from acetone to yield 2.51 g (86%) of methyl O-methyl-$\Delta^{5,6}$-7-5 (2.51 g, 86%) as a white crystalline solid: mp 174-176 °C.

Decarbomethoxylated Methyl O-Methyl-$\Delta^{5,6}$-7-ketopodocarpate (6). A mixture of 0.44 g (0.001 mol) of TTN and 0.26 g (0.001 mol) of ketone 2 in 2.5 mL of a 1:1 mixture of MeOH and trimethyl orthoformate was refluxed for 8 h, concentrated in vacuo, and diluted with $CHCl_3$. Thallium(I) nitrate was removed by filtration. The filtrate was washed successively with 2 × 50-mL portions of water and 5% aqueous $NaHCO_3$ and then dried with $MgSO_4$. Concentration of the extract and repetitive recrystallization from a 50:1 CH_2Cl_2-MeOH gave the α,β-unsaturated ketone 6 (87%): mp 122-124 °C.

Acknowledgment. We are indebted to Drs. Catherine Costello and Thomas Dorsey of the MIT Mass Spectrometry Facility for their invaluable assistance in obtaining the mass spectrum. We thank Dr. Jim Dechter and Vicki Farr of the University of Alabama NMR facility for their help in obtaining ^1H NMR data.

Nonconventional Friedel-Crafts Chemistry. 1. Reaction of α-Tetralone and Anthrone with Arenes under Friedel-Crafts Conditions

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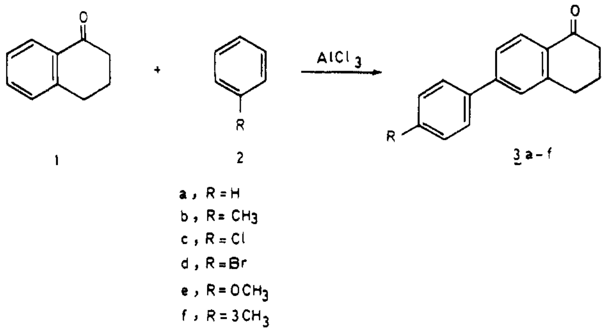
α-Tetralone and anthrone were reacted with arenes in the presence of aluminum chloride to give 6-aryl-1-tetralone and 10-arylanthrone, respectively. The mechanisms of these reactions are discussed.

Introduction

In previous reports[1], the condensation of phenols and naphthols with arenes in the presence of Lewis and Brønsted acids was investigated. The results of these investigations in addition to our interest in Friedel-Crafts chemistry[2-8] led us to consider the possibility of analogous reactions of α-tetralone and anthrone with arenes, with the aim of shedding more light on the mechanistic aspects of these reactions.

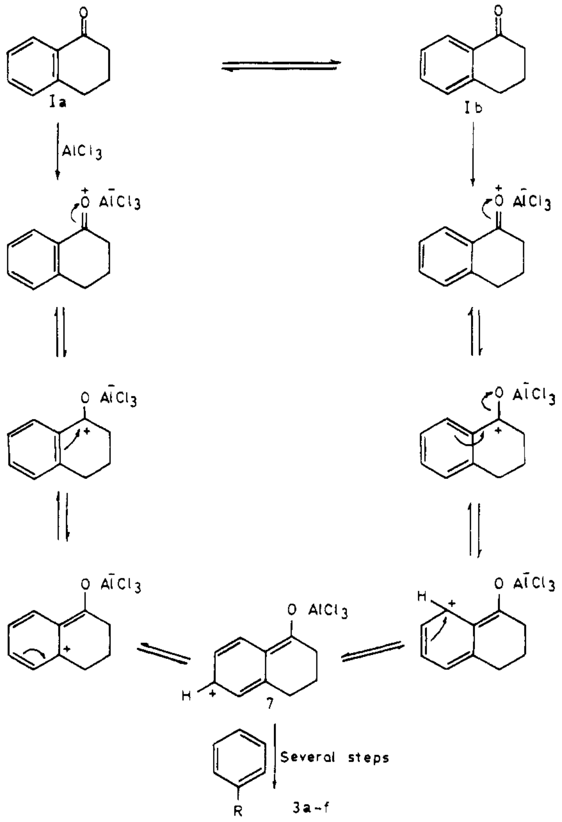
Results and Discussion

Surveying the results of Table I showed that, in the presence of $AlCl_3$ catalyst, α-tetralone reacted with arenes to yield 6-aryl-1-tetralone (eq 1) in fairly good yields (45-68%).



