Reactions of 2-Ethoxycarbonyl-1,3-indandione with Aromatic Amines, Diazonium Salts, and Phenols

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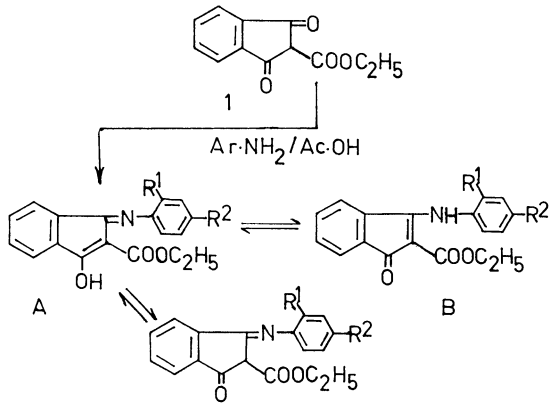
2-Ethoxycarbonyl-1,3-indandione (**1**) was treated with primary aromatic amines in acetic acid to afford 3-hydroxy-1-(arylimino) derivatives (**2**). Treatment of **1** with excess *p*-toluidine gave 3-tolylimino-4′-methyl-indancarboxanilide (**3**). With *o*- or *p*-phenylenediamine it yielded 3,3′-dihydroxy-1′-(*o*- or *p*-phenylenedinitrilo)di-2-indenecarboxylate. The tetraazacycloeicosene derivative was obtained by the action of *p*-phenylenediamine on **1** in a 1:1 molar ratio. In boiling toluene **1** gave the corresponding 2-carboxanilides (**8**) which cyclized to the diazepinone or to the quinolone. The coupling of **1** and **8** with diazonium salts was also investigated. The condensation of **1** with phenols afford **14**, which were hydrolysed to 3-aryl-1-indenones.

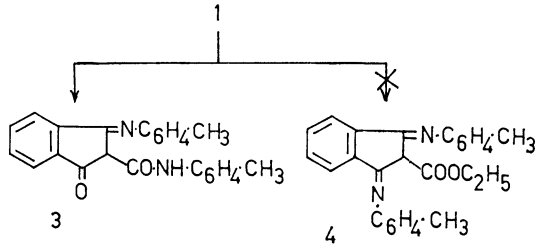
Many reactions of 1,3-indandione and its derivatives with various reagents have been reported.[1–5] However, little work has been done on the reactions of 2-ethoxycarbonyl-1,3-indandione.[6–7] The compound is considered to be an easily available starting material for the synthesis of some new 1,3-indandione derivatives.

We have found that 2-ethoxycarbonyl-1,3-indandione (**1**)[8] reacts with aromatic amines to give various products depending upon the reaction conditions. In hot acetic acid **1** reacts with primary aromatic amines to give ethyl 3-hydroxy-1-(arylimino)-2-indenecarboxylate (enolic form A; **2a**—**d**). Structure of **2** was elucidated on the basis of elemental analysis and IR spectra, which showed bands attributable to (C=N) 1630, enolised *β*-keto ester 1670, 3080 and (C=O) 1705 cm^{-1}. Furthermore, the lack of IR bands characteristic of the (NH) stretching mode ruled out the alternative structure B. Thus, it can be concluded that the structure of **2** is best represented by the enolic form A.

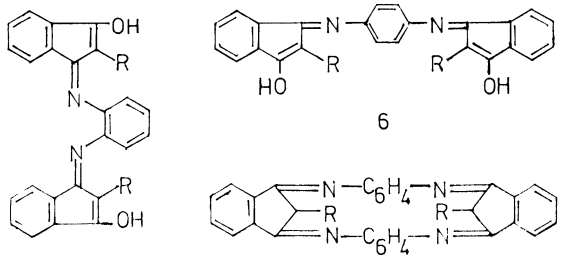


2 a: $R^1 = R^2 = H$
b: $R^1 = H$, $R^2 = OCH_3$
c: $R^1 = H$, $R^2 = CH_3$
d: $R^1 = NH_2$, $R^2 = H$

Treatment of **1** with excess *p*-toluidine in hot acetic acid gave 1-oxo-3-(*p*-tolylimino)-4′-methyl-2-indancarboxanilide (**3**). The IR spectrum of **3** indicated bands characteristic of the secondary amide group at 3400 (–NH) and 1630, 1540 cm^{-1} (–NHCO–). In particular, the presence of a –NHCO– grouping is confirmed by the NMR spectrum (*δ* 8.1, 1H, s). Attempts to prepare ethyl 1,3-bis(tolylimino)-2-indancarboxylate (**4**) failed.



