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Anodic Oxidation of Methylnaphthalenes and 9-Methylanthracene

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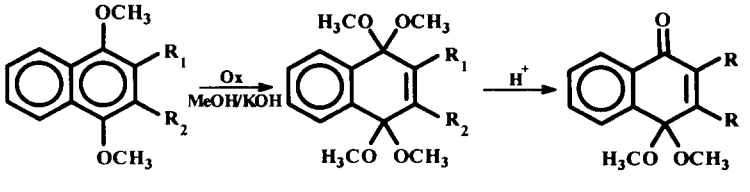
Abstract: Anodic methoxylation of a series of methylnaphthalenes (1-methylnaphthalene, 2-methylnaphthalene, 1,3-dimethylnaphthalene, 1,4-dimethylnaphthalene, 1,5-dimethylnaphthalene, 2,6-dimethylnaphthalene, 2,3,5-trimethylnaphthalene) and 9-methylanthracene afforded a series of nuclear-addition products. The process of nuclear addition is only observed in an aromatic ring and no chain methoxylated products were obtained. A probable mechanism is provided. © 1997 Elsevier Science Ltd.

Recent studies in our laboratories have focused on the anodic methoxylation of methyl aromatic rings[1]. Employing this method there can be obtained side-chain substitution products and nuclear addition products such as the *cis/trans* cyclohexa-1,4-diene ketals, could be obtained from unactivated substrates.

Following our investigations on these compounds, we studied, in the same medium, the electrochemical oxidation of a number of methylnaphthalenes and the 9-methylanthracene.

Previously, a number of studies about the anodic oxidation of naphthalene systems have been carried out. Of particular interest are the studies of the anodic oxidation of 1,4-dimethoxynaphthalene derivatives[2] (Scheme 1) giving the respective bisketals and functionalized quinone monoketals.

Scheme 1

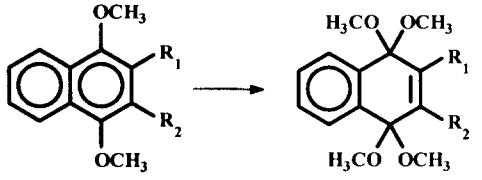


a, $R_1=R_2=H$ **c**, $R_1=H$, $R_2=Br$ **e**, $R_1=CH_3$, $R_2=SCH_3$

b, $R_1=H$, $R_2=CH_3$ **d**, $R_1=CH_3$, $R_2=Br$ **f**, $R_1=H$, $R_2=SCH_3$

Swenton also studied the anodic oxidation of substituted 1,4-dimethoxynaphthalenes obtaining the corresponding ketals[3]. Similar anodic oxidation of a series of oxygenated naphthalenes have been studied[4] giving the corresponding cyclohexadienic ketals (Scheme 2).

Scheme 2



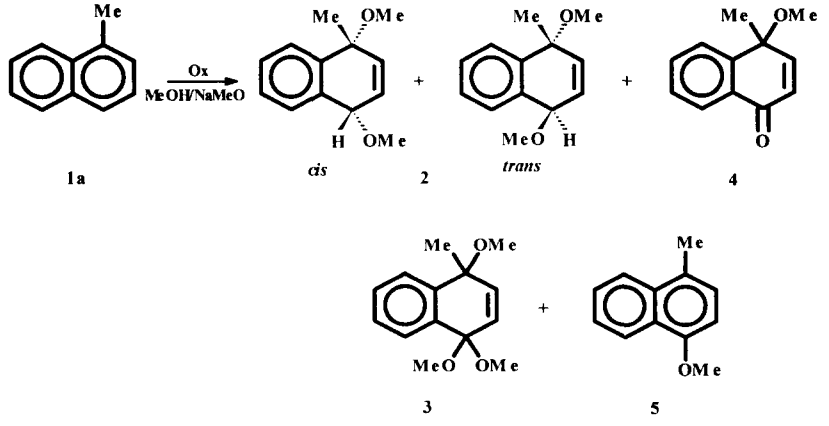
R_1	R_2
H	H
CH_3	H
OCH_3	H
CH_3	OCH_3
CH_3	$Si(CH_3)_3$

Anthracene derivatives have been studied and the anodic acetoxylation of 9,10-dimethylanthracene carried out a dimer compound derived from the radical cation or the 9,10-diacetate, depending on experimental conditions[5]. Anodic acetoxylation and methoxylation of anthracene, 9-methylanthracene and 9,10-dimethylanthracene lead to disubstituted 9,10-dihydroanthracenes[6].