(1)

Scheme I



For instance, in the case of benzene (Table I, entry 1), 6-phenyl-1-tetralone (3a; 56%) was formed in addition to biphenyl (10%), terphenyl (7%), and quarterphenyl (5%). The formation of biphenyl, terphenyl, and quaterphenyl under these reaction conditions could be explained via dimerization, trimerization, and tetramerization of benzene under prevailing reaction conditions.[9-11] The reaction of

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Table I. Reactions of 1-Tetralone with Various Arenes in the Presence of AlCl$_3$ Catalyst

entry	arene	1-T/arene/ catalyst[a]	temp, °C	time, h	observed products[b] (%)
1	benzene	0.01/25/0.06	25	48	6-phenyl-1-tetralone (56), biphenyl (10), terphenyl (7), quaterphenyl (5)
2	toluene	0.01/25/0.06	25	48	6-(4′-tolyl)-1-tetralone (60), phenyl-4-tolylmethane (15), bitolyl (12)
3	chlorobenzene	0.01/25/0.06	25	48	6-(4′-chlorophenyl)-1-tetralone (47), 6-phenyl-1-tetralone (16)
4	bromobenzene	0.01/25/0.06	25	48	6-(4′-bromophenyl)-1-tetralone (50), 6-phenyl-1-tetralone (18)
5	anisole	0.01/25/0.06	25	48	6-(4′-methoxyphenyl)-1-tetralone (40), 6-(4′-hydroxyphenyl)-1-tetralone (21)
6	mesitylene	0.01/25/0.06	25	48	6-(2′,4′,6′-trimethylphenyl)-1-tetralone (42), mesityl-*m*-xylylmethane (19)

[a] 1-T = 1-tetralone. Units: 1-T, moles; arene, milliliters; catalyst, moles. [b] The percentages of these products were calculated by HPLC. The separations of these products were performed by preparative thin-layer chromatography.

1 with toluene (Table I, entry 2) gave 6-(4′-tolyl)-1-tetralone (**3b**; 60%), phenyl-4-tolylmethane (**4**, 15%), and bitolyl[12] (12%). The formation of **4** could be explained through the formation of a benzyl carbocation from toluene by a hydride abstraction mechanism[13] and followed by subsequent alkylation of the solvent in the presence of AlCl$_3$.[12-15]

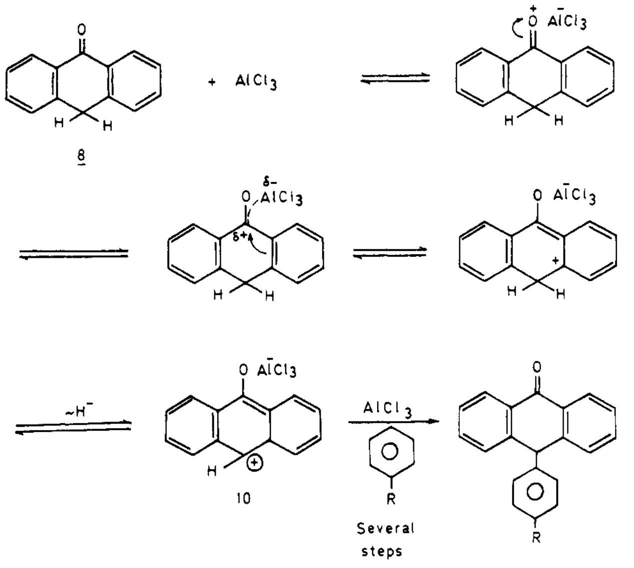
When **1** was reacted with **2c** and **2d** (Table I, entries 3 and 4), **3c** and **3d** were afforded in 47% and 50% yields, respectively, and **3a** was formed in 16% and 18% yields, respectively. The formation of **3a** in runs 3 and 4 was attributed to the Lewis acid dehalogenation[16] of **3c** and **3d**. The reaction of 1-tetralone with anisole (Table I, entry 5) gave 6-(4′-methoxyphenyl)-1-tetralone (**3e**; 40%) and 6-(4′-hydroxyphenyl)-1-tetralone (**5**; 21%). The formation of **5** was argued to the demethylation of **3e** in the presence of AlCl$_3$[4]. The reaction of **1** with mesitylene gave **3f** (42%) and mesityl-*m*-xylylmethane (19%).[12-15]