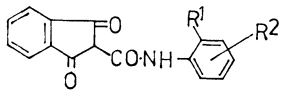
Condensation of **1** with aromatic diamines was also investigated. The reaction of **1** with *o*-phenylenediamine in a 2:1 molar ratio in hot acetic acid yielded a mixture of two compounds, which were separated by their different solubilities in ethanol. The insoluble compound was found to be diethyl 3,3′-dihydroxy-1,1′-(*o*-phenylenedinitrilo)di-2-indenecarboxylate (**5**), while the ethanol soluble one was identified to be **2d** by elemental analysis, mixed mp and IR spectrum. Under essentially the same conditions, condensation of **1** with *p*-phenylenediamine afforded diethyl 3,3′-dihydroxy-1,1′-(*p*-phenylenedinitrilo)di-2-indenecarboxylate (**6**). The assigned structure for the two compounds, **5** and **6**, is inferred from their elemental analyses and the IR spectra. In the mass spectra of both compounds, the basic peak was *m/e* 362 corresponding to the splitting of the two ethoxycarbonyl groups.



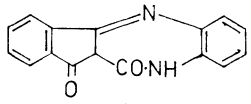
Treatment of **1** with *p*-phenylenediamine in 1:1 molar ratio in acetic acid at 80 °C for 1 h, gave diethyl 7,10 : 19,22 - dietheno - 5,24 : 12,17 - dimethanodibenzo-[*c*,*m*][1,6,11,16]tetraazacycloeicosene - 25,28-dicarboxylate (**7**). The results of the elemental analysis and the mass spectrum were in line with the molecular formula $C_{36}H_{28}N_4O_4$. The structure **7** was assigned

on the basis of its IR spectrum which shows bands at 1725 (ester group), 1200 (C–O stretching), and 1665 cm^{-1} (C=N), its mass spectrum showing a peak at mass *m/e* 490 ($M^+-2(OC_2H_5)$).

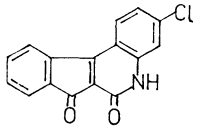
On the other hand, treatment of **1** with primary aromatic amines in boiling toluene afforded the corresponding 1,3-dioxo-2-indancarboxanilides (**8a**—**i**). The products **8** were characterized by analysis and spectral data. They all showed characteristic absorptions at 1680—1700 (C=O) and 1625, 1560 cm^{-1} (–NHCO–). The NMR spectra also exhibited one proton singlet at δ 8.1 due to (–NHCO–). Further, the identity is established by converting **8b** into **3**, by treatment with *p*-toluidine in acetic acid. The action of *o*-, or *p*-phenylenediamine on **1** in boiling toluene gave in each case a single product (**8f** and **g**), respectively. The mass spectra of both compounds, *m/e* 280 (M^+) and 173 ($M^+-NHC_6H_4NH_2$), confirm their structures. Upon treatment with boiling acetic acid, **8f** or **2d** afforded 10,10a,11,12-tetrahydrobenz-[*b*]indeno[1,2-*e*][1,4]diazepine-10,11-dione (**9**). The formation of the diazepine derivative **9** is in line with the work of Ried and Draisbach[9] on the condensation of ethyl 2-oxocyclohexanecarboxylate with *o*-phenylenediamine.



(8) a: $R^1=R^2=H$
b: $R^1=H$, $R^2=CH_3$-p
c: $R^1=H$, $R^2=Cl$-p
d: $R^1=H$, $R^2=Cl$-m
e: $R^1=H$, $R^2=NO_2$-p
f: $R^1=NH_2$, $R^2=H$
g: $R^1=H$, $R^2=NH_2$-p
h: $R^1=H$, $R^2=COCH_3$-p
i: $R^1=H$, $R^2=COOC_2H_5$-p



(9)



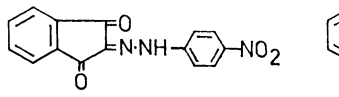
(10)

The synthesis of quinolones by the condensation between *β*-keto esters and primary aromatic amines was described in earlier reports.[10,11] The nature of the product depends on the conditions. Compounds **2** and **8** are considered to be suitable starting materials for the synthesis of new quinolones fused to the indanone and/or indenone nuclues. Thus, **8d** was cyclized according to the Knorr quinolone synthesis[10] by hot sulfuric acid to give 3-chloro-5*H*-indeno[2,1-*c*]quinoline-6,7-dione (**10**).

