An Efficient Synthesis of Benzofluorenes Via α-Alkoxycarbonyldiarylmethyl Cations

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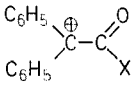
Rearrangements of α-alkoxycarbonyldiarylmethyl cations lead to 9-fluorenecarboxylic esters. Decarboxylation of these esters generates the fluorene derivatives **9a–f**. The precursors to the cations are the α-hydroxyesters **6a–g**. This conversion constitutes a facile synthesis of benzofluorenes.

We have recently reported the preparation and ^{13}C-N.M.R. spectra of α-ketodiphenylcarbocations of general formula **1**[1].



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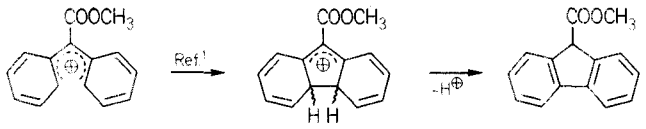
We also reported that in the case where X = OCH_3, at 0°C, this ion undergoes a 4π-electrocyclisation to give 9*H*-fluorene-9-carboxylic acid methyl ester **(2)** analogous to the cyclisation of pentadienyl cations previously reported[2].



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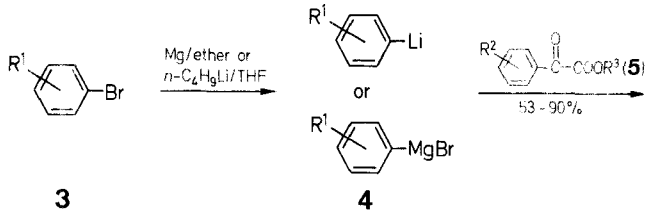
We have now extended this reaction to other α-alkoxycarbonyldiarylmethyl cations **7**, which undergo a similar cyclisation to give benzofluorenecarboxylic esters **8**. Decarboxylation of these esters provided the parent hydrocarbons **9**. The above three-step synthesis constitutes a short, high-yielding and synthetically flexible route to the cyclopentene-annellated polycyclic aromatic hydrocarbons,



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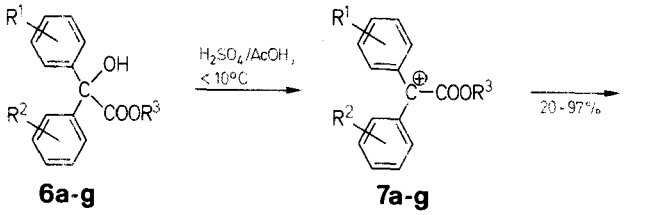
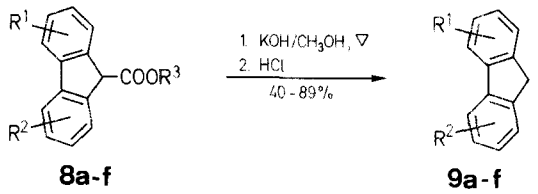


6a-g 7a-g



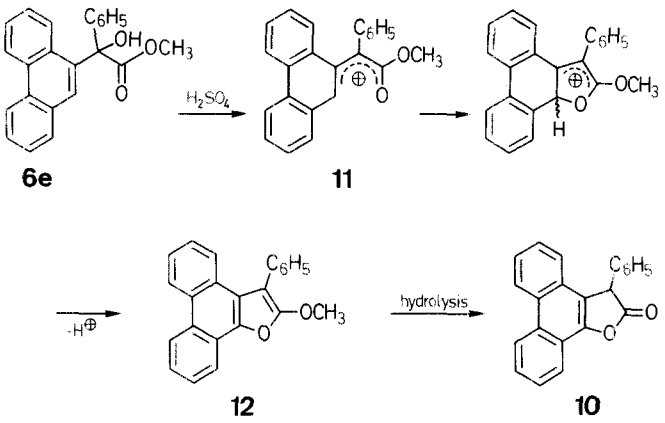
8a-f 9a-f

6	R^1	R^2	R^3	Products **8** (X = $COOR^3$) and **9** (X = H)
a			CH_3	
b			CH_3	
c			C_2H_5	
d			C_2H_5	
e			CH_3	
f			C_2H_5	
g			CH_3	intractable polymer

i.e., fluorenes, which have attracted recent attention[3]. The existing methods for preparation of fluorenes are long and tedious and usually provide low overall yields[4-9]. The starting diarylhydroxyacetic acid esters **6** were obtained from the reaction of suitable aryl Grignard or aryllithium reagents **4** with an alkyl α-oxoarylacetate **5** in yields ranging from 53 to 90%.