It is clear from the above-mentioned results that there are no arylation products at the benzylic carbon in 1-tetralone (C$_4$) and thus the formation of compounds **3a–f** could be explained as shown in Scheme I.

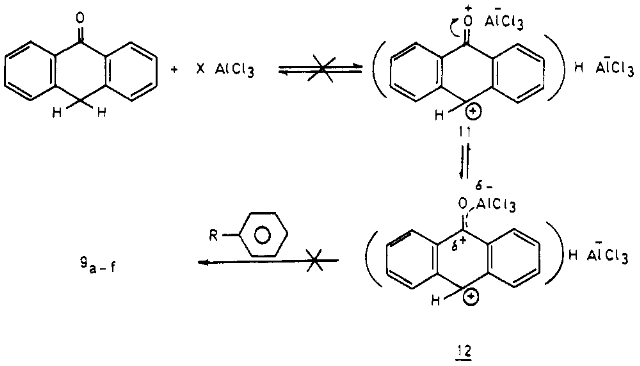
The preferential arylation of 1-tetralone at position 6 suggests that the intermediate carbocation **7** alkylates the arene to produce 6-aryl-1-tetralones **3a–f**. Confirmation of the proposed substitution pattern was based on ^{1}H NMR spectral analysis data. ^{1}H NMR data[17] for 1-tetralone (**1**) showed signals at δ 1.71–2.15 (2 H, m, H$_3$), 2.33–2.49 (2 H, t, H$_2$), 2.66–2.89 (2 H, t, H$_4$), 7–7.35 (3 H, m, H$_5$, H$_6$, H$_7$), and 7.82–8 (1 H, d, H$_8$), whereas ^{1}H NMR for **3a** showed the same signals as 1-tetralone (Table III) with the exception that the integration of the signals corresponding to the protons attached to C$_5$, C$_6$, and C$_7$ (in 1-tetralone) were higher by a value of four protons (in the spectrum of **3a**) at δ 7–7.4 (7 H, complex, H$_5$, H$_7$, and five protons of the phenyl group introduced at C$_6$).

For rigid identification of **3a**, an authentic sample of 6-phenyl-1-tetralone was prepared as reported;[18] and the melting point, mixed melting point, IR, ^{1}H NMR, and HPLC behavior were found to be identical in all aspects.

Scheme II



Scheme III



From the results in Table I, it seemed that the reaction yields of **3a–f** are so consistent irrespective of the nature of the substituent group in the used arenes (**2a–f**). This could be ascribed, for example in case of benzene, to the formation of the side products biphenyl, terphenyl, and quaterphenyl.[9-11] The formation of such products consumes a portion of the used catalyst and the arene itself and therefore decreases the arylation product **3a**. On the basis of the electronic behavior of the methyl group, we expect in the case of toluene a high yield of **3b**, but again the competing reaction products phenyl-4-tolylmethane and bitolyl have resulted in the formation of **3b** in low yield. In the case of anisole (**2e**), the complexation of the methoxy group with AlCl$_3$[4] makes **2e** less reactive than toluene and accordingly **3e** was formed in only 40% yield; also, the demethylation[4] of **3e** to form **5** in 21% yield decreases the overall yield of **3e**. With mesitylene as the arene, in spite of the presence of the three activating methyl groups, **3f** was formed in only 42% yield. This could be attributed to the steric effects of the three methyl groups that hindered the complex formation of mesitylene with the carbocation **7**, which should be converted under the reaction conditions to **3f**; additionally, the side reac-

