*Tetrahedron* Vol. 38, No. 11, pp. 1657 to 1660, 1982
Printed in Great Britain.

0040-4020/82/111657-04$03.00/0
Pergamon Press Ltd.

# 2-AROYLBENZOIC ACIDS—VIII[1]

## THE SYNTHESIS AND SOME REACTIONS OF 2-AROYLBENZOIC ACIDS FROM *m*-CHLOROPHENOL

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(*Received in UK* 23 *July* 1981)

**Abstract**—The syntheses of the novel 2-(4'-chloro-2'-hydroxybenzoyl) benzoic acid **1** and 2-(2'-chloro-4'-hydroxybenzoyl)benzoic acid **2** are described and some of their reactions are given.

In our continuing studies on 2-aroylbenzoic acids[1] we have devised optimal conditions for the syntheses of two new 2-aroylbenzoic acids. They were prepared by the condensation of *m*-chlorophenol with phthalic anhydride in the presence of anhydrous $AlCl_3$ in *sym*-tetrachloroethane. The yield of 2-(4'-chloro-2'-hydroxybenzoyl) benzoic **1** was *ca.* 48% and that of 2-(2'-chloro-4'-hydroxybenzoyl) benzoic acid **2** was 22%.

The structure of acids **1**, **2** has been elucidated by physico-chemical methods: derivatives (**1a–1f**, **2a–2e**), elemental analyses, UV, IR and NMR spectra. UV spectra of compounds **1**, **2** in ethanol have confirmed that **1** has two absorption bands, however, **2** has only one band as in similar cases.[2] The mass spectral (MS) fragmentation of acid **1** is shown and interpreted in Fig. 1.

The structural X-ray investigations of Skrzat[3–5] have shown, that chlorine in 4,[3] 5,[4] and 3[5] positions of **1** has an influence on the orientation of the -$C_6H_5COOH$ system in relation to the benzoyl system. This may be the reason for the different reactivity of these compounds, e.g. acid **1** cyclizes only with difficulty in conc. $H_2SO_4$ to derivatives of anthraquinone, in comparison to 2-(3'-chloro-2'-hydroxybenzoyl) benzoic acid,[5] which cyclizes much easier.