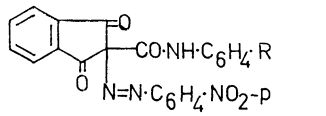
The structure of the quinolone derivative (**10**) was confirmed by elemental analysis and IR spectrum. An attempt to cyclize **2a** under conditions similar to those for Conrad-Limpach synthesis[11] gave only a black, intractable tar.

Treatment of **1** with diazotised *p*-nitroaniline or bis-(diazotised) benzidine, in basic medium, gave the expected result of a Japp-Klingemann reaction.[12] The ethoxycarbonyl group was cleaved with the formation of 1,2,3-indantrione 2-*p*-nitrophenylhydrazone (**11**), and 4,4'-bis(1,3-dioxo-2-indanylidenehydrazono) biphenyl (**12**) respectively. The melting point of **11** and **12** showed no depression on being mixed with an authentic sample.[13]

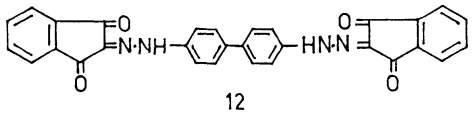
On the other hand, coupling of **8a**—**b** with diazotised



11



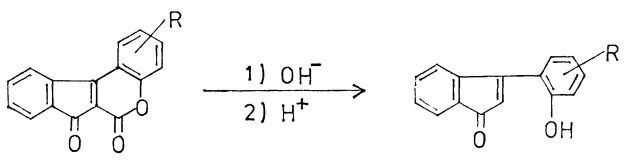
13 a: R=H
b: R=CH_3-p



12

p-nitroaniline in methanol afforded 2-(*p*-nitrophenylazo)-1,3-dioxo-2-indancarboxanilide (**13a**) and 2-(*p*-nitrophenylazo)-1,3-dioxo-4'-methyl-2-indancarboxanilide (**13b**), respectively. Structure of **13** was confirmed by elemental analysis and IR spectra. The coupling of *β*-keto anilides has been reported.[14] The formation of the azo derivatives (**13**) is in line with the work of Linstead and Wang[15] on the coupling of 2-oxocyclohexan-1-carboxanilide with benzenediazonium chloride.

The observation that *β*-keto esters react with phenols to form coumarin derivatives (Pechmann reaction),[16] prompted us to investigate the behaviour of **1** under similar reactions in order to obtain 3-arylindenones. Compound **1** was treated with resorcinol[17] and *p*-cresol in ethanolic-hydrogen chloride to give 3-hydroxy-, and 2-methyl-benz[*b*]indeno[1,2-*d*]pyran-6,7-diones (**14a**—**b**), respectively. Alkaline hydrolysis of **14**, followed by treatment with boiling hydrochloric acid afforded 3-(2-hydroxyphenyl)-1-indenones (**15**). The structure of indenones **15** has been confirmed by analytical data and IR spectroscopy.



14 15

a: R=OH-m
b: R=CH_3-p

Experimental

All melting points were determined in a capillary and are uncorrected. IR spectra were determined on KBr discs with a Unicam SP 2000 Infrared spectrophotometer. The NMR spectra in $CDCl_3$ solution were obtained on a Varian 60 MHz spectrometer with TMS as an internal standard. The mass spectra were obtained on MS 902 at 70 eV using a direct insertion probe at 250—300 °C.

Ethyl 3-Hydroxy-1-(arylimino)-2-indenecarboxylate (2). *General Procedure*: A mixture of **1** (1.1 g, 0.005 mol) and appropriate primary aromatic amine (2 g) in 10 ml acetic acid was heated at 60 °C for 5 min. After cooling and dilution with water the solid was filtered and crystallized from ethanol.

Ethyl 3-Hydroxy-1-(phenylimino)-2-indenecarboxylate (2a): Orange crystals, yield, 0.8 g (54.8%), mp 152 °C. IR: 3080, 1705, 1670, 1630, and 1040 cm^{-1}. Found: C, 73.81; H, 5.36; N, 4.31%. Calcd for $C_{18}H_{15}NO_3$: C, 73.70; H, 5.15; N, 4.77%.

