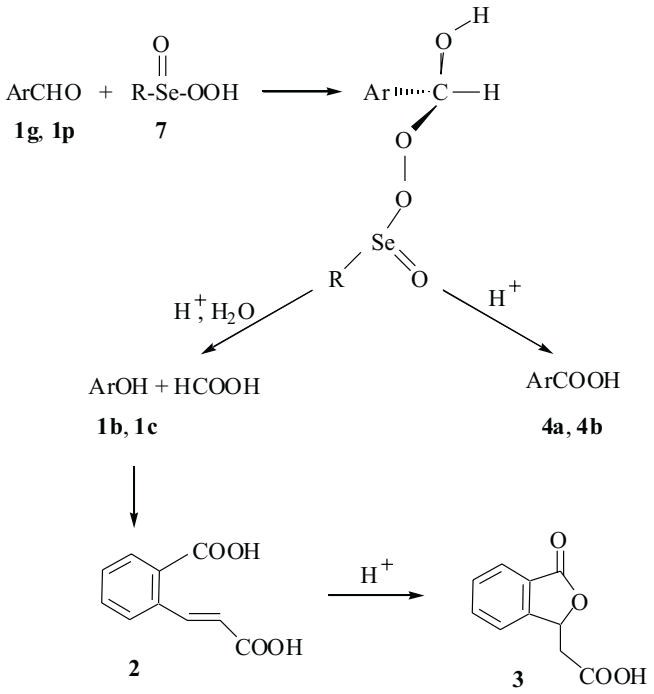


The results of the oxidation of 1-naphthaldehyde (**1g**) and its azomethine derivatives, such as oxime **1h**, azine **1i** and tosylhydrazone **1j**, were similar and only semicarbazone **1k** remained unreacted. 2-Carboxycinnamic acid (**2**), being the major product formed from **1g**–**1j** in 44–50%, was accompanied with 1-naphthoic acid (**4a**) (7–30%) and (1-oxo-1,3-dihydroisobenzofuran-1-yl)acetic acid (**3**) (20–36%). The oxidation of 1-acetonaphthone (**1l**) and its tosylhydrazone (**1m**) was less effective than oxidation of aldehyde **1g** and its derivatives **1h**–**j**, nevertheless, the same compounds **2**, **3** and **4a** were identified as the reaction products. When 2-hydroxy-1-naphthaldehyde (**1n**) and its tosylhydrazone (**1o**) were oxidized, only acid **2** and its isomer **3** were produced.

2-Naphthaldehyde (**1p**) and its tosylhydrazone (**1q**) were oxidized to 2-naphthalenecarboxylic acid (**4b**) exclusively, while oxidation of 1-hydroxy-2-naphthaldehyde (**1r**) and its tosylhydrazone (**1s**) gave both acids **2** and **3**. Other 2-substituted naphthalenes **1t**–**x** produced the mixtures of acids **2**, **3** and **4** in the ratio depending on the substrate used. Oxidation of 1-methoxy-2-napthaldehyde tosylhydrazone (**1w**) afforded lactone **3** in a high yield.

The reaction can be explained according to the mechanism presented in Scheme 3. It is known that hydroperoxide oxidation of aromatic aldehydes in the presence of selenium catalysts involves two competitive pathways, leading to arenecarboxylic acids or/and phenols [6,18,19]. When 1-naphthaldehyde (**1g**) is oxidized, one pathway leads directly to the 1-naphthoic acid (**4a**), while second one proceeds *via* Baeyer-Villiger rearrangement and the produced naphthol formate is directly hydrolyzed to 1-naphthol (**1b**), that is subsequently oxidized to 2-carboxycinnamic acid (**2**). In this case, the second pathway is preferred and acid **2** is produced in a higher yield than acid **4a**, especially when tosylhydrazone **1j** was used as a precursor of 1-naphthaldehyde (**1g**). On the contrast, oxidation of 2-naphthaldehyde (**1p**) and its tosylhydrazone (**1q**) proceeds directly to 2-napthoic acid (**4b**), Baeyer-Villiger rearrangement has not taken place and acid **2** is not formed. In all cases the same of diastereoisomer of acid **2** was produced. Its structure was established as *trans*, because the high coupling constant, J = 16.0 Hz was observed for alkenyl protons, similarly as it has been previously reported [13].