RESULTS AND DISCUSSION

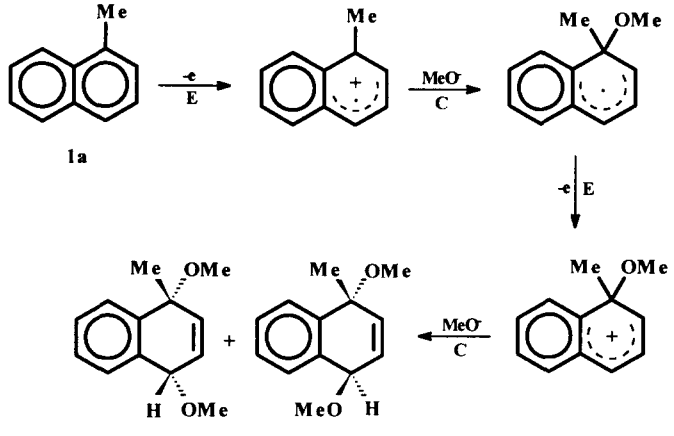
Anodic oxidation of 1-methylnaphthalene (**1a**) in methanol-sodium methoxide carried out under constant current intensity afforded a product **2** as the major product, a nuclear-addition product with larger methoxylation degree **3**, its hydrolysis product **4**[4] and 1-methoxy-4-methylnaphthalene **5**[4], obtained from **2** (Scheme 3).

Scheme 3



The compound **2** it is a mixture of *cis* and *trans* isomers, showing a higher abundance of the *trans*-isomer as compared with the *cis*-isomer (1.65 versus 1). The structures of *cis and trans* isomers were assigned by using ^{1}H NMR[1e, 1f]. For 1-methylnaphthalene **(1a)** the ECEC sequence explains formation of **2**, being C a chemical step and E an electrochemical step (Scheme 4).

Scheme 4



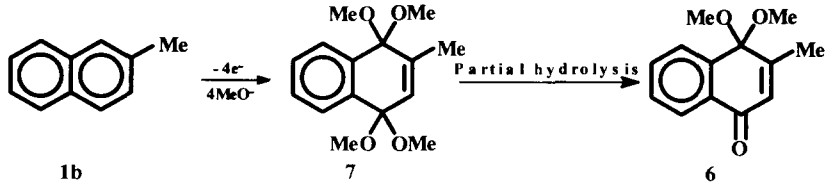
This mechanistic sequence is the same as that given in the 1,4-stereoselective addition to pseudocumene[1f].

It is note worthy that in the electrooxidation of all the methyl compounds studied, we have only observed the nuclear addition process in the aromatic ring bearing a methyl group. The lack of attack on the second ring might be a surface phenomenon that could be explained considering the geometric change in the molecule when the cyclohexadienic system is formed. Thus, the groups in the 1,4 positions are virtually situated on a plane perpendicular to the hexagonal ring[1e] and the second adsorption on the electrode surface could be more difficult. Furthermore, removing an electron fron a Π^* orbital of a methyl stabilized naphthalene **(1a)** is much easier than removing one from a disubstituted benzene ring.

Nuclear trimethoxylated product **3** is formed in a second sequence ECEC with **5** as substrate. Compound **5** has the aromatic nucleus activated by the methoxy group and rapidly leads to the nuclear-trimethoxylated product **3**, through another methoxylation process.

When the substrate was 2-methylnaphthalene **(1b)**, only one product **6** was obtained[7] (Scheme 5). The formation of **6** could be explained through an intermediate compound **7** not detected in the reaction medium. Compound **6** is formed in a regioselective partial hydrolysis at C-4.