Ethyl 3-Hydroxy-1-(p-methoxyphenylimino)-2-indenecarboxylate (2b): Brown crystals, yield, 1 g (62.5%), mp 162 °C (dec).

IR: 3085, 1700, 1665, 1630, and 1045 cm^{-1}. Found: C, 70.34; H, 5.11; N, 4.10%. Calcd for $C_{19}H_{17}NO_4$: C, 70.57; H, 5.30; N, 4.32%.

*Ethyl 3-Hydroxy-1-(p-tolylimino)-2-indenecarboxylate (**2c**)*: Orange crystals, yield, 0.9 g (58.8%), mp 174 °C. IR: 3080, 1700, 1660, 1625, and 1050 cm^{-1}. Found: C, 74.48; H, 5.61; N, 4.33%. Calcd for $C_{19}H_{17}NO_3$: C, 74.24; H, 5.57; N, 4.55%.

*Ethyl 3-Hydroxy-1-(o-aminophenylimino)-2-indenecarboxylate (**2d**)*: Dark brown crystals, yield, 0.5 g (32.4%), mp>300 °C. IR: 3280, 3100, 1690, 1650, and 1625 cm^{-1}. Found: C, 69.82; H, 5.13; N, 8.88%. Calcd for $C_{18}H_{16}N_2O_3$: C, 70.11; H, 5.20; N, 9.08%.

*1-Oxo-3-(p-tolylimino)-4'-methyl-2-indancarboxanilide (**3**)*: From 1.1 g (0.005 mol) of **1** and 3 g of *p*-toluidine dissolved in (10 ml) acetic acid, **3** was obtained by the same procedure as that for **2**. Recrystallization from methanol gave 0.8 g (43%) of **3** as a brown crystalline powder; mp 205 °C (dec). IR: 3400, 2920, 1670, 1630, 1540, 1525, and 1250 cm^{-1}; NMR ($CDCl_3$): δ; 2.6 (1H s, -CO-CH-CO-); 2.41 (6H s, $C_6H_4CH_3$); 7.33 (8H s, $C_6H_4CH_3$); 7.62 (4H d, *o*-substituted C_6H_4) and 8.1 ppm (1H s, -NH-CO-). Found: C, 78.31; H, 5.50; N, 7.31%. Calcd for $C_{24}H_{20}N_2O_2$: C, 78.23; H, 5.47; N, 7.60%.

*Diethyl 3,3'-Dihydroxy-1,1'-(o-phenylenedinitrilo)di-2-indenecarboxylate (**5**)*: A mixture of 1.1 g (0.005 mol) of **1** and 0.3 g (0.0025 mol) of *o*-phenylenediamine in 10 ml acetic acid was heated with stirring for 15 min. The reaction mixture gave a dark brown solid (0.8 g) on dilution with water, which on treatment with boiling ethanol gave **5** as an insoluble red solid (0.5 g). Recrystallization from acetic acid afforded **5** as red crystals; mp>300 °C. IR: 3100, 1700, 1650, and 1635 cm^{-1}. MS, *m/e* 362 (base, M^+-2 $COOC_2H_5$), 274, 104, and 76. Found: C, 71.00; H, 4.95; N, 5.37%. Calcd for $C_{30}H_{24}N_2O_6$: C, 70.85; H, 4.75; N, 5.51%.

Concentration of the ethanolic filterate after separation of **5** gave 0.14 g of **2d** as dark brown crystals, the IR spectrum of which was identical with that of the authentic sample of **2d** obtained above.

*Diethyl 3,3'-Dihydroxy-1,1'-(p-phenylenedinitrilo)di-2-indenecarboxylate (**6**)*: From 1.1 g (0.005 mol) of **1** and 0.3 g (0.0025 mol) of *p*-phenylenediamine dissolved in 10 ml acetic acid, **6** was obtained according to the same procedure as that for **5**. Recrystallization from benzene-ethanol gave 0.7 g (27.5%) of **6** as dark brown powder; mp>300 °C. IR: 3105, 1690, 1655, and 1640 cm^{-1}. MS, *m/e* 362 (base, M^+-2 $COOC_2H_5$), 274, 104, and 76. Found; C, 70.91; H, 4.66; N, 5.40%. Calcd for $C_{30}H_{24}N_2O_6$: C, 70.85; H, 4.75; N, 5.51%.