Scheme 3



2-Carboxycinnamic acid (**2**), in the protic medium, undergoes isomerization to (1-oxo-1,3-dihydroisobenzofuran-1-yl)acetic acid (**3**) by intramolecular Michael addition as it has been reported earlier [20]. Concluding, we can say that oxidation of 1- or 2-hydroxynaphthalenes with hydrogen peroxide in the presence of PPDS as a catalyst is a convenient way for synthesis of *trans*-2-carboxycinnamic acid (**2**). The results are similar for both of the substrates, since the reaction proceeds *via* intermediate 1,2-naphthoquinone. When naphthalenes having methoxy, formyl, acetyl and azomethine groups are oxidized, *trans*-2-carboxycinnamic acid (**2**) and/or other acids **3** and **4** are produced, depending on the character of substituent and its position. The results can be explained according to the mechanism involving competitive transformations of the carbonyl group into carboxy or hydroxy group.

EXPERIMENTAL

Melting points were determined with a Digital Melting Point Apparatus Electrothermal IA 9100. ^{1}H NMR spectra were recorded in $CDCl_3$ or DMSO-d_6 on a Bruker DRX 300 (300 MHz) spectrometer. Chemical shifts (δ) are reported in ppm with Me_4Si as internal standard. THF was distilled with $LiAlH_4$ and redistilled before using. TMEDA was distilled from NaOH before using. Naphthalene (**1a**), 1-hydroxynaphthalene (**1b**), 2-hydroxynaphthalene (**1c**), 1,2-naphthoquinone (**1d**), 2-methoxynaphthalene (**1f**), 1-formylnaphthalene (**1g**), 1-acetonaphthone (**1l**), 2-hydroxy-1-naphthaldehyde (**1n**), 2-formylnaphthalene (**1p**), 2-acetonaphthone (**1t**), 2-hydroxy-1-nitrosonaphthalene (**1x**), hydrogen peroxide, TMEDA, DMF, *n*-butyllithium and solvents were purchased from Aldrich or Merck. Semicarbazone **1k** was obtained by treating of 1-formylnaphthalene (**1g**) with $H_2NCONHNH_2$ in methanol. 1-Methoxynaphthalene (**1e**) was prepared by methylation of 1-naphthol (**1b**) with Me_2SO_4. 2-Formyl-1-methoxynaphthalene (**1v**) was prepared by *n*-butyllithium – TMEDA metalation of 1-methoxynaphthalene (**1e**) according to Shirley methodology [21] reinvestigated by Mannschreck *et al*. [22], Schlosser *et al*. [23] and Betz *et al.* [24]. Formed *in situ* 2-lithium-1-methoxynaphthalene was treated by DMF.

2-Formyl-1-hydroxynaphthalene (**1r**) was prepared by heating of ether **1v** with $AlCl_3$ as described in ref. [25]; ^{1}H NMR ($CDCl_3$): 12.67 (s, 1H, OH), 9.97 (s, 1H, CHO), 8.44 (dd, 1H, J = 8.2, 1.2 Hz, H-8), 7.79 (dd, 1H, J = 8.2, 1.2 Hz, H-5), 7.66 (ddd, 1H, J = 8.2, 6.9, 1.2 Hz, H-6 or H-7), 7.55 (ddd, 1H, J = 8.2, 6.9, 1.2 Hz, H-6 or H-7), 7.51 (d, 1H, J = 8.6 Hz, H-3), 7.37 (d, 1H, J = 8.6 Hz, H-4). Poly(bis-1,2-diphenylene) diselenide (PPDS), aldoxime **1h** and aldazine **1i** derived from 1-naphthaldehyde (**1g**) and tosylhydrazones **1j**,**m**,**o**,**q**,**s**,**u**,**w** were prepared as reported earlier [7,10]. The previously unknown compounds **1o**,**s** and **1w** were fully characterized.

