Synthesis of Arylnaphthoquinones and Their Reactions with *o*-Substituted Primary Aromatic Amines

Ragab F. Fandy[a]*, Hessan H. Abbas[a], Ayman S. Al-Hussaini[a] and Ahmed S. Hammam[b]
[a]*Chemistry Department, Faculty of Science, South Valley University, Qena, Egypt*
[b]*Chemistry Department, Faculty of Science, Assiut University, Assiut, Egypt*

Cycloaddition reaction of 2-aryl-1,4-benzoquinones **1a-d** with a number of different dienes, namely 2,3-dimethylbutadiene; 1,4-diphenylbutadiene and anthracene yield 2-aryl-6,7-dimethyl-1,4- naphthoquinones **3a,b**; 2,5,8-triphenyl-1,4-naphthoquinone **4** and 2-aryl-1,4,9,10-tetrahydro-9,10-*o*-benzoanthracene-1,4-dione **5**, respectively were investigated. In addition, the cycloaddition reaction of 2-aryl-1,4-benzoquinones **1d,e** with 2,3-dimethylbutadiene was also investigated to yield 2-aryl-5,8-dihydro-6,7-dimethyl-1,4-naphthohydroquinones **2a,b**. Cyclocondensation reactions of Diels-Alder ducts **2b**, **3b**, **5a** with ethylenediamine, *o*-substituted primary aromatic amines gave quinoxaline, phenazine, phenoxazine and phenothiazine ocyclic derivatives **6-14**.

INTRODUCTION

In continuation of our study for the synthesis of heterocyclic nitrogen dervatives,[1-6] we were interested in the reaction of arylnaphthoquinones with thioglycolic acid and primary aromatic amines giving two types of the condensation products.[6] The quinoxaline,[4,7-14] phenazine,[15-18] phenoxazine[15,19] and phenothiazine,[20,21] ring systems have been extensively studied and many such compounds have now become commercially available and are widely used in medical practice[22,23] and in the dyeindustry.[24] This led us to synthesize some new related compounds. In this work, 5-arylbenzo-[*f*]quinoxalin-6-one **6**, 5-arylbenzo[*f*]quinoxalin-6-ol **7**, 6-aryl-5-hydroxybenzo[*a*]phenazine **8**, 6-arylbenzo[*a*]phenothiazin-5-one **9** and 6-arylbenzo[*a*]phenoxazin-5-one **10** were prepared by the reactions of the synthesized Diels-Alder adducts, arylnaphthoquinones with ethylenediamine and/or *o*-substituted primary aromatic amines.

RESULTS AND DISCUSSION

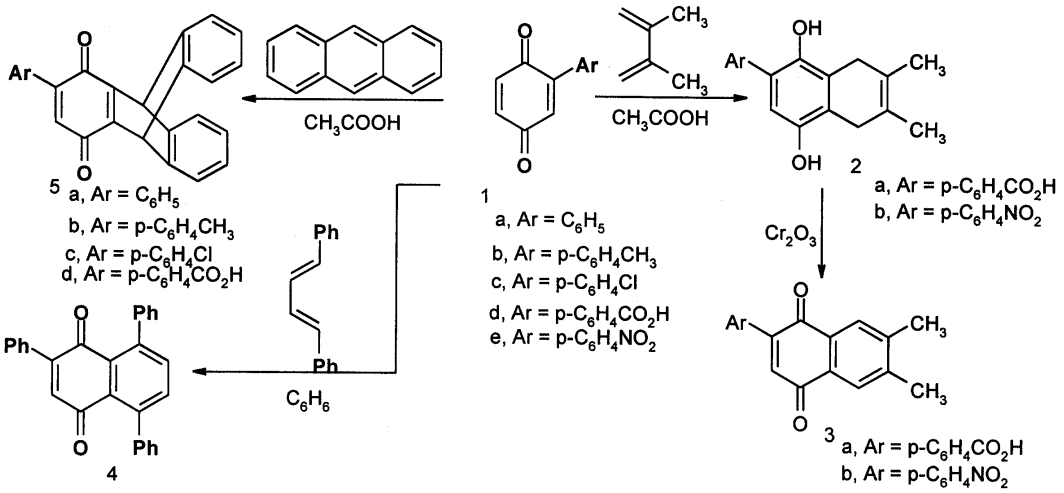
Treatment of 2-(*p*-carboxyphenyl)-1,4-benzoquinone **1d** and/or 2-(*p*-nitrophenyl)-1,4-benzoquinone **1e** with 2,3-dimethylbutadiene in glacial acetic acid yields colorless compounds of 2-aryl-5,8-dihydro-6,7-dimethyl-1,4- naphthohydroquinones **2a,b**. The oxidation of compounds **2a,b** with aqueous solution of chromium trioxide in hot glacial acetic acid gives yellow compounds of 2-aryl-6,7-dimethy-1,4-naphthoquinones **3a,b**. Diels-Alder adduct from the reaction of 2-phenyl-1,4-benzoquinone **1a** with 1,4-diphenylbutadiene in dry benzene was obtained in an oxidized state as 2,5,8-triphenyl-1,4-naphthoquinone **4**. The cycloaddition reaction of **1a-d** with anthracene proceeded also in hot glacial acetic acid, afforded the adducts **5a-d**, namely 2-aryl- 1,4,9,10-tetrahydro-9,10-*o*-benzoanthracene-1,4-diones (Scheme I).