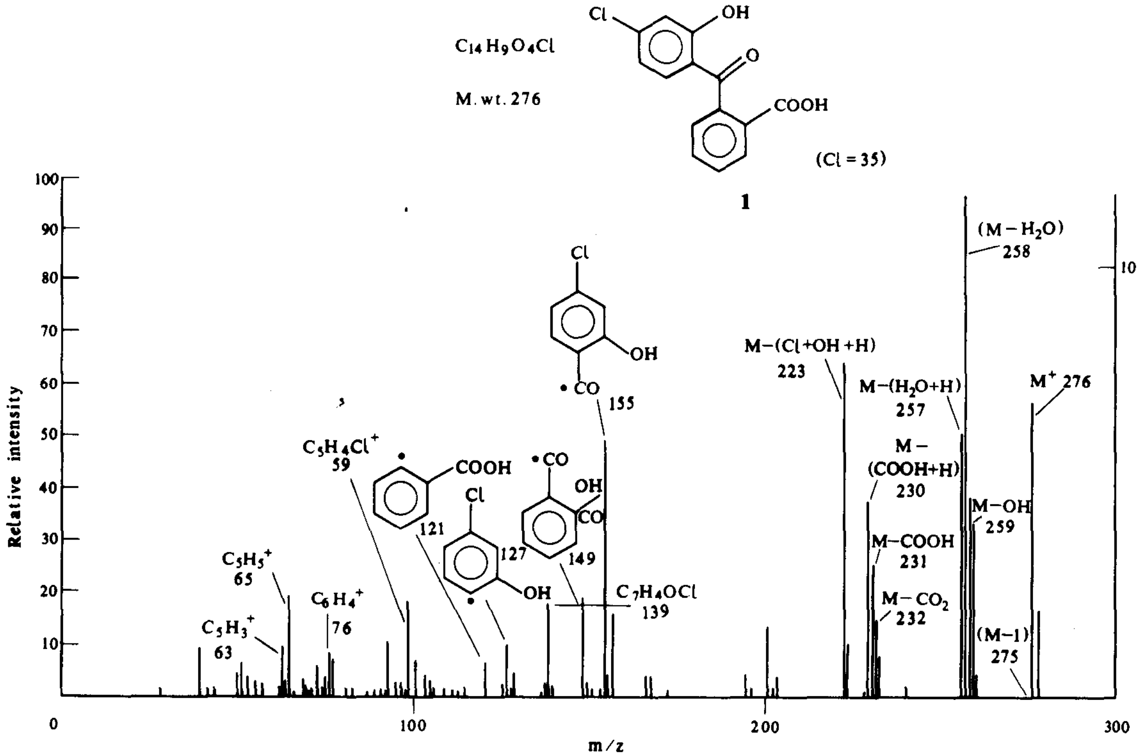
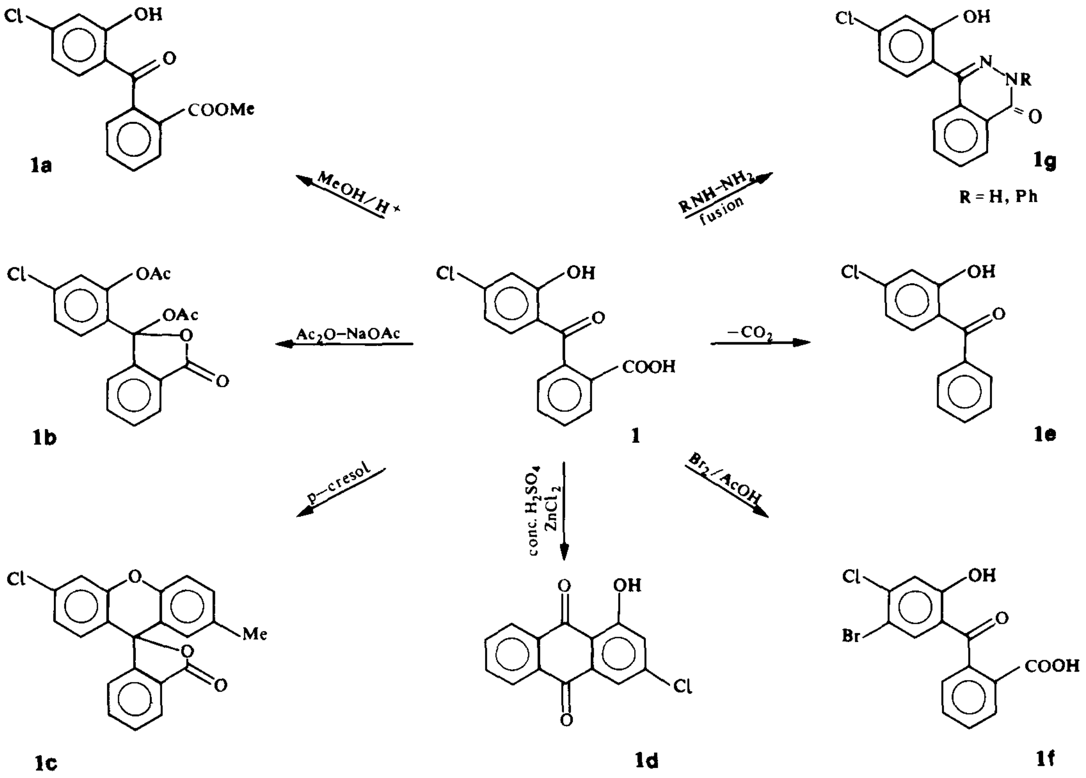
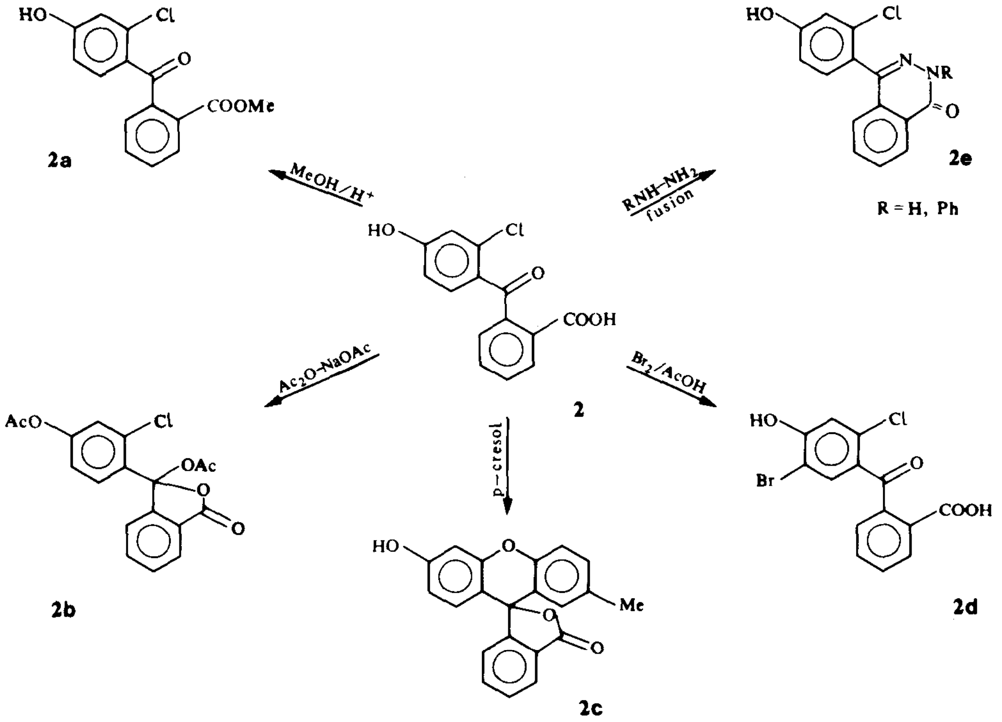