Scheme 5

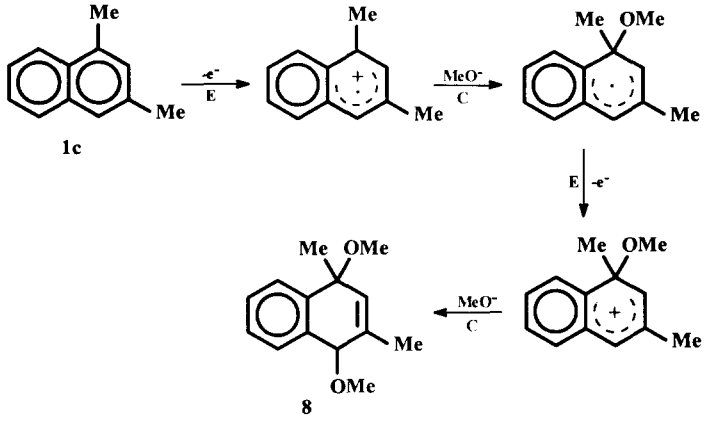


1b **7** **6**

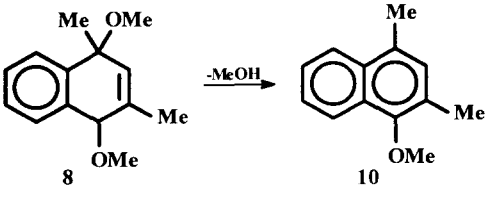
Anodic methoxylation of 1,3-dimethylnaphthalene (**1c**) afforded two products **8** and **9**. Product **8**[8] was obtained in a two electron process, but the major product **9** was obtained in a different process, four electrons in this case.

The formation of the nuclear dimethoxylated product **8** could be explained by a ECEC sequence (Scheme 6), but **8** is not very stable and would decompose to **10** (Scheme 7). In **10**, one ring is activated by a methoxy group, which stabilizes the initial cation-radical intermediate and rapidly leads to the nuclear-trimethoxylated product **9** through another methoxylation process (Scheme 8).

Scheme 6

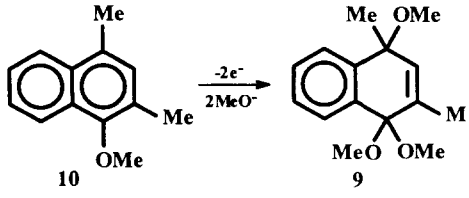


Scheme 7



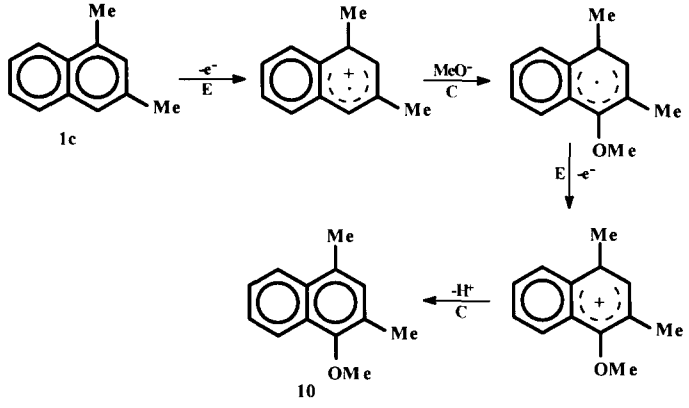
8 **10**

Scheme 8



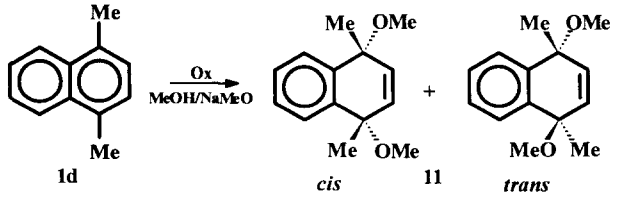
There is another path to account for the formation of **10**, which consists of a nuclear methoxylation of the substrate through a ECEC sequence (Scheme 9).