The structures of these products **2-5** were supported by their analytical and spectral data (IR, NMR and MS) which were in full agreement with the proposed structures (cf. Tables 1 and 2).

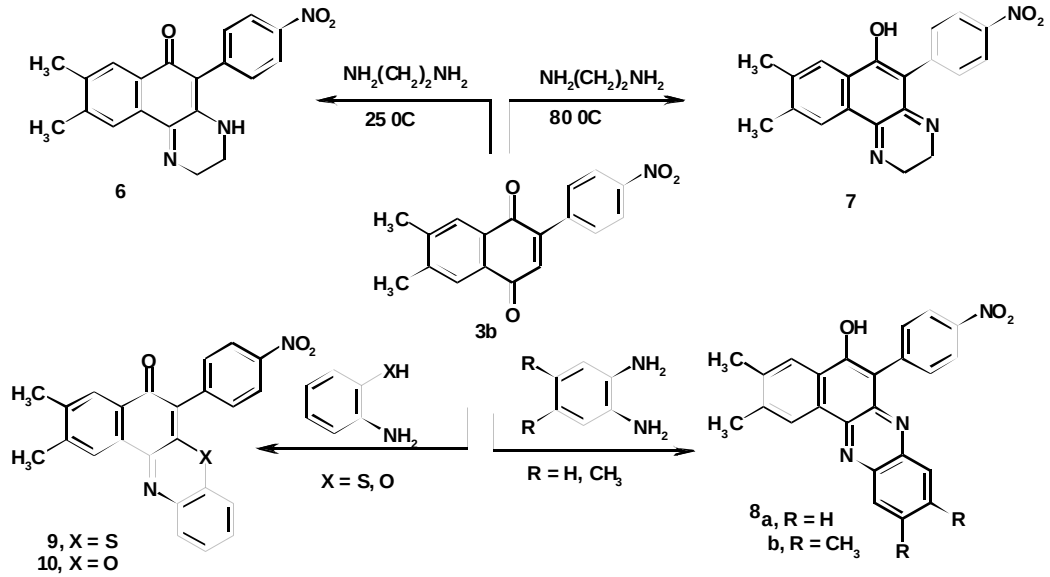
Treatment of Diels-Alder adducts, 6,7-dimethyl-2-(*p*-nitrophenyl)-1,4-naphthoquinone **3b** with ethylenediamine at room temperature and at 80 °C in absolute ethanol gave 8,9-dimethyl-5-(*p*-nitrophenyl)-2,3,4,6-tetrahydrobenzo[*f*]-quinoxalin-6-one **6** and 2,3-dihydro-8,9-dimethyl-5-(*p*-nitrophenyl)benzo[*f*]quinoxalin-6-ol **7**, respectively (Scheme II). IR spectra of compound **6** revealed the presence of νCO at 1710 cm^{-1} and the presence of νNH band at 3350 cm^{-1}. But IR spectra of compound **7** revealed the absence of νCO of quinone and presence of νOH band at 3400-3450 cm^{-1}; analytical and spectroscopic data of these compounds are listed in Tables 1 and 2. On the other hand the reactions of **3b** with *o*-substituted aromatic amines, namely *o*-phenylenediamine; 4,5-dimethyl-*o*-phenylenediamine; *o*-aminothiophenol and *o*-aminophenol gave 2,3-dimethyl-6-(*p*-nitrophenyl)-5- hydroxybenzo[*a*]phenazine **8**; 2,3-dimethyl-5H-6-(*p*-nitrophenyl)benzo[*a*]phenothiazin-5-one **9** and 2,3-dimethyl-5H-6-(*p*-nitrophenyl)benzo[*a*]phenoxazin-5-one **10**, respectively (Scheme II).