The hydroxy esters **6** are all white crystalline compounds exhibiting characteristic hydroxy (3500–3650 cm^{-1}) and carbonyl (1720–1740 cm^{-1}) absorption bands in the I.R. spectra. The ^{1}H-N.M.R. and mass spectra of the compounds **6a–g** were also consistent with the structures.

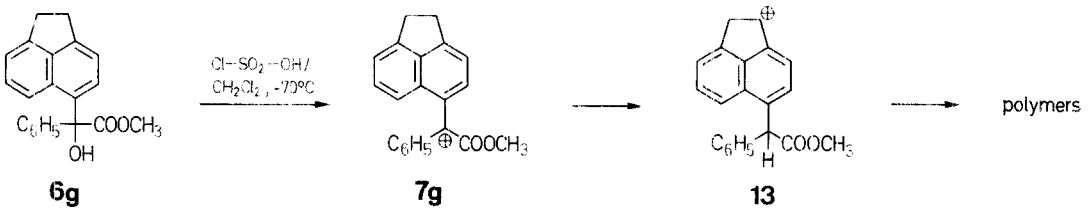
The hydroxy esters **6** produce intensely-coloured solutions (blue to green) in sulphuric acid (96%) arising from the formation of the cations **7** which decompose very rapidly at room temperature. Thus, solutions of the carbinols **6** in glacial acetic acid were titrated with concentrated sulphuric acid (96%) until no further colouration due to the intermediate cations **7** was observed. In this way, good to excellent yields of benzofluorene-carboxylic esters **8** were obtained in all cases with the exception of the carbinols **6e** and **6g**. The hydroxy ester **6e** afforded only 20% yield of the desired fluorene **8e**. The major product (75%) of that reaction was a lactone to which we assigned the structure **10** based on the following spectroscopic data and combustion analysis.



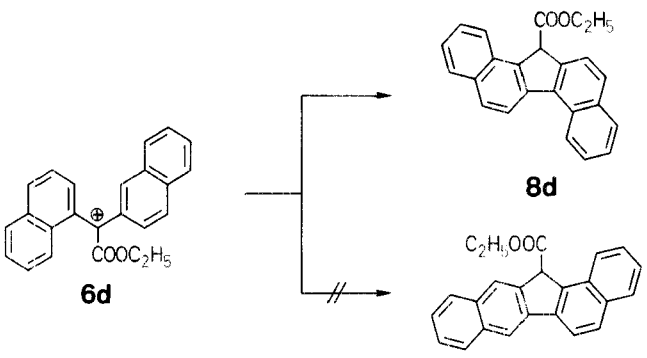
6e 11 12 10

Lactone **10** (white crystalline solid, m.p. 235–237 °C) exhibits a carbonyl absorption band at 1800 cm^{-1} consistent with a five-membered ring lactone. ^{1}H-N.M.R. (δ 7.28–8.80, m. 13 H, H_{arom}; δ 5.28, s, 1 H), mass spectra (m/e 310 M^+) and elemental analysis ($C_{22}H_{14}O_2$ requires 85.14% C, 4.55% H, found: 84.82% C, 4.59% H) were also in agreement with those expected from the compound **10**. The formation of the lactone **10** can be rationalized by 4π-electrocyclisation of ion **11** on oxygen, analogous to cyclisation of diphenylmethylcation (**1**; X = C_6H_5) reported recently[1,10] to give the enol ether **11** which, under the reaction condition, hydrolyses to the lactone **10**.