Scheme 9



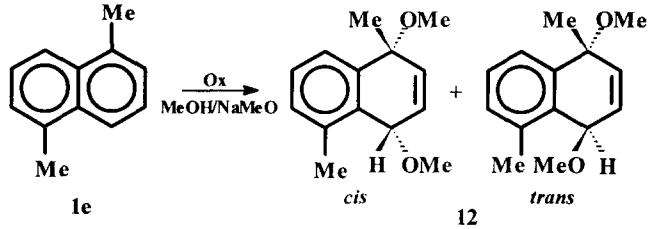
With 1,4-dimethylnaphthalene (1d), the anodic oxidation led to the two isomers *cis* and *trans* of 11 (Scheme 10). The *trans*-isomer shows a higher abundance as compared with the *cis*-isomer (3.8:1). Formation of compound **11** could be explained through the same sequence described for 1-methylnaphthalene.

Scheme 10

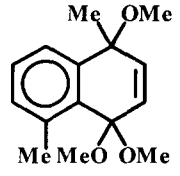


When the substrate was 1,5-dimethylnaphthalene (**1e**), the anodic methoxylation led to compounds **12** (*cis* and trans isomers) and **13** (Scheme 11). The formation of compounds **12** (*trans/cis* ratio = 6.4/1) could be explained through the same sequence previously described for the obtention of nuclear methoxylated products obtained in the anodic oxidation of 1-methylnaphthalene. Compound **13** is obtained in the same sequence previously described for compound **9**.

Scheme 11



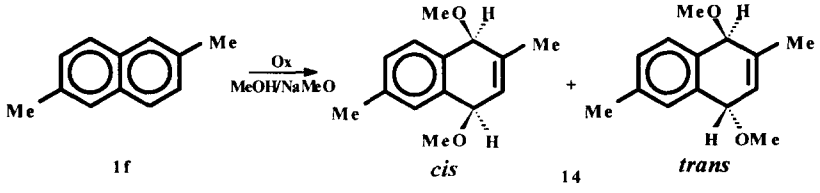
1e **12**



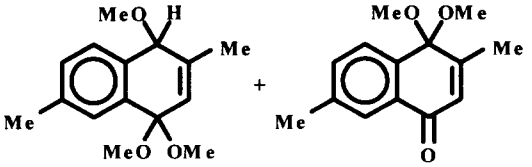
13

Anodic methoxylation of 2,6-dimethylnaphthalene (**1f**) afforded the dimethoxylated product **14** (*cis* and *trans* isomers), the trimethoxylated product **15** and two hydrolysis products **16** and **17**[9] (Scheme 12).

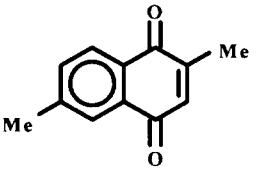
Scheme 12



1f **14**



15 16

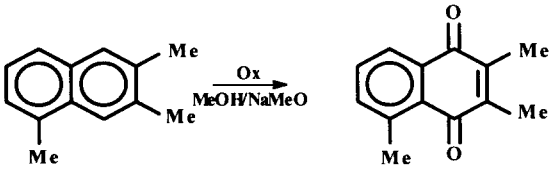


17

Formation of compounds **14** (*trans/cis* ratio = 2.3/1) and **15** could be explained through the same sequence previously described for 1,3-dimethylnaphthalene. Product **14** is obtained in a two electron process and the product **15** in a four electrons process.

When the substrate was 2,3,5-trimethylnaphthalene **(1g)** only one product **18** was obtained (Scheme 13). The formation of **18** could be explained through the same sequence previously described for 2-methylnaphthalene.

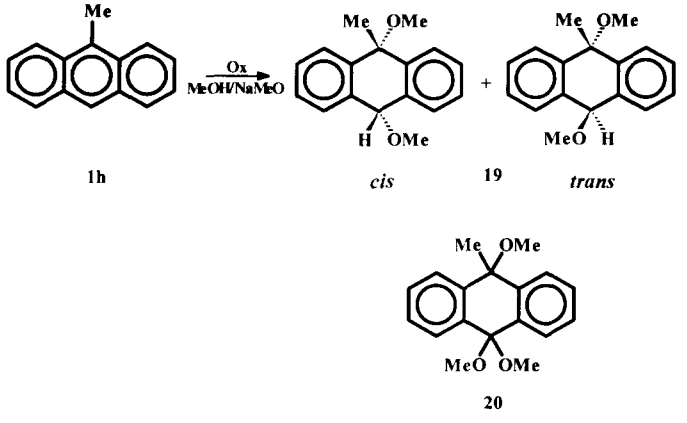
Scheme 13



1g 18

Anodic methoxylation of 9-methylanthracene **(1h)** afforded two nuclear addition products **19**[6,10] and **20** (Scheme 14). Product **19** (*cis* and *trans* isomers) is obtained in a two electron process and the product **20** in a four electrons process. The *trans/cis* ratio for **19** is 1.68/1.