Fig. 1. The mass spectral fragmentation of acid **1**.


1a
1b
1c
1
1d
1e
1f
1g
R = H, Ph
MeOH/H+
RNH-NH2 fusion
Ac2O-NaOAc
−CO2
p-cresol
conc. H2SO4 ZnCl2
Br2/AcOH


2a
2b
2c
2
2d
2e
R = H, Ph
MeOH/H+
RNH-NH2 fusion
Ac2O-NaOAc
p-cresol
Br2/AcOH

## EXPERIMENTAL

M.ps were taken in open glass capillaries and are uncorrected. UV spectra were measured with a Unicam SP 1750 instrument in EtOH. IR spectra were recorded within the range of 625–3800 cm$^{-1}$ with an Unicam SP-1025 recording spectrophotometer using KBr (pellets), and were corrected based on a polystyrene spectrum, bands are reported as being strong (s), medium (m), weak (w) and broad (b). NMR spectra were run on a Varian HA 100 instrument and MS-spectrum on a Varian M 66 spectrophotometer. Elemental analyses were performed in the Micro-analytical Laboratory, Cairo University, Egypt.

2-(*4'-Chloro-2'-hydroxybenzoyl)benzoic acid* **1**. 3-Chlorophenol (13 g, 0.1 mole) and phthalic anhydride (14.8 g, 0.1 mole) in 1, 1, 2, 2-tetrachloroethane (400 ml) were heated with stirring with anhydrous, finely powdered $AlCl_3$ (40 g, 0.3 mole) in the following manner: the first portion of $AlCl_3$ (13.3 g, 0.1 mole) was added at 90° over 1 h and the second one (26.6 g, 0.2 mole) at 100° during a period of 1 h. After the addition was completed, the temperature of the oil bath was raised to 130–140° and the mixture was kept for 5 h at this temperature with continuous stirring. The mixture was cooled and the resulting grey semi-solid mixture was treated with 20 mil dil. HCl (1:1) to decompose the formed complex. After a vigorous evolution of gaseous HCl, the solvent was removed by steam distillation. A residual mixture was decanted leaving a dark brown mass, which was washed with water and extracted several times with boiling $Na_2CO_3$ aq. (20%). The combined alkaline extracts were filtered off, acidified carefully with dil. HCl (1 : 1) with vigorous stirring (up to pH 6–7) resulting in a grey amorphous mass, consisting of a mixture of phthaleins but mainly 3, 3-bis (2'-chloro-4'-hydroxyphenyl) phthalide.