The chemical structures of these compounds were based on spectral and elemental analyses (cf. Tables 1 and 2). The reaction between **2b**, **5a** and *o*-substituted primary aro-

Scheme I



Scheme II



matic amines pro duced **11-14** in a sim i lar man ner to that described above (Scheme III). The chem i cal struc tures of these com pounds were based on spec tral and el e men tal anal y ses (cf. Ta bles 1 and 2).

EXPERIMENTAL

Melting points were de ter mined on an elec tric melt ing point ap pa ra tus (Gallenkamp) and were un cor rected. The IR spec tra (KBr) were re corded on a Shimadzu 408 spec trom e-ter. The ^{1}H-NMR spectra were recorded by a 300 MHz Varian NMR spec trom e ter; chem i cal shifts are re ported in ppm with TMS as an in ter nal stan dard and are given in δ units. Elec tron im pact mass spec tra were ob tained at 70 eV us ing a GCMS sp. 1000 Shimadzu. El e men tal anal y ses were car ried out at the Microanalysis Unit at Cairo Uni ver sity.

Synthesis of 2-aryl-5,8-dihydro-6,7-dimethyl-1,4-naphthohydroquinones 2a,b

A so lu tion of 2-(*p*-carboxyphenyl)-1,4-benzoquinone **1d** (0.005 mol) or 2-(*p*-ni trophenyl)-1,4-benzoquinone **1e**

Table 1. Physical Data of Compounds **2-14**

Comp. No.	Ar	Cryst. Solvent	Yield %	Appearance	M.P. °C	Molecular Formula
2a	*p*-$C_6H_4CO_2H$	acetic acid	67	colorless needles	257	$C_{19}H_{18}O_4$ (310.34)
2b	*p*-$C_6H_4NO_2$	acetic acid	74	colorless needles	265-8	$C_{18}H_{17}NO_4$ (311.33)
3a	*p*-$C_6H_4CO_2H$	acetic acid	63	yellow flakes	270-2	$C_{19}H_{14}O_4$ (306.32)
3b	*p*-$C_6H_4NO_2$	acetic acid	71	yellow flakes	256-8	$C_{18}H_{13}NO_4$ (307.30)
4	C_6H_5	petroleum ether (60-80)	70	yellowish orange needles	103-5	$C_{28}H_{18}O_2$ (386.45)
5a	C_6H_5	aqueous ethanol	88	yellowish brown crystals	157-8	$C_{26}H_{16}O_2$ (360.41)
5b	*p*-$C_6H_4CH_3$	aqueous ethanol	92	yellowish orange crystals	115-6	$C_{27}H_{18}O_2$ (374.44)
5c	*p*-C_6H_4Cl	aqueous ethanol	63	pale brown fine crystals	139-140	$C_{26}H_{15}O_2Cl$ (394.86)
5d	*p*-$C_6H_4CO_2H$	aqueous ethanol	70	gray fine crystals	148	$C_{27}H_{16}O_4$ (404.42)
6	-	ethanol/petr. ether	80	brown crystals	184	$C_{20}H_{17}N_3O_3$ (347.34)
7	-	aqueous ethanol	64	yellowish gray micro crystals	128-130	$C_{20}H_{17}N_3O_3$ (347.34)
8a	-	acetone	60	orange red crystals	320-2	$C_{24}H_{17}N_3O_3$ (395.42)
8b	-	acetone	66	red flakes	346-8	$C_{26}H_{21}N_3O_3$ (423.47)
9	-	acetone	43	cherry red needles	326-8	$C_{24}H_{16}N_2SO_3$ (412.47)
10	-	methanol	30	brownish yellow flakes	324-5	$C_{24}H_{16}N_2O_4$ (396.40)
11	-	chloroform/pet. ether	40	red micro crystals	310	$C_{24}H_{18}N_2SO_3$ (414.48)
12a	-	ethanol/pet. ether	52	red micro crystals	270-2	$C_{24}H_{19}N_3O_3$ (397.43)
12b	-	ethanol/pet. ether	56	red micro crystals	> 360	$C_{26}H_{23}N_3O_3$ (425.49)
13	-	benzene/pet. ether	39	dark brown crystals	170	$C_{32}H_{19}NSO$ (465.56)
14a	-	aqueous ethanol	62	brownish orange crystals	182	$C_{32}H_{20}N_2O$ (448.51)
14b	-	aqueous ethanol	66	deep brown crystals	168	$C_{34}H_{24}N_2O$ (476.58)