Scheme 14



1h *cis* **19** *trans*

20

Formation of compounds **19** and **20** could be explained through the same sequence previously described for the obtention of the nuclear methoxylated products (**2** and **3** respectively) obtained in the anodic oxidation of 1-methylnaphthalene.

Only the *trans* isomer has been isolated by flash chromatography[11].

It is worthy to note that in the electrooxidation of all the alkyl-aromatics studied, we have only observed nuclear-addition products and not detected side-chain methoxylated products, always present in the alkylbenzenes previously studied. Side-chain oxidation could be occurring, but possibly the reactive intermediates lead to dimeric or polymeric material that was not characterized.

Table 1

initial substrate	reaction time, min	product (yield,%)[a]
1-methylnaphthalene (**1a**)	60	**2** (11)[b], **3** (2), **4** (2), **5** (3)
2-methylnaphthalene (**1b**)	120	**6** (23)
1,3-dimethylnaphthalene (**1c**)	45	**8** (8), **9** (44)
1,4-dimethylnaphthalene (**1d**)	45	**11** (14)[c]
1,5-dimethylnaphthalene (**1e**)	45	**12** (18)[d], **13** (6)
2,6-dimethylnaphthalene (**1f**)	45	**14** (10)[e], **15** (15), **16** (20) **17** (4)
2,3,5-trimethylnaphthalene (**1g**)	45	**18** (11)
9-methylanthracene (**1h**)	45	**19** (41)[f], **20** (13)

[a]Deduced by GC analysis using cyclohexanone as internal standard.

trans/cis ratio: 1.6/1[b], 3.8/1[c], 6.4/1[d], 2.3/1[e], 1.7/1[f].

EXPERIMENTAL SECTION

A generator with a maximum output of 60 V and 2 A was used. Nuclear magnetic resonance spectra were recorded at 60 Mz, the chemical shifts (δ values) are given in parts per million relative to TMS as an internal standard in $CDCl_3$ solutions, and coupling constants are reported in hertz. IR spectra are reported in cm^{-1}. Mass spectra were obtained at 70 eV (EI). GC analysis utilized a fused silica capillary column (25 m × 0.2 mm) using nitrogen as the carrier gas (2 mL/min) and was equipped with a flame ionization detector.

Electrolysis procedure. Electrolysis were carried out in cylindrical, water refrigerated cells without separate compartments. The temperature was controlled at 30°C, and stirring was magnetic. A carbon-paste plate was used as anode and a stainless-steel plate as cathode. Solvent/supporting electrolyte system was prepared by adding a mixture of metallic sodium (0.2 g) and sodium perchlorate (0.05 g + 0.15 g) to dry methanol (70 mL) for electrolysis of 2-methylnaphthalene and adding metallic sodium (0.2 g) to dry methanol (70 mL) for electrolysis in the other cases. Reactions with the initial substrate were carried out under constant current intensity of 1 A and an anodic density of 55 mA/cm^2, and the electron consumptions were 10.6 F/mol **(1a** and **1b)**, 14.6 F/mol **(1c**, **1d** and **1f)**, 10.2 F/mol **(1e)**, 9.4 F/mol **(1g)** and 18 F/mol **(1h)**.