Diethyl 7,10:19,22-Dietheno-5,24:12,17-dimethanodibenzo [c,m]-*[1,6,11,16]tetraazacycloeicosene-25,28-dicarboxylate (**7**)*: A mixture of 1.1 g (0.005 mol) of **1** and 0.6 g (0.006 mol) of *p*-phenylenediamine in 20 ml acetic acid was heated at 80 °C for 1 h with stirring. The reaction mixture was diluted with water, and filtered to give dark green material (0. 6g). Crystallization from ethanol-benzene (1:1) afforded **7** as a dark green powder (0.4 g); mp 255 °C, IR: 1725, 1665, and 1200 cm^{-1}; MS, *m/e* 490 ($M^+-2(OC_2H_5)$), 366, 169, and 76. Found: C, 74.60; H, 4.90; N, 9.42%. Calcd for $C_{36}H_{28}N_4O_4$: C, 74.46; H, 4.86; N, 9.65%.

*1,3-Dioxo-2-indancarboxanilides (**8a—i**)*: A mixture of 1.1 g (0.005 mol) of **1** and 0.0055 mol of the appropriate primary aromatic amine in 50 ml toluene was refluxed for 30 min. The solid obtained after concentration and cooling was filtered. Recrystallization from benzene gave **8a—i** as yellow crystalline solids. The results are summarized in Table 1.

*Formation of **3** from **8b***: A mixture of 0.28 g (0.001 mol) of **8b** and 0.11 g (0.001 mol) of *p*-toluidine in 10 ml acetic acid was heated at 70 °C for 10 min. The solid obtained on dilution with water was filtered and crystallized from benzene to give 0.08 g of **3**. The IR spectrum of the product was identical with that of the authentic sample of **3**.

10,10a,11,12-Tetrahydrobenz [b] *indeno* [1,2-e] [1,4] *diazepine-10,11-dione (**9**)*: 0.5 g of **8f** or **2d** was refluxed in 30 ml acetic acid. The dark red solid obtained on cooling was filtered and recrystallized from acetic acid to give 0.22 g of **9** as a red crystalline solid; mp>300 °C. IR: 3210, 1705, 1650, 1630, and 1575 cm^{-1}. Found: C, 73.34; H, 4.10; N, 10.31%. Calcd for $C_{16}H_{10}N_2O_2$: C, 73.27; H, 3.84; N, 10.68%.

*3-Chloro-5H-indeno[2,1-c]quinoline-6,7-dione (**10**)*: 0.5 g of **8d** was heated with 20 ml of H_2SO_4 (85%) in an oil bath at 110 °C for 1 h. After cooling, the reaction mixture was diluted with 80 ml cold water, and the brown solid obtained was filtered. Crystallization from benzene-ethanol gave 0.16 g of **10**; mp>300 °C. IR: 2900, 1725, 1670, 1620, 1590, and 1450 cm^{-1}. Found: C, 67.91; H, 3.01; N, 4.66%. Calcd for $C_{16}H_8NO_2Cl$: C, 68.22; H, 2.86; N, 4.97%.

*Coupling of **1** with p-Nitrobenzenediazonium Chloride and Bis-(diazotised) Benzidine*: Diazotised *p*-nitroaniline (0.005 mol), or bis(diazotised) benzidine (0.0025 mol), was added with stirring to a cold solution of **1** (0.005 mol) in 50 ml 2.5% aq NaOH. Sodium acetate (2 g) was added, and the reaction mixture was left to stand under cooling overnight. The brown solid obtained was crystallized from acetic acid to give **11** and **12**, respectively, in a 75% yield. Compounds **11** and **12** were identical with the corresponding authentic samples prepared from 1,3-indandione by the method of Das and Ghosh.[13]

*Coupling of **8a—b** with Diazotised p-Nitroaniline*: Diazotised *p*-nitroaniline (0.003 mol) was added with stirring to a cold solution of **8a** or **8b** (0.003 mol) in 60 ml methanol, followed by addition of lg of sodium acetate in 5 ml water. The

Table 1. 1,3-Dioxo-2-indancarboxanilides (**8**)