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Table II. Reactions of Anthrone with Various Arenes in the Presence of $AlCl_3$ Catalyst

entry	arene	An/arene/catalyst[a]	temp, °C	time, h	observed products[b] (%)
1	benzene	0.01/25/0.06	25	48	10-phenylanthrone (81.48)
2	toluene	0.005/15/0.03	25	48	10-(4′-tolyl)anthrone (84)
3	chlorobenzene	0.005/15/0.03	25	48	10-(4′-chlorophenyl)anthrone (65.8), 10-phenylanthrone (22)
4	bromobenzene	0.005/15/0.03	25	48	10-(4′-bromophenyl)anthrone (57.3), 10-phenylanthrone (22.2)
5	anisole	0.005/15/0.03	25	48	10-(4′-methoxyphenyl)anthrone (60), 10-(4′-hydroxyphenyl)anthrone (14)
6	mesitylene	0.005/15/0.03	25	48	10-(2′,4′,6′-trimethylphenyl)anthrone (70.5), mesityl-*m*-xylylmethane (21)

[a] An = anthrone. Units: An, moles; arene, milliliters; catalyst, moles. [b] The percentages of these products were calculated by HPLC. The separations of these products were performed by preparative thin-layer chromatography.

Table III. Physical Data of 6-Aryl-1-tetralones 3a–f and 10-Phenylanthrones 9a–f

compd[a,b]	Ar	yield, %	molecular formula	anal. calcd (found) C	H	X	IR (KBr), cm^{-1}	^1H NMR (solvent), δ (TMS)
3a	C_6H_5	56	$C_{16}H_{14}O$	86.45 (86.31)	6.34 (6.23)		1680 (C=O), 2700 (CH aliph), 3050	($CDCl_3$) 1.82–2.12 (2 H, m), 2.45–2.6 (2 H, t), 2.7–2.82 (2 H, t), 7–7.4 (7 H, complex), 7.8–7.93 (1 H, d)
3b	$C_6H_4CH_3$-4	60	$C_{17}H_{16}O$	86.41 (86.30)	6.81 (6.60)		1675 (C=O), 2710, 2840 (CH aliph), 045 (CH arom)	($CDCl_3$) 2–2.15 (2 H, m), 2.3 (3 H, s), 2.45–2.66 (2 H, t), 2.79–2.92 (2 H, t), 6.91–7.40 (6 H, complex), 7.8–8 (1 H, d)
3c	C_6H_4Cl-4	45	$C_{16}H_{13}OCl$	74.85 (74.60)	5.09 (5.00)	13.81 (13.71)	1680 (C=O), 2700 (CH aliph), 3040 (CH arom)	($CDCl_3$) 2–2.15 (2 H, m), 2.45–2.66 (2 H, t), 2.8–2.9 (2 H, t), 7–7.5 (6 H, complex), 7.8–8 (1 H, d)