α-Hydroxy-α-(5-acenaphthyl)-phenyl acetic acid methyl ester (**6g**), when treated with concentrated sulphuric acid, resulted only in recovery of unidentifiable polymers. Although we have prepared and identified the intermediate carbenium ion **7g** under stable ion conditions[11], we believe that, at higher temperatures such as the ones used in the present study, ion **7g** undergoes a hydride transfer giving the more stable cation **13** which, in turn, may lose a proton to give the dehydro compound which is expected to polymerise under acidic conditions. Trans-annular 1,5-hydride transfers of a benzylic hydrogen to a highly reactive cationic center have been shown to occur in the case of other related carbocations[12].



In the case of α-hydroxy-α-(β-naphthyl)-α-naphthylacetic acid ethyl ester **(6d)** where there is a possibility for the intermediate carbocation to cyclise at the α- or β-position of the β-naphthyl substituent, we only isolated a single product arising from the cyclisation at the α-position.



We did not detect any trace of the other possible isomer in ^{1}H-N.M.R. spectrum of the crude reaction mixture. The identities of the benzofluorene-carboxylic esters **8a–f** were established by their spectral data, namely I.R. (—COOR[3] at 1720–1735 cm^{-1}), ^{1}H-N.M.R. (a singlet signal appearing in the range of 5.00 to 5.50 ppm due to > CH—COOR[3]), U.V., and mass spectra.

The final step of the synthesis was achieved by decarboxylation of the benzofluorene-carboxylic esters **8** in aqueous methanolic potassium hydroxide solutions. Good yields of the previously known hydrocarbons **9a–f** were obtained. The benzofluorenes **9a–f** were identified by spectral data, especially the appearance of a singlet signal (3.90–4.51 ppm) in the ^{1}H-N.M.R. spectra associated with the methylene hydrogens of the fluorene ring system and also the U.V. spectra which are very similar to those obtained for **8a–f**.

Melting points were determined on a Reichert melting point apparatus (hot plate method) and are uncorrected. The I.R. spectra were recorded on a Pye Unicam SP1000 instrument. A Hewlett

Table 1. Diarylhydroxyacetic Acid Esters **6a–g** prepared

Product	Yield[a] [%]	m.p. [°C] (C_2H_5OH)	R_f (Solvent)	Molecular Formula[b]	M.S. m/e (M^+)	I.R. (KBr)[c] ν [cm^{-1}]	^{1}H-N.M.R. ($CDCl_3$/TMS) δ [ppm]
6a	85	152–154°	–	$C_{19}H_{16}O_3$ (292.3)	292 (233; 100%)	3520, 1720	3.80 (s, 3H, OCH_3); 4.37 (s, 1H, OH, D_2O exchangeable); 6.96–8.20 (m, 12H, aromatics)
6b	90	126°	–	$C_{19}H_{16}O_3$ (292.3)	292 (233; 100%)	3520, 1720	3.87 (s, 3H, OCH_3); 5.58 (s, 1H, OH, D_2O exchangeable); 7.35–7.75 (m, 12H, aromatics)
6c	72	118–120°	0.64 (benzene)	$C_{24}H_{20}O_3$ (356.4)	356 (283; 100%)	3520, 1725	1.20 (t, 3H, J = 7 Hz, CH_3); 3.47 (q, 2H, J = 7 Hz, —OCH_2—); 4.73 (s, 1H, OH, D_2O exchangeable); 7.19–7.90 (m, 14H, aromatics)
6d	56	50–52°	0.52 (benzene)	$C_{24}H_{20}O_3$ (356.4)	356	3510, 1725	1.15 (t, 3H, J = 7.1 Hz, —CH_3); 4.29 (q, 2H, J = 7 Hz, —OCH_2—); 4.47 (s, 1H, OH, D_2O exchangeable); 7.17–8.17 (m, 14H, aromatics)
6e	53	138.5–139.5°	0.39 (benzene)	$C_{23}H_{18}O_3$ (342.4)	342 (283; 100%)	3500, 1730	3.86 (s, 3H, —OCH_3); 4.32 (s, 1H, OH, D_2O exchangeable); 7.32 (s, 1H, aromatic); 7.38–7.72 (m, 10H, aromatics); 8.14 (d, 1H, J = 8 Hz, aromatic); 8.63 (d, 1H, J = 8 Hz, aromatics); 8.72 (d, 1H, J = 8 Hz, aromatic)
6f	75	172–175°	0.57 (benzene)	$C_{28}H_{22}O_3$ (406.5)	405 (333; 100%)	3495, 1740	1.06 (t, 3H, J = 7.1 Hz, CH_3); 4.30 (q, 2H, J = 7.1 Hz, —OCH_2—); 4.40 (s, 1H, OH, D_2O exchangeable); 7.27–7.60 (m, 16H, aromatics)
6g	60	139–141°	0.59 (15% C_2H_5OAc) in Petrol	$C_{21}H_{18}O_3$ (318.4)	318 (259; 100%)	3520, 1725	3.34 (s, 4H, —CH_2—CH_2—); 3.80 (s, 3H, —OCH_3); 4.20 (s, 1H, OH, D_2O exchangeable); 7.05–7.56 (m, 10H, aromatics)