and (0.41 g; 0.005 mol) of 2,3-dimethylbutadiene in 20 mL glacial acetic acid was left for 12 hours in a closed vessel at room temperature. The reaction mixture was heated under reflux for 3-5 hours and cooled. The solid product so formed was collected by filtration and recrystallized from the appropriate solvent to yield the desired colorless products **2a,b**; yields, melting points, analytical and spectroscopic data are listed in Tables 1 and 2.

Synthesis of 2-aryl-6,7-dimethyl-1,4-naphthoquinones 3a,b

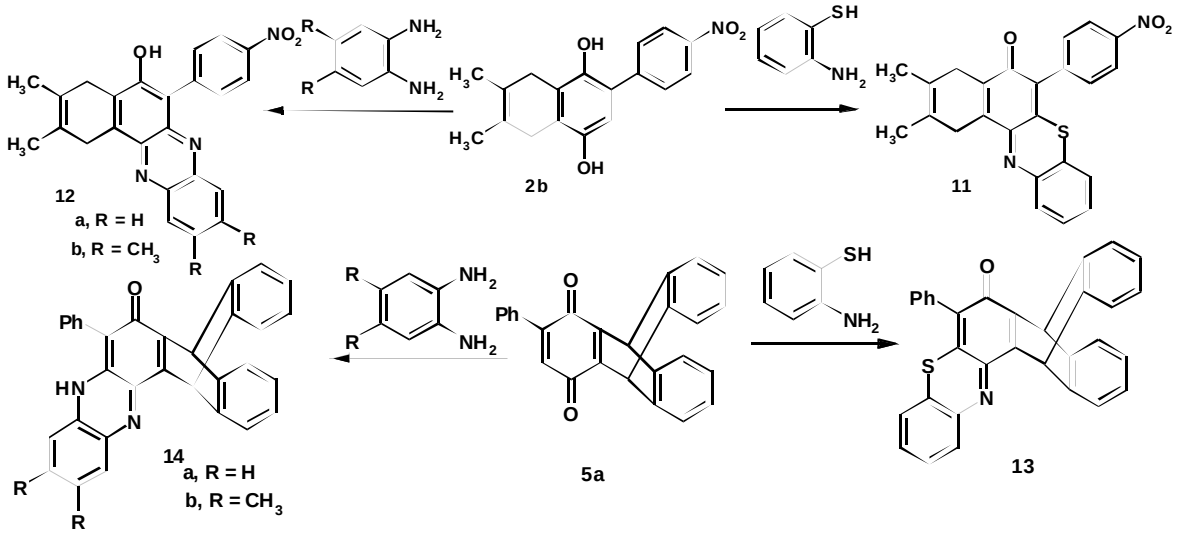
To a hot solution of **2a** or **2b** (0.005 mol) in 25 mL glacial acetic acid was gradually added a solution of chromic acid (1.35 g of chromium trioxide in 3 mL distilled water).