The electrolysis was interrupted when all starting material was consumed (followed by GC); the reaction mixture was evaporated to dryness under reduced pressure; 100 mL of a buffer solution KH_2PO_4/Na_2HPO_4 (0.025 M, pH 7) was then added and the suspension was extracted with diethyl ether (3 × 30 mL). The extracts were dried (anhydrous Na_2SO_4), and the ether was evaporated at reduced pressure. Products were isolated by chromatography on a carbon-celite column (1:1) with ether as eluent for the extracts from the electrolysis of 1-methylnaphthalene, 1,3-dimethylnaphthalene and 9-methylanthracene; chromatography on silica gel column with n-hexane-ethyl acetate (90:10 v/v) as eluent for the extracts from 2-methylnaphthalene, n-hexane-ethyl acetate (98:2 v/v) for the extracts from 1,4-dimethylnaphthalene, 1,5-dimethylnaphthalene and 2,3,5-trimethylnaphthalene and n-hexane-ethyl acetate (95:5 v/v) for the extract from 2,6-dimethylnaphthalene. Yields were calculated from gas chromatography using cyclohexanone as the internal standard. In the anodic oxidations of **(1a)**, **(1d)**, **(1e)**, **(1f)** and **(1h)** the structures of *cis* and *trans* isomers were assigned by using ^1H NMR[1e,1f]

All compounds gave satisfactory (± 0.4% C and H) combustion analyses.

***cis*-and *trans*-1-Methyl-1,4-dimethoxy-1,4-dihydronaphthalene (2):** IR (film) 3060, 3020, 2980, 2920, 2820, 1090, 1070, 880, 750, 680 cm^{-1}; *cis isomer* ^1H NMR δ 7.60-7.20 (4H, m), 6.10 (2H, m), 5.00 (1H, m), 3.25 (3H, s), 3.00 (3H, s), 1.45 (3H, s); *trans isomer* ^1H NMR δ 7.60-7.20 (4H, m), 6.10 (2H, m), 5.00 (1H, m), 3.15 (3H, s), 2.90 (3H, s), 1.55 (3H, s); MS (EI) m/e (rel. intensity) 204 (M^+, 3), 190 (13), 189 (100), 173 (44), 158 (64), 141 (43), 115 (67), 77 (11), 63 (10). Anal. calcd for $C_{13}H_{16}O_2$: C, 76.47; H, 7.84. Found: C, 76.41; H, 7.79.

1-Methyl-1,4,4-trimethoxy-1,4-dihydronaphthalene (3): IR (film) 3060, 2980, 2920, 2820, 1070, 880, 760 cm^{-1}; ^1H NMR δ 7.60-7.20 (4H, m), 6.15 (2H, m), 3.00 (3H, s), 2.90 (3H, s), 2.85 (3H, s), 1.50 (3H, s); MS (EI) m/e (rel. intensity) 234 (M^+, 0.5), 203 (100), 188 (33), 173 (24), 172 (28), 157 (11), 128 (35), 115 (15). Anal. calcd for

$C_{14}H_{18}O_3$: C, 71.80; H, 7.69. Found: C, 71.75; H, 7.64.

1,3-Dimethyl-1,4-dimethoxy-1,4-dihydronaphthalene (8): IR (film) 3060, 2980, 2920, 2820, 1600, 1060, 880 cm^{-1}; ^1H NMR δ 7.70-7.10 (4H, m), 5.90 (1H, m), 4.85 (1H, m), 3.00 (3H, s), 2.90 (3H, s), 1.95 (3H, d, *J*=1.5 Hz), 1.50 (3H, s); MS (EI) m/e (rel. intensity) 218 (M^+, 0.2), 187 (100), 171 (50), 159 (37), 143 (39), 128 (93), 115 (75), 77 (20). Anal. calcd for $C_{14}H_{18}O_2$: C, 77.06; H, 8.26. Found: C, 77.14; H, 8.22.

1,3-Dimethyl-1,4,4-trimethoxy-1,4-dihydronaphthalene (9): IR (film) 3060, 2980,2920, 2820, 1600, 1070, 860, 760, 670 cm^{-1}; ^1H NMR δ 7.70-7.10 (4H, m), 5.95 (1H, m), 2.95 (3H, s), 2.90 (3H, s), 2.85 (3H, s), 1.90 (3H, d, *J*=1.5 Hz), 1.50 (3H, s); MS (EI) m/e (rel. intensity) 248 (M^+, 0.2), 233 (100), 217 (83), 202 (69), 187 (52), 186 (63), 171 (35), 159 (53), 128 (92), 115 (66), 89 (32). Anal. calcd for $C_{15}H_{20}O_3$: C, 72.58; H, 8.07. Found: C, 72.53; H, 8.01.