3d	C_6H_4Br-4	40	$C_{16}H_{13}OBr$	63.80 (63.72)	4.34 (4.29)	26.53[d] (26.21)	1670 (C=O), 2715, 2845 (CH aliph), 3045 (CH arom)	($CDCl_3$) 1.8–2.09 (2 H, m), 2.37–2.5 (2 H, t), 2.66–2.83 (2 H, t), 6.92–7.47 (6 H, complex), 7.75–7.90 (1 H, d)
3e	$C_6H_4OCH_3$-4	40	$C_{17}H_{16}O_2$	80.93 (80.71)	6.38 (6.29)		1680 (C=O), 2700, 2840 (CH aliph), 3050 (CH arom)	($CDCl_3$) 1.8–2.12 (2 H, m), 2.4–2.6 (2 H, t), 2.66–2.81 (2 H, t), 3.6 (3 H, s), 6.81–7.33 (6 H, complex), 7.85–8 (1 H, d)
3f	$C_6H_2(CH_3)_3$-1,3,5	42	$C_{19}H_{20}O$	86.32 (86.22)	7.61 (7.41)		1675 (C=O), 2715, 2850 (CH aliph), 3060 (CH arom)	($CDCl_3$) 2–2.15 (2 H, m), 2.33 (9 H, s), 2.45–2.66 (2 H, t), 2.7–2.9 (2 H, t), 7–7.3 (4 H, m), 7.9–8 (1 H, d)
9a	C_6H_5	81.5	$C_{20}H_{14}O$	88.86 (88.62)	5.21 (5.17)		1670 (C=O), 2890–2910 (CH aliph), 3080 (CH arom)	(CF_3COOH), 4.66 (1 H, s), 6.85 (2 H, d), 7.39 (4 H, t), 7.8 (5 H, complex), 8.15 (2 H, d)
9b	$C_6H_4CH_3$-4	84.42	$C_{21}H_{16}O$	88.70 (88.51)	5.66 (5.42)		1675 (C=O), 2880–2900 (CH aliph), 3050 (CH arom)	(CF_3COOH) 2.3 (3 H, s), 4.65 (1 H, s), 6.82 (2 H, d), 7.3 (4 H, t), 7.72 (4 H, m), 8.15 (2 H, d)
9c	C_6H_4Cl-4	65.78	$C_{20}H_{13}OCl$	78.82 (78.66)	4.29 (4.25)	11.63[c] (11.52)	1680 (C=O), 2890 (CH aliph), 3060 (CH arom)	(CF_3COOH) 4.67 (1 H, s), 6.83 (2 H, d), 7.43 (4 H, m), 7.83 (4 H, m), 8.17 (2 H, d)
9f	C_6H_4Br-4	68.76	$C_{20}H_{13}OBr$	68.78 (68.71)	3.74 (3.63)	22.88[d] (22.66)	1678 (C=O), 2890 (CH aliph), 3040–3060 (CH arom)	(CF_3COOH) 4.7 (1 H, s), 6.89 (2 H, d), 7.38 (4 H, m), 7.83 (4 H, m), 8.22 (2 H, d)
9e	$C_6H_4OCH_3$-4	60	$C_{21}H_{16}O$	83.98 (83.67)	5.36 (5.29)		1660 (C=O), 2850 (CH aliph), 3020 (CH arom)	(CF_3COOH) 3.6 (3 H, s), 4.7 (1 H, s), 6.83 (2 H, d), 7.3 (4 H, t), 7.83 (4 H, m), 8.15 (2 H, d)
9f	$C_6H_2(CH_3)_3$-1,3,5	70.51	$C_{23}H_{20}O$	88.43 (88.29)	6.44 (6.37)		1665 (C=O), 2910 (CH aliph), 3080 (CH arom)	(CF_3COOH) 2.17–2.39 (9 H, s), 4.72 (1 H, s), 6.9 (2 H, d), 7.44 (4 H, complex), 7.85 (2 H, s), 8.25 (2 H, d)