Table 2. Analytical and Spectroscopic Data of Compounds **2-14**

Comp. No.	Ar	Elemental Analysis Found/Calcd.			MS m/e base peak	IR (cm^{-1})	^{1}H-NMR
		C	H	N			
2a	*p*-$C_6H_4CO_2H$	73.42 73.54	5.94 5.85			1680, 2925 (b) 3525 (c)	1.75(s, 6H, $2CH_3$); 3.12(s, 4H, $2CH_2$); 6.60(s, 1H, H-3); 7.69; 7.90(2s, 2H, 2 OH) and 7.60; 7.96(2d, 4H, Ar) (f).
2b	*p*-$C_6H_4NO_2$	69.32 69.44	5.41 5.50	4.43 4.50		3525 (c)	1.72(s, 6H, $2CH_3$); 3.16(s, 4H, $2CH_2$); 6.68(s, 1H, H-3); 7.76; 8.14(2s, 2H, 2 OH) and 8.18; 8.36(2d, 4H, Ar) (f).
3a	*p*-$C_6H_4CO_2H$	74.42 74.50	4.56 4.61		306($M^{+\cdot}$, 30.8) 261(100)	1665 (a) 1700, 2925 (b)	2.52(s, 6H, $2CH_3$); 7.17(s, 1H, H-3); 7.73; 8.05(2d, 4H, Ar) and 7.80; 7.86 (2s, 2H, H-5, 8) (f).
3b	*p*-$C_6H_4NO_2$	70.48 70.35	4.39 4.26	4.58 4.56		1670 (a)	2.43(s, 6H, $2CH_3$); 7.05(s, 1H, H-3); 7.67; 7.78(2s, 2H, H-5, 8) and 7.92; 8.38(2d, 4H, Ar) (e).
4	C_6H_5	87.15 87.02	4.82 4.69			1630, 1665 (a)	6.82(s, 1H, H-3); 6.85-6.92(m, 5H, Ar); and 7.46-7.58(m, 12H, Ar) (e).
5a	C_6H_5	86.58 86.65	4.38 4.47		360($M^{+\cdot}$, 87.8) 202(100)	1650, 1715 (a)	5.88(s, 2H, H-9, 10); 6.67(s, 1H, H-3); 7.04-7.10(m, 5H, Ar) and 7.41-7.50 (m, 8H, Ar) (e).
5b	*p*-$C_6H_4CH_3$	86.75 86.60	5.14 4.85		374($M^{+\cdot}$, 100)	1650, 1715 (a)	2.35(s, 3H, CH_3); 5.91(s, 2H, H-9,10); 6.72(s, 1H, H-3); 7.25; 7.33(2d, 4H, Ar) and 7.48-7.52(m, 8H, Ar) (f).
5c	*p*-C_6H_4Cl	79.15 79.09	3.71 3.83			1650, 1715 (a)	5.89(s, 2H, H-9,10); 6.66(s, 1H, H-3); 7.05; 7.11(2d, 4H, Ar) and 7.36-7.49(m, 8H, Ar) (f).
5d	*p*-$C_6H_4CO_2H$	80.07 80.19	3.89 3.99		404($M^{+\cdot}$, 87.1) 202(100)	1650 (a); 1690, 3450 (b)	5.96(s, 2H, H-9,10); 6.78(s, 1H, H-3); 7.42-7.58(m, 12H, Ar) and 10.84(s, 1H, COOH) (f).
6	-	69.09 69.15	4.88 4.93	12.13 12.10	347($M^{+\cdot}$,24.9) 220(100)	1710 (a) 3350 (d)	2.38; 2.44(2s, 6H, $2CH_3$); 3.62-3.87(m, 4H, $2CH_2$); 6.18(m, 1H, NH); 7.15(m, 4H, Ar); 7.92and 8.12(2s, 2H, Ar) (e).