[a] Yield of pure product after recrystallization.
[b] Satisfactory microanalyses obtained: C ±0.34, H ±0.34.
[c] Absorption for OH and C=O are given respectively.

Table 2. Benzofluorene-carboxylic Esters **8a–f** prepared

Product	Yield[a] [%]	m.p. [°C] (C_2H_5OH)	R_f (solvent)	Molecular Formula[b] or Lit. m.p. [°C]	M.S. m/e (M^+)	I.R. (KBr) $\nu_{C=O}$ [cm^{-1}]	^{1}H-N.M.R. ($CDCl_3$/TMS) δ [ppm]	U.V. (C_2H_5OH) λ [nm]
8a	64	140°	0.55 (benzene)	$C_{19}H_{14}O_2$ (274.3)	274	1730	3.57 (s, 3H, OCH_3); 5.11 (s, 1H, CHCOOCH$_3$); 7.25–7.97 (m, 10H, aromatics)	214, 228, 246, 265, 295, 306, 315
8b	75	133–134°	0.49 (benzene)	133–133.5°[19]	274	1735	3.79 (s, 3H, OCH_3); 4.98 (s, 1H, CH—COOCH$_3$); 7.39 (dt, 1H, J = 1.0 Hz, 7.5 Hz, aromatic); 7.51–7.58 (m, 2H, aromatics); 7.66 (ddd, 1H, J = 1.4 Hz, 7.1 Hz, 8.5 Hz); 7.74 (d, 1H, J = 6.8 Hz, aromatic); 7.76 (d, 1H, J = 8.3 Hz, aromatic); 7.86 (d, 1H, J = 8.3 Hz, aromatic); 7.96 (d, 1H, J = 7.5 Hz, aromatic); 8.37 (d, 1H, J = 8.8 Hz, aromatic); 8.74 (d, 1H, J = 8.4 Hz, aromatic)	232, 314, 328, 334
8c	80	140–142°	0.76 (benzene)	$C_{24}H_{18}O_2$ (338.4)	338 (254; 100%)	1730	1.02 (t, 3H, J = 7 Hz, CH_3); 4.09 (q, 2H, J = 7 Hz, OCH_2); 5.51 (s, 1H, CHCOOC$_2$H$_5$); 7.46 (ddd, 2H, J = 1.1 Hz, 6.9 Hz, 8.1 Hz, aromatic); 7.56 (ddd, 2H, J = 1.1 Hz, 6.9 Hz, 8.1 Hz, aromatic); 7.92 (d, 2H, J = 8 Hz, aromatics); 7.95 (s, 4H, aromatics); 8.19 (d, 2H, J = 8 Hz, aromatics)	216, 268, 280, 290, 332
8d	97	153–155°	0.62 (benzene)	$C_{24}H_{18}O_2$ (338.4)	338	1720	1.16 (t, 3H, J = 7 Hz, CH_3); 4.18 (q, 2H, J = 7 Hz, OCH_2); 5.29 (s, 1H, CHCOOC$_2$H$_5$); 7.47–7.59 (m, 2H, aromatics); 7.68 (ddd, 1H, J = 1.4 Hz, 7.0 Hz, 8.5 Hz, aromatics); 7.83 (d, OH, J = 8.4 Hz, aromatic); 7.87 (d, 1H, J = 8.4 Hz, aromatic); 7.94–8.05 (m, 4H, aromatic); 8.56 (d, 1H, J = 10.6 Hz, aromatic); 8.85 (d, 1H, J = 8.3 Hz, aromatic)	216, 248, 256, 284, 294, 336, 352
8e	20	195–197°	0.67 (benzene)	$C_{23}H_{16}O_2$ (324.4)	324 (265; 100%)	1725	3.67 (s, 3H, OCH_3); 5.27 (s, 1H, CHCOOCH$_3$); 7.38 (t, 1H, J = 7.5 Hz, aromatic); 7.53 (t, 1H, J = 7.5 Hz, aromatic); 7.63–7.78 (m, 5H, aromatic); 7.93–7.96 (m, 1H, aromatic); 8.37 (d, 1H, J = 7.8 Hz, aromatic); 8.71–8.86 (m, 3H, aromatics)	208, 214, 246, 264, 332, 336
8f	68	241°	0.72 (benzene)	$C_{28}H_{20}O_2$ (388.5)	388	1725	0.78 (t, 3H, J = 7.2 Hz, CH_3); 3.88 (q, 2H, J = 7.2 Hz, OCH_2); 4.94 (s, 1H, CHCOOC$_2$H$_5$); 7.28–8.58 (m, 14H, aromatics)	206, 250, 258, 282
–[c]	–	–	–	–	–	–	–	–