Compound No.	Mp °C	Yield %	Formula	Found (%)			Calcd (%)			IR(KBr) ν_{max}/cm^{-1}
				C	H	N	C	H	N	
8a	148	61	$C_{16}H_{11}NO_3$	72.11	4.23	5.00	72.44	4.18	5.27	3402, 1680, 1560
8b	188	43	$C_{17}H_{13}NO_3$	73.31	4.37	5.15	73.10	4.69	5.01	3400, 1685, 1560
8c	200	77	$C_{16}H_{10}NO_3Cl$	64.33	3.41	4.52	64.10	3.36	4.67	3390, 1680, 1565
8d	170	70	$C_{16}H_{10}NO_3Cl$	64.25	3.51	4.53	64.10	3.36	4.67	3390, 1680, 1560
8e	228	53	$C_{16}H_{10}N_2O_5$	61.56	3.50	8.95	61.93	3.24	9.03	3400, 1685, 1560
8f	>300	45	$C_{16}H_{12}N_2O_3$	68.43	4.11	10.03	68.56	4.31	9.99	3400, 1685, 1550
8g	280	41	$C_{16}H_{12}N_2O_3$	68.37	4.20	10.01	68.56	4.31	9.99	3400, 1680, 1550
8h	185	66	$C_{18}H_{13}NO_4$	70.26	4.31	4.38	70.35	4.26	4.55	3400, 1700, 1555
8i	161	52	$C_{19}H_{15}NO_5$	67.41	4.20	4.00	67.64	4.48	4.15	3400, 1740, 1560

solid obtained was crystallized from methanol to give **13a—b**, respectively.

*2-(p-Nitrophenylazo)-1,3-dioxo-2-indancarboxanilide (**13a**)*: Orange crystals, yield, 0.19 g (15.3%), mp 132 °C. IR: 3060, 1700, 1650, 1580, 1340, and 890 cm^{-1}. Found: C, 63.61; H, 3.17; N, 13.68%. Calcd for $C_{22}H_{14}N_4O_5$: C, 63.76; H, 3.40; N, 13.52%.

*2-(p-Nitrophenylazo)-1,3-dioxo-4'-methyl-2-indancarboxanilide (**13b**)*: Yellow crystals, yield, 0.16 g (12.5%), mp 158 °C. IR: 3065, 1700, 1640, 1585, 1345, and 890 cm^{-1}. Found: C, 64.22; H, 3.68; N, 12.88%. Calcd for $C_{23}H_{16}N_4O_5$: C, 64.48; H, 3.76; N, 13.07%.

*Condensation of **1** with Phenols*: Cold saturated ethanolic hydrogen chloride (10 ml) was added with stirring to a mixture of 2 g of **1** and resorcinol or *p*-cresol (6 g) in 30 ml ethanol. After 24 h the yellow crystalline product was filtered, washed with ethanol and acetone to give **14a—b** respectively.

3-Hydroxybenz[b]*indeno*[*1,2*-d]*pyran-6,7-dione (**14a**)*: Yellow crystals, yield, 1 g (37.8%). Its mp and IR spectrum were identical with those of an authentic sample.[17]

2-Methylbenz[b]*indeno*[*1,2*-d]*pyran-6,7-dione (**14b**)*: Recrystallized form ethanol, pale yellow crystals, yield, 0.5 g (19%), mp 265 °C. IR: 1740, 1720, 1590, and 1470 cm^{-1}. Found: C, 77.67; H, 4.00%. Calcd for $C_{17}H_{10}O_3$: C, 77.85; H, 3.84%.

*3-(2-Hydroxyphenyl)indenones (**15**)*: A solution of aq NaOH (5%, 20 ml) was added to boiling solution of **14a** or **14b** (0.002 mol) in ethanol (40 ml). The reaction mixture was refluxed for 1 h, acidified with dil hydrochloric acid and the resulting precipitates were filtered and dried to give **15a**, 0.15 g (32%). Recrystallization from methanol afforded greenish-yellow crystals; mp 177 °C. IR: 3410, 1740, 1630, 1375, 1270, and 1180 cm^{-1}. Found: C, 75.46; H, 4.06%. Calcd for $C_{15}H_{10}O_3$: C, 75.62; H, 4.23%.

*3-(2-Hydroxy-*m*-tolyl)indenone (**15b**)*: Orange crystals from methanol, yield, 0.2 g (42%), mp 147 °C. IR: 3400, 1735, 1620, 1370, 1270, and 1185 cm^{-1}. Found: C, 81.16; H, 5.22%. Calcd for $C_{16}H_{12}O_2$: C, 81.33; H, 5.12%.

We wish to express our sincere gratitude to Dr. A. M. Dawidar for the NMR and mass measurements.

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