7	-	69.31 69.15	4.85 4.93	12.19 12.10	347($M^{+\cdot}$,15.4) 299(100)	3450 (c)	2.41; 2.49(2s, 6H, $2CH_3$); 3.82(s, 4H, $2CH_2$); 7.18(m, 4H, Ar); 8.12; 8.86(2s, 2H, Ar) and 9.15(s, 1H, OH) (e).
8a	-	72.74 72.90	4.38 4.33	10.38 10.63	395($M^{+\cdot}$,100)	3400 (c)	2.58(s, 6H, $2CH_3$); 7.80-7.85(m, 4H, Ar); 8.37(s, 2H, H-1, H-4); 8.41-8.42(m, 4H, Ar) and 9.07(s, 1H, OH) (f).
8b	-	73.59 73.74	5.07 4.99	9.97 9.92		3400 (c)	2.48; 2.51; 2.61; 2.66(4s, 12H, $4CH_3$); 7.62-7.72(m, 4H, Ar); 7.98(s, 2H, Ar); 8.12; 8.33(2s, 2H, Ar) and 9.04(s, 1H, OH) (f).
9	-	69.87 69.89	3.98 3.91	6.64 6.79		1665 (a)	2.46; 2.52(s, 6H, $2CH_3$); 7.33-7.55(m, 4H, Ar); 7.58; 8.39(2d, 4H, Ar) and 8.07; 8.68(2s, 2H, H-1,4) (e).
10	-	72.55 72.72	4.13 4.07	6.92 7.07	396($M^{+\cdot}$,100)	1650 (a)	2.52; 2.60(s, 6H, $2CH_3$); 7.30-7.53(m, 4H, Ar); 7.58- 7.99(m, 4H, Ar) and 8.12; 8.58(2s, 2H, H-1,4) (e).
11	-	69.57 69.55	4.42 4.38	6.72 6.76		1665 (a)	1.65(s, 6H, $2CH_3$); 3.11(s, 4H, $2CH_2$); 7.33-7.52(m, 4H, Ar) and 7.57-7.62(m, 4H, Ar) (e).
12a	-	72.31 72.53	4.85 4.82	10.49 10.57		3450 (c)	1.56(s, 6H, $2CH_3$); 3.08(s, 4H, $2CH_2$); 7.57-7.68(m, 4H, Ar); 7.72-7.78(m, 4H, Ar) and 8.90(s, 1H, OH) (e).
12b	-	73.34 73.40	5.51 5.45	9.85 9.88		3500 (c)	1.66(s, 6H, $2CH_3$); 2.47; 2.50(2s, 6H, $2CH_3$); 3.12(s, 4H, $2CH_2$); 7.57-7.68(m, 4H, Ar); 7.75; 7.79(2s, 2H, H-8,11) and 8.94(s, 1H, OH) (e).
13	-	82.68 82.55	4.23 4.11	2.92 3.01		1710 (a)	5.89(s, 2H, H-9′, 10′); 7.06-7.12(m, 5H, Ar) and 7.48-7.56(m, 12H, Ar) (f).
14a	-	85.56 85.69	4.54 4.49	6.20 6.24	448($M^{+\cdot}$,4.1) 345(100)	1700 (a) 3350 (d)	5.68(s, 2H, H-9′, 10′); 7.12-7.19(m, 5H, Ar); 7.42-7.59(m, 12H, Ar) and 8.1(s,1H, NH) (f).
14b	-	85.52 85.69	5.12 5.07	5.82 5.87		1700 (a) 3350 (d)	2.60, 2.64(2s, 6H, $2CH_3$); 5.82(s, 2H, H-9′, 10′); 7.06-7.12(m, 5H, Ar); 7.45-7.51(m, 8H, Ar) and 8.11, 8.18(2s, 2H, Ar) (f).