***cis*-and *trans*-1,4-Dimethyl-1,4-dimethoxy-1,4-dihydronaphthalene (11):** IR (film) 3020, 2980, 2910, 2810, 1080, 870, 680 cm^{-1}; *cis isomer* ^1H NMR δ 7.60-7.20 (4H, m), 5.95 (2H, s), 2.95 (6H, s), 1.45 (6H, s); *trans isomer* ^1H NMR δ 7.45 (4H, m), 5.90 (2H, s), 2.85 (6H, s), 1.50 (6H, s); MS (EI) m/e (rel. intensity) 218 (M^+, 0.2), 203 (100), 188 (30), 172 (64), 128 (37), 115 (21). Calcd for $C_{14}H_{18}O_2$: C, 77.06; H, 8.26. Found: C, 77.13; H, 8.29.

***cis*-and *trans*-1,5-Dimethyl-1,4-dimethoxy-1,4-dihydronaphthalene (12):** IR (film) 3020, 2980, 2923, 2820, 1640, 1600, 1090, 840, 780 cm^{-1}; *cis isomer* ^1H NMR δ 7.60-7.20 (3H, m), 6.85 (1H, d, *J*=1.5 Hz), 6.43 (1H, d, *J*=1.5 Hz), 5.10 (1H, s), 3.14 (3H, s), 2.87 (3H, s), 2.45 (3H, s), 1.56 (3H, s); *trans isomer* ^1H NMR δ 7.30 (3H, m), 6.85 (1H, d, *J*=1.5 Hz), 6.43 (1H, d, *J*=1.5 Hz) 5.10 (1H, s), 3.02 (3H, s), 2.76 (3H, s), 2.47 (3H, s), 1.58 (3H, s); MS (EI) m/e (rel. intensity) 218 (M^+, 2), 203 (100), 188 (17), 156 (14), 128 (33), 115 (21). Calcd for $C_{14}H_{18}O_2$: C, 77.06; H, 8.26. Found: C, 77.11; H, 8.28.

1,5-Dimethyl-1,4,4-trimethoxy-1,4-dihydronaphthalene (13): IR (film) 3020, 2980, 2920, 2820, 1640, 1600, 1090, 840, 780 cm^{-1}; ^1H NMR δ 7.60-7.20 (3H, m), 6.88 (1H, d, *J*=1.5 Hz), 6.48 (1H, d, *J*=1.5 Hz), 3.10 (3H, s), 2.95 (3H, s), 2.85 (3H, s), 2.35 (3H, s), 1.55 (3H, s); MS (EI) m/e (rel. intensity) 248 (M^+, 0.5), 217 (100), 202 (32), 187 (32), 186 (35), 171 (23), 159 (18), 128 (45), 115 (40). Calcd for $C_{15}H_{20}O_3$: C, 78.58; H, 8.07. Found: C, 78.63; H, 8.12.

***cis*-and *trans*-2,6-dimethyl-1,4-dimethoxy-1,4-dihydronaphthalene (14):** IR (film) 3060, 3020, 2980, 2920, 2820, 1070, 750, 680 cm^{-1}; *cis isomer* ^1H NMR δ 7.40-7.20 (3H, m), 5.90 (1H, m), 4.80 (2H, m), 3.00 (3H, s), 2.80 (3H, s), 2.40 (3H, s), 1.90 (3H, d, *J*=1.5Hz); *trans isomer* ^1H NMR δ 7.40-7.20 (3H, m), 5.90 (1H, m), 4.80 (2H, m), 2.90 (3H, s), 2.70 (3H, s), 2.40 (3H, s), 1.90 (3H, d, *J*=1.5Hz); MS (EI) m/e (rel. intensity) 218 (M^+, 15), 187 (100), 172 (60), 141 (13), 115 (11). Calcd for $C_{14}H_{18}O_2$: C, 77.06; H, 8.26. Found: C, 76.95; H, 8.20.