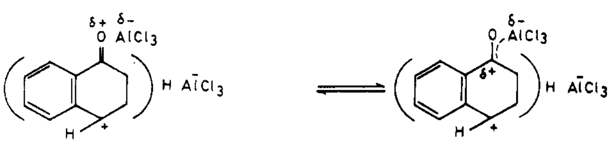
[a] **3a–f** were separated by preparative TLC: **3a** (eluent benzene, R_f 0.8), oil solidified, crystallized from ether/petroleum ether, mp 106–108 °C; **3b**, pale yellow oil (eluent benzene, R_f 0.56); **3c**, viscous yellow oil (eluent benzene/ether (4:1), R_f 0.71); **3d**, heavy yellow oil (eluent benzene/ether (4:1), R_f 0.73); **3e**, viscous yellow oil (eluent benzene/ether (3:2), R_f 0.07); **3f**, heavy viscous yellow oil (eluent benzene/ether (4:1), R_f 0.67). [b] **9a–f** were isolated by preparative TLC and then were recrystallized from the proper solvent: **9a**, pale yellow crystals (dilute acetic acid), mp 297–299 °C; **9b**, pale yellow crystals (dilute acetic acid) mp 260–263 °C. **9c–f** were purified only by chromatography: **9c** (eluent benzene/ether (1:1), R_f 0.8), mp 300–302 °C sublimed; **9d** (eluent benzene/ether (1:1), R_f 0.76), mp 275–277 °C; **9e** (eluent benzene/ether (1:1), R_f 0.78); **9f** (eluent benzene/ether (1:1), R_f 0.83), yellow crystals, mp 290–292 °C. [c] Chlorine. [d] Bromine.

tions of mesitylene itself[12–15] resulted the formation of **3f** in low yield.

The interesting results of the reaction of arenes with α-tetralone encouraged us to investigate the reaction of anthrone (**8**) with arenes in the presence of $AlCl_3$. Surprisingly, the reaction of benzene with **8** gave exclusively 10-phenylanthrone (**9a**) (Table II, entry 1). Toluene and other arenes (Table II) have in all cases 10-arylanthrones **9b–f** in addition to Friedel–Crafts side products in some cases. The formation of **9a–f** could be explained as depicted in Scheme II.

The failure to detect any arylation product at the carbon atoms of the two benzene rings in anthrone (**8**) was based on the favorable formation of the double benzylic carbocation **10** (Scheme II). An alternative mechanism (Scheme III) involving hydride anion abstraction from C_{10} in anthrone (**8**) to give the intermediate carbocations **11** and **12** was ruled out due to the difficulty of formation of the intermediates **11** and **12** with two positive centers on each.

The same argument could be used to rationalize the failure to detect any arylation products at the benzylic position (C_4) in α-tetralone since the reaction should proceed through the energetically unfavored intermediates **13** and **14**.



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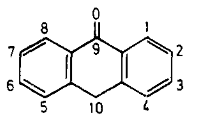
Structures of the formed 10-arylanthrones **9a–f** were elucidated by microanalytical and spectral analysis. The

^1H NMR spectrum of anthrone[17] (**8**) shows signals at δ 4.2



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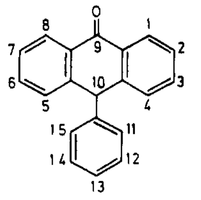

9a

(2 H, S, H_{10}), 7.37 (6 H, complex, $H_{2,3,4,5,6,7}$), and 8.22 (2 H, d, $H_{1,8}$) whereas 10-phenylanthrone (**9a**) showed signals at δ 4.66 (1 H, S, H_{10}), 6.85 (2 H, d, $H_{4,5}$), 7.39 (4 H, m, $H_{2,3,6,7}$), 7.8 (5 H, complex, $H_{11,12}$, $H_{13,14,15}$), and 8.15 (2 H, d, $H_{1,8}$). Comparison and accurate examination of ^1H NMR spectra of **8** and **9a** gave clear and rigid confirmation of the introduction of an aryl group in position 10 of anthrone.

Experimental Section

Melting points are uncorrected. Nuclear magnetic spectra were measured on EM-360 90-MHz spectrophotometer. Infrared spectra were recorded on a Pye-Unicam SP 200-G spectrophotometer. Isolation of products was achieved on a 20 × 15 cm glass plate covered with thin silica gel film. UV absorbances were measured on a Perkin-Elmer 552 spectrophotometer. Elemental analyses were recorded on a Perkin-Elmer 240 C microanalyser.