(a) νCO; (b) νCOOH; (c) νOH; (d) νNH; (e) $CDCl_3$; (f) d_6-DMSO.

Scheme III



When all the oxidant had been added, the reaction solution was heated for 20 minutes without boiling and cooled to room temperature. The deposited yellow precipitate was then collected, washed with ethanol and recrystallized from the proper solvent to afford **3a,b**; yields, melting points, analytical and spectroscopic data are listed in Tables 1 and 2.

Synthesis of 2,5,8-triphenyl-1,4-naphthoquinone 4

To 0.46 g; (0.0025 mol) of 2-phenyl-*p*-benzoquinone **1a** in 25 mL dry benzene was added 0.515 g; (0.0025 mol) of 1,4-diphenylbutadiene. The reaction mixture was refluxed for 16 hours (TLC using petroleum ether/benzene 1:1 as eluent). The solvent was removed under reduced pressure, the residue formed was recrystallized from petroleum ether to afford **4**; yield, melting point, analytical and spectroscopic data are listed in Tables 1 and 2.

Synthesis of 2-aryl-1,4,9,10-tetrahydro-9,10-*o*-benzoanthracene-1,4-dione 5a-d

To a solution of 0.002 mol of **1a-d** in 15 mL glacial acetic acid was added 0.37 g, (0.002 mol) of anthracene. The reaction mixture was refluxed for 10-12 hours then cooled and diluted with 30 mL of distilled water. The resulting precipitate was filtered off, dried and recrystallized from the proper solvent to give **5a-d**; yields, melting points, analytical and spectroscopic data are listed in Tables 1 and 2.

Synthesis of 8,9-dimethyl-5-(*p*-nitrophenyl)-2,3,4,6-tetrahydrobenzo[*f*]quinoxalin-6-one 6

To 0.307 g, (0.001 mol) of **3b** in 20 mL absolute ethanol was added 0.06 mL, (0.06 g, 0.001 mol) of ethylenediamine. The reaction mixture was stirred at room temperature for 16 hours. The solid product so formed was recrystallized to give **6**; yield, melting point, spectroscopic and analytical data are listed in Tables 1 and 2.

Synthesis of 2,3-dihydro-8,9-dimethyl-5-(*p*-nitrophenyl)-benzo[*f*]quinoxalin-6-ol 7

Ethylenediamine (0.13 mL, 0.12 g, 0.002 mol) was added to 0.62 g, (0.002 mol) of **3b** in 25 mL absolute ethanol. The reaction mixture was refluxed for 3-4 hours, then concentrated and cooled. The precipitated solid was collected by filtration and recrystallized from the proper solvents to give **7**; yields, melting points, spectroscopic and analytical data are listed in Tables 1 and 2.

Synthesis of 2,3-dimethyl-6-(*p*-nitrophenyl)-5-hydroxybenzo[*a*]phenazine 8; 2,3-dimethyl-5*H*-6-(*p*-nitrophenyl)-benzo[*a*]phenothiazin-5-one 9 and 2,3-dimethyl-5*H*-6-(*p*-nitrophenyl)benzo[*a*]phenoxazin-5-one 10

General procedure

A suspension of 0.62 g, (0.002 mol) of **3b** in 25 mL absolute ethanol was treated with the appropriate *o*-substituted aromatic amine (0.002 mol). The reaction mixture was heated under reflux for 70-80 hours. The solvent was then evaporated under reduced pressure. The residual solids were purified on a column chromatography using ethanol/petroleum ether (2:8) as an eluent. The solids were collected, recrystallized from the appropriate solvents to give **8**, **9** and **10**, respectively; yields, melting points, spectroscopic and analytical data are listed in Tables 1 and 2.

Synthesis of 2,3-dimethyl-6-(*p*-nitrophenyl)-1,4,5-trihydrobenzo[*a*]phenothiazin-5-one 11 and 2,3-dimethyl-1,4-dihydro-5-hydroxy-6-(*p*-nitrophenyl)benzo[*a*]phenazine 12a,b

General procedure

To a solution of **2b** (0.31 g, 0.001 mol) in 30 mL absolute ethanol was added the appropriate *o*-substituted aromatic amine (0.001 mol) and heated under reflux for 7-12 hours. The reaction mixture was concentrated to one third of its volume and cooled. The precipitated products were collected and recrystallized from the proper solvent to yield **11** and **12**; yields, melting points, spectroscopic and analytical data are listed in Tables 1 and 2.

Synthesis of 1-phenyl-2*H*-phenothiazino[3,4-9',10']-9'*H*,10'*H*-anthracene-2-one 13 and 1-Phenyl-2*H*,10*H*-phenazino[3,4-9',10']-9'*H*,10'*H*-anthracene-2-one 14

General procedure

To a solution of **5a** (0.36 g, 0.001 mol) in absolute ethanol was added the appropriate *o*-substituted amine (0.001 mol). The reaction mixture was refluxed for 3-6 hours. The reaction solution was cooled to room temperature and diluted with two volumes of water. The precipitated products were collected, dried and recrystallized from the proper solvent to give **13** and **14a,b**; yields, melting points, spectroscopic and analytical data are listed in Tables 1 and 2.

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Key Words

2-Aryl-1,4-benzoquinones; Dienes; 2-Aryl-1,4-naphthoquinones; Quinoxaline; Phenazine; Phenoxazine and phenothiazine.

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