2,6-Dimethyl-1,4,4-trimethoxy-1,4-dihydronaphthalene (15): IR (film) 3060, 2980, 2920, 2820, 1610, 1070, 880, 780 cm^{-1}; ^1H NMR δ 7.50-7.00 (3H, m), 6.00 (1H, m), 4.80 (1H, m), 2.95 (3H, s), 2.75 (3H, s), 2.65 (3H, s), 2.25 (3H, s), 1.70 (3H, m); MS (EI) m/e (rel. intensity) 248 (M^+, 1), 216 (52), 201 (100), 173 (18), 115 (12). Calcd for $C_{15}H_{20}O_3$: C, 72.58; H, 8.07. Found: C, 72.62; H, 8.11.

2,6-Dimethyl-1,1-dimethoxy-4(4H)naphthalene (16): IR (film) 3040, 2980, 2920, 2820, 1660, 1640, 1600, 1070, 880, 780 cm^{-1}; ^1H NMR δ 7.80-7.30 (3H, m), 6.40 (1H, m), 2.85 (6H, s), 2.45 (3H, s), 2.00 (3H, d, *J*=1.5 Hz); MS (EI) m/e (rel. intensity) 232 (M^+, 14), 217 (25), 202 (13), 201 (100), 173 (10), 115 (10). Calcd for $C_{14}H_{16}O_3$: C, 72.41; H, 6.90. Found: C, 72.10; H, 6.79.

2,3,5-Trimethyl-1,4-naphthoquinone (18): IR (film) 3015, 2925, 2855, 2820, 1715, 1690, 1675, 1600, 820 cm^{-1}; ^1H NMR δ 7.80-7.20 (3H, m), 2.66 (3H, s), 2.46 (3H, s), 2.43 (3H, s); MS (EI) m/e (rel. intensity) 200 (M^+, 0.2), 170 (100), 155 (69), 128 (38), 115 (26). Calcd for $C_{13}H_{12}O_2$: C, 78.00; H, 6.00. Found: C, 77.81; H, 6.10.

***trans*-9-Methyl-9,10-dimethoxy-9,10-dihydroanthracene (19):** IR (film) 3060, 3020, 2960, 2920, 2820, 1100, 1070, 760 cm^{-1}; *Trans isomer* ^1H NMR δ 7.70-7.30 (8H, m), 5.15 (1H, s), 3.15 (3H, s), 2.85 (3H, s), 1.80 (3H, s); MS (EI) m/e (rel. intensity) 254 (M^+, 0.2), 239 (14), 223 (34), 222 (79), 208 (39), 207 (31), 193 (100), 192 (72), 191 (42), 189 (23), 178 (28), 165 (41). Calcd for $C_{17}H_{18}O_2$: C, 80.32; H, 7.09. Found: C, 80.27; H, 6.95.

9-Methyl-9,10,10-trimethoxy-9,10-dihydroanthracene (20): IR (film) 3060, 3020, 2960, 2920, 2820, 1100, 1070, 760 cm^{-1}; ^1H NMR δ 7.70-7.30 (8H, m), 3.00 (3H, s), 2.90 (3H, s), 2.80 (3H, s), 1.65 (3H, s); MS (EI) m/e (rel. intensity) 284 (M^+, 1), 253 (100), 223 (81), 222 (64), 207 (90), 178 (58), 152 (34). Calcd for $C_{18}H_{20}O_3$: C, 76.06; H, 7.04. Found: C, 76.15; H, 7.12.

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8. Compound **8** has been analyzed through a GC-MS system, which showed a single chromatographic peak, therefore the anodic methoxylation of 1,3-dimethylnaphthalene has afforded only one isomer of 1,3-dimethyl-1,4-dimethoxy-1,4-dihydronaphthalene. The structure of this isomer not can be assigned by using ^{1}H NMR data.

9. "Dictionary of Organic Compounds", Chapman and Hall, 5ª Ed. New York, 1982, Vol. 2, p. 2163.

10. The compound **19** has been described previously in the literature, though MS data only have been reported.

11. The structure has been assigned by using ^{1}H NMR data and it has been supported by mass spectrometry.

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