The solid was filtered off and the orange-yellow filtrate was acidified with conc HCl to pH 2 yielding a cream mass (7.1 g, 70%). This ppt consisted of a mixture of two isomers of acids *o*-(OH) **1** and *p*-(OH) **2**. These isomers were separated by boiling the ppt with water several times and filtred hot according to Gronowska's method.[6]

The *o*-isomer (acid **1**) being insoluble in water (4.0 g) was dried and purified by several recrystallizations from EtOH yielding almost colourless crystals, m.p. 203° UV: $\lambda_{max}$ ($\epsilon_{max}$): 263 (15541), 332 (6484) nm; $\lambda_{min}$ ($\epsilon_{min}$): 246 (10297), 292 (3337) nm. IR: 1410 s, 1280 s (C–O and O–H dimeric COOH), 2650 w, 2520 w (OH, dimeric COOH), 1695 s (C=O, aromatic conjugated ketone and COOH), 1630 s (C=O, aromatic ketone, intramolecular hydrogen bond), 1625 m, 1585 w, 1500 m (C=C, aromatic), 1350 w, 1295 m, 1150 s, (OH, phenolic), 865 m, 810 w, 790 s, 710 m, 660 m (aromatic, 1,3,4-trisubst. 740 m, 725 m (aromatic 1,3-disubst.) cm$^{-1}$. $^1$H NMR: $\delta$ 6.71–8.32 (m, 7 H, aromatic rings); 8.20 (s, 1 H, OH); 10.81 (s, 1 H, COOH) ppm. Its MS spectrum (Fig. 1) confirms its structure and gives all the expected fragments. (Found: C 60.7; H 3.4; Cl 13.1. Calc. for $C_{14}H_9O_4Cl$: C 60.9, H 3.8, Cl 12.9%).

*Methyl* 2-(*4'-chloro-2'-hydroxybenzoyl*) *benzoates* **1a**. A soln of **1** (0.5 g) in MeOH (40 ml) containing a few drops of conc $HSO_4$ was refluxed for 5 h. After distillation of most of the alcohol, the mixture was poured into ice-water giving a crude solid of **1a** (0.8 g). It was purified by several recrystallizations from EtOH and colourless crystals were obtained, m.p. 149°. UV: $\lambda_{max}$ ($\epsilon_{max}$): 264 (17088), 324 (6664); $\lambda_{min}$ ($\epsilon_{min}$): 248 (11962), 294 (3589) nm. IR: 1730 s (C=O, ester group), 1635 s (C=O, aromatic conjugated ketone, intramolecular hydrogen bond), 3370 sb, 1290 sb, 1226 w, 1135 s (OH, phenolic) cm$^{-1}$. $^1$H NMR: $\delta$ 3.78 (s,3 H, $COOCH_3$); 6.68–7.95 (m, 7 H, aromatic rings), *ca* 8.0 (s, 1 H, OH) ppm. Found: 62.4% C; H 4.0; Cl 12.6. Calc. for $C_{15}H_{11}O_4Cl$: C 62.0; H 3.8; Cl 12.2%).

2-*Acetoxy*-3-(*2'-acetoxy-4'-chlorophenyl*) *phthalide* **1b**. This compound was prepared by O-acetylation of **1** (0.5 g) by boiling it with $Ac_2O$ (20 ml) and anhyd NaOAc (2 g) for 3 h. The mixture was poured onto crushed ice and the solid that separated out was filtered and washed water (yield 90%). After recrystallization from dil EtOH colourless crystals were obtained, m.p. 160°. UV: $\lambda_{max}$ ($\epsilon_{max}$): 265 (9270); $\lambda_{min}$ ($\epsilon_{min}$) 255 (8927) nm. IR: 1800 s (C=O, $\gamma$-lactone), 1730 s (C=O, acetate), 1375 m ($CH_3$, acetate), 1180 s, 1175 s, 1210 s (C–O, acetate), 1240 s, 1090 m, 1070 m, 945 s, 918 m (C–O–C, $\gamma$-lactone), 1045 s, 1020 m (C–O–C, ester) cm$^{-1}$. $^1$H NMR: $\delta$ 2.07 (s, 3 H, 3-$OCOCH_3$); 2.25 (s, 3 H, 2'–$OCOCH_3$), 7.21–8.11 (m, 7 H, aromatic rings) ppm. Found: C 60.1 H 3.5, Cl 9.7. Calc. for $C_{18}H_{13}O_6Cl$: C 59.9, H 3.6, Cl 9.8%).