High-pressure liquid chromatography (HPLC) analyses were performed on a Hitachi apparatus (Japan) supplied with a variable-wavelength monitor (190–600 nm), with a sulfonated silica gel type column, Nucleosil 55 A$^-$, and methanol as a solvent.

Materials. All arenes, α-tetralone, and anthrone were purchased from Aldrich Chemical Co. Silica gel GF_{254} (Type 60) for thin-layer chromatography (Merck) was used in preparative thin-layer chromatography.

Reaction of 1-Tetralone and/or Anthrone with Arenes in the Presence of $AlCl_3$ Catalyst. General Procedure. A sample of 0.061 mol of $AlCl_3$ was added to a solution of 0.01 mol of 1-tetralone or anthrone in 25 mL of the arene in a two-necked flask equipped with a reflux condenser capped with calcium chloride tube, a magnetic stirrer, and a dropping funnel. The reaction mixture was stirred for 48 h at room temperature (RT), decomposed with 10% HCl solution, and extracted with chloroform and methylene chloride; the combined extracts were washed with water, 10% sodium carbonate solution, and again water and dried over magnesium sulfate. The solvents and the unreacted arene were removed by distillation under reduced pressure on a rotatory evaporator, and the residue was subjected for separation and purification to preparative thin layer chromatography. Products were identified as described under each individual run. Results are found in Tables I and II, and the physical data of the products **3a–f** and **9a–f** are depicted in Table III.

The Reactions of an *o*-Quinone Monoimide with Some Phenols

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The *o*-quinone monoimide *N*-(2,4-dichloro-6-oxo-2,4-cyclohexadien-1-ylidene)-4-nitrobenzamide (**1**) reacts with 2,6-dimethoxy-, 2,6-dimethyl-, and 2,6-dichlorophenols (**2a–c**) to form *N*-(2,4-dichloro-6-hydroxyphenyl)-*N*-(3,5-dimethoxy-, 3,5-dimethyl-, 3,5-dichloro-4-hydroxy)-4-nitrobenzamides (**3a–c**), respectively. Oxidation of **3a** by **1** and **3b** by DDQ lead to *N*-aryl-*p*-iminoquinones **4a,b**. Oxidative dimerizations by either C–C or C–O coupling occur when **1** is admixed with the sterically hindered 2,6-di-*tert*-butyl-, 2,4-di-*tert*-butyl-, 4-bromo-2,6-di-*tert*-butyl-, and 2,4,6-tri-*tert*-butylphenols.

The reactions of 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (DDQ) with a wide variety of mono- and dihydric phenols have been studied extensively, especially by Becker.[1a–c] Surprisingly, little work has been done on the reactions of *o*-quinones and their derivatives with phenols. A few articles have appeared on the treatment of naphthols with chloranil,[2a–d] but there is only one report on the reactions of *o*-chloranil with phenols[3] and one paper involving the interaction of an *o*-quinone diimide and phenol.[4] This paper describes the reactions of the *o*-quinone monoimide *N*-(2,4-dichloro-6-oxo-2,4-cyclohexadien-1-ylidene)-4-nitrobenzamide (**1**) with substituted monohydric phenols and the subsequent oxidations of some of the *p*-amidophenols that were prepared during the course of this study.

Results and Discussion

Admixing of **1** with 2,6-dimethoxy-, 2,6-dimethyl-, and 2,6-dichlorophenols (**2a–c**) in methanol formed the *p*-amidophenols **3a–c** (Scheme I). The action of 2 equiv of **1** on **2a** or 1 equiv of **1** with **3a** afforded the *N*-aryl-*p*-iminoquinone **4a** (84%) and compound **5**. Reduction of **4a** with sodium borohydride reformed **3a**. No reaction occurred between **1** and **3b,c**; however, **3b** was converted to

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