2-Hydroxy-1-naphthaldehyde tosylhydrazone (1o): Colorless prisms; yield 96%, m.p. 173–174°C (methanol). ^{1}H NMR ($CDCl_3$): 11.46 (s, 1H, OH), 8.93 (s, 1H, CH=N), 8.12 (s, 1H, TsNH), 7.85–7.90 (m, 3H, ArH and TsH), 7.70–7.80 (m, 2H, ArH), 7.49 (t, J = 7.1 Hz, 1H, ArH), 7.30–7.40 (m, 3H, ArH and TsH), 7.14 (d, J = 9.1 Hz, 1H, ArH), 2.40 (s, 3H, Me). Anal. calcd. for $C_{18}H_{16}N_2O_3S$ (340.40): C, 63.51; H, 4.74; N, 8.23%. Found: C, 63.40; H, 4.95; N, 8.35%.

1-Hydroxy-2-naphthaldehyde tosylhydrazone (1s): Yellow crystals; yield 88%, m.p. 172–173°C (methanol). ^{1}H NMR ($CDCl_3$): 11.23 (s, 1H, OH), 8.35 (dd, J = 8.4, 1.5 Hz, 1H, ArH), 8.11 (s, 1H, CH=N), 7.99 (s, 1H, TsNH), 7.88 (d, J = 8.2 Hz, 2H, TsH), 7.28 (dd, J = 8.4, 1.5 Hz, 1H, ArH), 7.53 (ddd, J = 8.4, 5.9, 1.5 Hz, 1H, ArH), 7.48 (ddd, J = 8.4, 5.9, 1.5 Hz, 1H, ArH), 7.34 (d, J = 8.2 Hz, 2H, TsH), 7.29 (d, J = 8.5 Hz, 1H, ArH), 7.13 (d, J = 8.5 Hz, 1H, ArH), 2.40 (s, 3H, Me). Anal. calcd. for $C_{18}H_{16}N_2O_3S$ (340.40): C, 63.51; H, 4.74; N, 8.23%. Found: C, 63.60; H, 4.58; N, 8.19%.

1-Methoxy-2-naphthaldehyde tosylhydrazone (1w): Colorless needles; yield 92%, m.p. 131–132°C (ethyl acetate). ^{1}H NMR ($CDCl_3$): 8.28 (s, 1H, CH=N), 8.05–8.15 (m, 2H, ArH and TsNH), 7.92 (d, J = 8.1

Hz, 2H, TsH), 7.90 (d, J = 8.7 Hz, 1H, ArH), 7.77–7.87 (m, 1H, ArH), 7.57 (d, J = 8.7 Hz, 1H, ArH), 7.46–7.56 (m, 2H, ArH), 7.32 (d, J = 8.1 Hz, 2H, TsH), 3.92 and 3.58 (s, 3H, ratio 93:7, OCH_3), 2.47 and 2.40 (s, 3H, ratio 7:93, Me). Anal. calcd. for $C_{19}H_{18}N_2O_3S$ (354.42): C, 64.39; H, 5.12; N, 7.92%. Found: C, 64.29; H, 5.20; N, 7.98%.