*6'-Chloro-2'-methylfluoran* **1c**. A mixture of **1** (0.5 g) and 0.7 g *p*-cresol and 1.0 g $ZnCl_2$ was heated in an oil bath at 110–120° for 8 h. The resin-like product thus obtained was dissolved in small amount of hot glacial AcOH, then poured into cold water with stirring. The title compound separated as amorphous solid was filtered, washed with dil NaOH aq (5%), then with water and dried (yield 0.9 g). Purification by several recrystallizations from EtOH gave pale cream crystals, m.p. 220°. IR: 1770 s (C=O, $\gamma$-lactone); 1610 m, 1580 m, 1490 m, 1425 (C=, aromatic); 1330 m, 1290 m, 1265 m, 1250 m (C–O–C, aromatic, etheral), 1165 w, 1100 w, 1080 m (C–H, aromatic trisubst asym), 1015 w, 950 m (C–O–C, aromatic, $\gamma$-lactone), 1140 w, 1110 s, 1085 m (C–H, aromatic trisubst. ring) cm$^{-1}$. $^1$H NMR: $\delta$ 2.20 (s, 3 H, 2'–$CH_3$), 6.61–7.82 (*m*, 10 H, aromatic rings) ppm. (Found: C 72.4, H 3.8, Cl 9.8. Calc. for $C_{21}H_{13}O_3Cl$: C 72.3, H 3.7, Cl 10.2%).

3-*Chloro*-1-*hydroxyanthraquinone* **1d**. A mixture of **1** (1 g), conc $H_2SO_4$ (1 ml) and freshly fused anhyd $ZnCl_2$ was heated in an oil bath at 110–115° for 5h. After cooling, the mixture was poured into 200 ml ice-water and the product was filtered, dissolved in 10% NaOH aq, then filtered again. The alkaline filtrate was acidified with HCl to pH 2 giving (*ca* 0.1 g) of crude lemon-yellow anthraquinone derivative **1d**. After several crystallizations from dil EtOH yellowish crystals were obtained, m.p. 210°. UV: $\lambda_{max}$ ($\epsilon_{max}$) 227 (42911), 275 (10857); $\lambda_{min}$ ($\epsilon_{min}$) 217 (40921), 266 (9823) nm. IR: 3500 sb (OH, intermolecular association), 1800 s (C=O, quinone), 1600 s, 1620 s, 1585 s, 1570 s, 1512 w (C=C, aromatic), 1290 s, 2296 w (OH, phenolic), 1310 s, 1280 s, 1245 m, 1080 m, 960 m, 930 m, 778 s, 725 s cm$^{-1}$. (Found: C 65.3, H 2.8. Calc. for $C_{14}H_7O_3Cl$: C 65.0, H 2.7%).

2-(*5'Bromo-4'-chloro-2'-hydroxybenzoyl*) *benzoic acid* **1e**. This compound was prepared by refluxing 1 mole of **1** with a soln of $Br_2$ (1 mole) in glacial AcOH (30 ml) for 3 h. The mixture was poured onto crushed ice and the solid that separated out was filtered, washed with water and dried (yield *ca* 90%). Several crystallization from dil EtOH gave colourless crystals, m.p. 176°. UV: $\lambda_{max}$ ($\epsilon_{max}$) 273 (15490), 341 (6856); $\lambda_{min}$ ($\epsilon_{min}$) 264 (14474), 307 (3809) nm. IR: 2660 w, 2560 w (OH, dimeric COOH), 1695 s (C=O, aromatic conjugated COOH), 1685 s (C=O, aromatic ketone, intramolecular H-bond), 1600 m, 1580 w, 1570 w, 1550 m (C=C, aromatic), 1425 s, 1290 s (C–O and O–H, dimeric COOH), 1325 m, 1300 s, 1170 m (OH, phenolic), 1085 m, 1045 w, 910 mb (OH, dimeric COOH), 860 m 800 m, 775 s, 710 s, 690 m (aromatic 1, 2, 4, 5, tetrasubst.) cm$^{-1}$. $^1$H NMR: $\delta$ 7.21–8.22 (m, 6 H, aromatic rings) ppm. (Found: C 49.7, H 2.9. Calc. for $C_{14}H_8O_3BrCl$: C 49.5, H 2.8%).

*Methyl 2-(5'-bromo-4'-chloro-2'-hydroxybenzoyl)benzoate.* This ester was prepared by refluxing a soln of **1e** (0.3 g) with MeOH (40 ml) containing few drops of conc $H_2SO_4$ for 5h similarly as for **1a**.The yield 0.26 g. It was crystallized from dil EtOH to give colourless crystals, m.p. 125° UV: $\lambda_{max}$ ($\epsilon_{max}$) 265 (29560), 328 (11701); $\lambda_{min}$ ($\epsilon_{min}$) 249 (24017), 296 (8006) nm. IR: 1730 s (C=O, ester group), 1650 s (C=O, aromatic conjugated ketone, intramolecular H-bond), 1295 sb, 1220 m, 1140 s (OH, phenolic) cm$^{-1}$. (Found: C 51.2, H 2.9. Calc. for $C_{15}H_{10}O_3BrCl$: C 50.9, H 2.8%).

4-*Chloro*-2-*hydroxybenzophenone* **1f**. A soln of **1** (1 g) in freshly-distilled quinoline (15 ml) was refluxed for 5 min and then $Cu(AcO)_2 \cdot H_2O$ (0.15 g) was added in one portion and the mixture refluxed for 1h. After cooling, the mixture was extracted with ether several times and quinoline was separated by shaking the etheral soln with dil HCl (1:1).

The ether layer was washed with $H_2O$ several times, then dried over $MgSO_4$. After distillation of ether, the title compound was separated out (0.6 g) as a brown oil and it was identified as its 2, 4-dinitrophenylhydrazone which was crystallised from $CHCl_3$/MeOH mixture as red crystals, m.p. 253°. UV: $\lambda_{max}$ ($\epsilon_{max}$): 265 (16581), 394 (18077); $\lambda_{min}$ ($\epsilon_{min}$): 324 (7684) nm. IR: 3100 wb (NH group), 3300 m (OH), 1610 s (C=N), 1350 m ($NO_2$), 850 m cm$^{-1}$. (Found: C 53.8, H 3.2, N 12.9. Calc. for $C_{19}H_{13}N_4O_5Cl$: C 54.0, H 3.1, N 13.3%).

*Phthalazone derivatives* **1g**. (a) R = H. This ketone was prepared by fusion of **1** (1 mole) with $NH_2 \cdot NH_2 \cdot H_2O$ (99%) (3 moles) at 120–130° for 3 h. The mixture was cooled, diluted with water and the colourless solid that separated out was filtered, washed with water and dried (80% yield). It was purified by several crystallizations from EtOH to give pale brown plates, m.p. 269°. UV: $\lambda_{max}$ ($\epsilon_{max}$): 253 s (10570), 289 (9909); $\lambda_{min}$ ($\epsilon_{min}$) 266 (7929). IR: 2700–3300 sb (due to overlap of NH and OH groups), 3180 sb (NH), 1680 s (C=O, aromatic ketone, intramolecular H-bond), 1330 s, 1220 w, 1120 s (OH, phenolic), 1610 m, 1590 w, 1570 w, 1490 m (C=C, aromatic) cm$^{-1}$. (Found: C 61.8, H 3.5, N 10.1. Calc. for $C_{14}H_9N_2O_2Cl$: C61.6, H 3.3, N 10.3%).