Preparation of 2-formyl-1-methoxynaphthalene (1v): A solution of N,N,N′,N′-tetramethylethylenediamine (TMEDA, 9.00 ml, 60.0 mmol) and 1.6 M hexane solution of *n*-butyllithium (37.5 ml, 60.0 mmol), and cyclohexane (60 ml) was cooled to 0°C and after 10 min. 1-methoxynaphthalene (7.26 ml, 50.0 mmol) was added. The reaction was continued at room temperature for 2 h. The dark-red mixture was cooled in the ice bath and DMF (4.6 ml, 60 mmol) was added. The resulting yellow solution was treated with 2.0 M hydrochloric acid (pH 1–2) and the products were extracted with diethyl ether (3×100 ml). The combined organic layers were washed with the solution of sodium hydrogen carbonate (2.5%) and sodium chloride (7.5%) in water, dried with Na_2SO_4 and concentrated *in vacuo*. The residue was adsorbed on silica gel and eluted with hexane – ethyl acetate in gradient (50:1 – 10:1) to give **1v** as a pale yellow oil containing 1-formyl-8-methoxynaphthalene as minor product. Crystallized from oil 2-formyl-1-methoxynaphthalene (**1v**) was filtered of, washed with pentane and dried to afford colorless prisms; 5.31 g (57%), m.p. 61–63°C (lit. [25] m.p. 58°C). ^{1}H NMR ($CDCl_3$): 10.62 (s, 1H, CHO), 8.27 (d, 1H, J = 8.1 Hz, ArH), 7.8–8.0 (m, 2H, ArH), 7.50–7.70 (m, 3H, ArH), 4.16 (s, 3H, CH_3). From the filtrate 1-formyl-8-methoxynaphthalene was isolated using column chromatography and silica gel using the eluent as above and recrystallized from ethyl acetate. White needles; 0.587 g (6.3%) m.p. 89–90.5°C (lit. [25] m.p. 91–92°C). ^{1}H NMR ($CDCl_3$): 11.11 (s, 1H, CHO), 7.90–8.10 (m, 2H, ArH), 7.40–7.60 (m, 3H, ArH), 7.00 (d, 1H, J = 7.2 Hz, ArH), 4.03 (s, 3H, CH_3O).

Oxidation of naphthalenes, general procedure: The mixture of naphthalene **1a**–**x** (5.0 mmol), tetrahydrofuran (20 ml), 30% hydrogen peroxide (5.0 ml, 50 mmol) and poly(bis-1,2-diphenylene) diselenide (0.25 mmol, 59 mg, 5.0 mol %) was magnetically stirred under gentle reflux for 20 h. Then the mixture was treated with a pinch of Pt/C (15 min) and the solution of sodium hydrogen carbonate (2.5 g) and NaCl (7.5 g) in water (75 ml) was added stepwise, until evolution of carbon dioxide ceased. The solution was washed with diethyl ether (50 ml and 2×25 ml) and the aqueous layer was acidified with concentrated hydrochloric acid to pH 1–2, then the carboxylic acids were extracted with diethyl ether to solid vanished. The extract was dried over anhydrous Na_2SO_4, the solvent was distilled off and the residue was washed with CH_2Cl_2 (3×2 ml) to give pure *trans*-2-carboxycinnamic acid (**2**). M.p. 204–205°C, (lit. [13] m.p. 190–191°C). ^{1}H NMR (DMSO-d_6): 12.9 (s, 2H, COOH), 8.29 (d, J = 16.0 Hz, 1H, CH), 7.86 (d, J = 6.9 Hz, 1H, ArH), 7.81 (d, J = 7.7 Hz, 1H, ArH), 7.58 (dd, J = 6.9 Hz, 1H, ArH), 7.49 (dd, J = 7.7, 6.9 Hz, 1H, ArH), 6.40 (d, J = 16.0 Hz, 1H, CH).

Lactone **3** and 1-methoxy-2-naphthoic acid (**4c**) were isolated from the dichloromethane solution by column chromatography on silica gel and identified by comparison of their IR and ^{1}H NMR spectra with these reported in ref. [7] and [24] respectively.

Oxidation of hydroxynaphthalenes in preparative scale: Poly(bis-1,2-diphenylene) diselenide (1.17 g, 5.0 mmol) was suspended in a stirred, and heated to 50°C solution of hydroxynaphthalene **1b** or **1c** (14.4 g, 0.1 mol) in THF (250 ml). To this suspension 30% aqueous hydrogen peroxide (60 ml, 0.60 mol) was added dropwise during 1 h and the reaction was continued, under gently reflux for 20 h. After the reaction was finished most of the solvent (150 ml) was distilled off by using short vacuum-jacked column. The acid **2** crystallized directly after cooling of the residue in 60% and 70% yield respectively and was filtered off. The filtrate was treated with a pinch of Pt/C and additional amounts of acid **2** (36% and 26% respectively) were isolated as in the small scale.

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