(b) *R = Ph.* This compound was prepared by a similar procedure to (a) using $PhNH{\cdot}NH_2$. After dilution of the mixture with water and filtration of the deposited solid, it was boiled several times with dil HCl and decanted to remove the excess $PhNH \cdot NH_2$, filtered, washed with water and dried. It was purified by several crystallizations from EtOH to give colourless crystals, m.p. 221°. UV: $\lambda_{max}$ ($\epsilon_{max}$) 297 (13136), $\lambda_{min}$ ($\epsilon_{min}$) 274 (10991) nm. IR: 1650 s (C=O, aromatic ketone, intramolecular H-bond), 3030 w (C–H, aromatic), 1615 w, 1584 s, 1505 w, 1490 w (C=C, aromatic), 1290 w, 1310 m, 1120 w (OH, phenolic) cm$^{-1}$. (Found: C 69.1, H 4.1, N 7.8. Calc. for $C_{20}H_{13}N_2O_2Cl$: C 68.9, H 3.7, N 8.0%).

2-(2′-*Chloro-4′-hydroxybenzoyl) benzoic acid* **2**. From the water filtrate (after separation of **1**, *o*-OH isomer), the *p*-OH isomer (acid **2**) was crystallized as yellow crystals. It was purified by several crystallizations from dil EtOH as yellowish prisms, m.p. 230° (yield 1.6 g). UV: $\lambda_{max}$ ($\epsilon_{max}$) 269 (12927); $\lambda_{min}$ ($\epsilon_{min}$) 250 (10234) nm. IR: 3320 sb (OH, intermolecular association), 3025 w (C–H, aromatic), 2970 m, 2600 w, 930 wb (OH, dimeric COOH), 1695 s (C=O, aromatic conjugated ketone and COOH), 1620 m, 1580 s, 1570 w, 1485 w, 1460 w (C=C, aromatic), 1280 sb, 1240 s, 1130 w, 1120 s (OH, phenolic), 860 m, 820 m, 800 s (aromatic 1, 3, 4-trisubst.) 740 m, 725 m (aromatic 1, 3-disubst.) cm$^{-1}$. $^1$H NMR: $\delta$ 6.37–7.70 (m, 7 H, aromatic rings), 8.91 (s, 1 H, OH), 10.61 (s, 1 H, COOH) ppm. (Found: C 61.2, H 3.4, Cl 13.0. Calc. for $C_{14}H_9O_4Cl$: C 60.8, H 3.3, Cl 12.8%).

*Methyl* 2-(2′-*chloro-4′-hydroxybenzoyl) benzoate* **2a**. A mixture of **2** (0.3 g), MeOH (30 ml) and a few drops of conc $H_2SO_4$ was refluxed for 5 h, and isolated as **1a**. The yield 0.22 g. Several crystallizations from dil EtOH afforded colourless crystals, m.p. 140°. UV: $\lambda_{max}$ ($\epsilon_{max}$) 265 (17431), 325 (7026); $\lambda_{min}$ ($\epsilon_{min}$) 248 (12161) 293 (3648) nm. IR: 3500 sb (OH, intermolecular association), 1730 s (C=O, ester), 1650 s (C=O, aromatic conjugated ketone), 1290 sb (OH, phenolic) cm$^{-1}$. $^1$H NMR: $\delta$ 3.71 (s, 3 H, $CH_3$), 6.61–8.23 (m, 7 H + 1 H, aromatic rings and OH group) ppm. (Found: C 61.9, H 3.7, Cl 11.9 Calc. for $C_{15}H_{11}O_4Cl$: C 62.0, H 3.8, Cl 12.2%).

2-*Acetoxy*-3-(4′-*acetoxy-2′-chlorophenyl) phthalide* **2b**. A routine *o*-acetylation of **2** (0.5 g) with boiling $Ac_2O$-AcONa was carried out as **1b**. The yield 0.51 g, m.p. 160° (from dil EtOH colourless crystals). UV: $\lambda_{max}$ ($\epsilon_{max}$) 255 (8412) nm. IR: 1800 (C=O, $\gamma$-lactone), 1750 s (C=O, acetate), 1380 m ($CH_3$, acetate), 1215 s (C–O, acetate), 1240 s, 1080 m, 1070 m, 945 s, 920 m (C–O–C, $\gamma$-lactone), 1040 m, 1027 m (C–O–C, ester) cm$^{-1}$. $^1$H NMR: $\delta$ 2.13 (s, 3 H, 3-$OCOOH_3$), 2.32 (s, 3H, 4′-$OCOCH_3$), 7.01–8.12 (m, 7 H, aromatic rings) ppm. (Found: C 60.2, H 3.8, Cl 9.9. Calc. for $C_{18}H_{13}O_6Cl$: C 59.9, H 3.6, Cl 9.8%).

6′-*Hydroxy-2′-methylfluoran* **2c**. 3-Arylphthalidylation of 4-methylphenol (0.35 g) with **2** (0.5 g) carried out at 130–135° for 5 h with an excess of anhyd $ZnCl_2$ as condensing agent afforded 0.7 g of a crude sample of **2c**. After recrystallization from dil AcOH, crystals were obtained, m.p. 230°. IR: 2960 m, 2930 m, 2860 w ($CH_3$, stretching), 1765 s (C=O, aromatic conjugated $\gamma$-lactone) 1290 s, 1250 s, 1230 s (C–O–C, aromatic ether), 1105 s, 1090 s, 930 s, 920 m (C–O–C, aromatic and $\gamma$-lactone), 1610 m, 1500 m, 1430 m (C=C, aromatic), 1140 w, 1110 s, 1080 m (C–H, aromatic trisubst.), 3450 s (OH) cm$^{-1}$. (Found: 76.5, H 4.2. Calc. for $C_{21}H_{14}O_4$: C 76.4, H 4.2%).

2-(5′-*Bromo-2′-chloro-4′-hydroxybenzoyl) benzoic acid* **2d**. This compound was prepared by bromination of **2** (1 mole) by refluxing with soln $Br_2$ (1 mole) in the AcOH (20 ml) for 3 h as for **1e**. The yield 83%, m.p. 1750 (from dil EtOH colourless crystals). UV: $\lambda_{max}$ ($\epsilon_{max}$) 262 (14604), 334 (6918), $\lambda_{min}$($\epsilon_{min}$) 254 (14220) nm. IR: 3325 sb (OH, intermolecular association), 2975 m, 2610 m (OH, dimeric COOH), 1690 s (C=O, aromatic conjugated ketone and COOH), 1285 sb, 1245 s, 1125 m (OH, phenolic), 855 m, 810 m, 775 s, 655 m (aromatic 1, 3, 4-trisubst.) cm$^{-1}$. (Found: C 49.8, H 2.8. Calc. for $C_{14}H_8O_3BrCl$: C 49.5, H 2.6%).

*Phthalazone derivatives* **2e**, (a) R = H. This compound was obtained by fusion of **2** as in the case of **1** and the yield was 87.3%. After recrystallization from EtOH yellow crystals were obtained, m.p. 221°. UV: $\lambda_{max}$ ($\epsilon_{max}$) 253 (16350), 285 (14307); $\lambda_{min}$ ($\epsilon_{min}$) 267 (12603) nm. IR: 1700 s (C=O, aromatic CO group), 2900–3080 sb (NH group), 3200–3300 sb (OH group), 1330 s, 1220 w, 1133 w (OH, phenolic), 1617 m, 1590 w, 1570 s, 1490 m (C=C, aromatic) cm$^{-1}$. (Found: C 61.4, H 3.5, N 10.5. Calc. for $C_{14}H_9N_2O_2Cl$: C 61.6, H 3.3, N 10.3%).

(b) R=Ph. This ketone was prepared from **2** and $PhNH{\cdot}NH_2$ and carrying out the same procedure as for **1** with yield 85%. After recrystallization from EtOH, colourless crystals were obtained, m.p. 206°. UV: $\lambda_{max}$ ($\epsilon_{max}$) 298 (12808), $\lambda_{min}$ ($\epsilon_{min}$) 277 (11319) nm. IR: 3380 sb (OH, intermolecular association), 1652 s (C=O, aromatic ketone), 3030 w (C–H, aromatic), 1625 m, 1580 s, 1571 s, 1515 w, 1490 w (C=C, aromatic), 1288 sb, 1244 s, 1137 w, 1122 s (OH, phenolic) cm$^{-1}$. (Found: C 69.0, H 3.8, N 8.4. Calc. for $C_{20}H_{13}N_2O_2Cl$: C 68.9, H 3.7, N 8.0%).

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