[a] Yield of pure product after preparative thin-layer chromatography.
[b] Satisfactory microanalyses obtained: C ±0.34, H ±0.19.
[c] Intractable polymer formed (see Text).

Table 3. Benzofluorenes **9a–f** prepared

Product	Reaction Time	Yield[a] [%]	R_f (1/1 benzene/hexane	n.p. [°C] (solvent)	Lit. m.p. [°C]	M.S. *m/e* (M^+)	^{1}H-N.M.R. ($CDCl_3$/TMS) δ [ppm]	U.V. (C_2H_5OH) λ [nm]
9a	4 h	89	0.69	186° (C_2H_5OH)	189–190°[4]	266	4.08 (s, 2H, CH_2); 7.27–7.99 (m, 10H, aromatics) (cf. Ref. [20])	214, 230, 245, 254, 263, 295, 303, 315 (cf. Ref. [21])
9b	2 h	70	0.77	125–126° (C_2H_5OH)	124–125°[5]	266	4.00 (s, 2H, CH_2); 7.45 (dt, 1H, J = 7.0 Hz, 1.0 Hz, aromatic); 7.46–7.55 (m, 2H, aromatics); 7.61–7.70 (m, 3H, aromatic); 7.81 (d, 1H, J = 8.2 Hz, aromatics); 7.95 (d, 1H, J = 8.4 Hz, aromatic); 8.39 (d, 1H, J = 7.9 Hz, aromatic); 8.76 (d, 1H, J = 8.4 Hz, aromatic) (cf. Ref. [20])	216, 232, 312, 322, 328, 336 (cf. Ref. [22])
9c	4 d	75	0.67	234–236° (C_2H_5OAc)	234°[7]	266	4.46 (s, 2H, CH_2); 7.46 (ddd, 2H, J = 1.3 Hz, 6.9 Hz, 8.1 Hz, aromatic); 7.57 (ddd, 2H, J = 1.3 Hz, 6.9 Hz, 8.1 Hz, aromatics); 7.90 (d, 2H, J = 8.3 Hz, aromatic); 7.92 (d, 2H, J = 7.5 Hz, aromatic); 7.96 (d, 2H, J = 8.3 Hz, aromatic); 8.11 (d, 2H, J = 7.7 Hz, aromatics)	216, 266, 280, 288, 332
9d	20 h	89	0.72	176–178° (C_2H_5OH)	175–176°[6]	266	4.28 (s, 2H, CH_2); 7.45–7.58 (m, 3H, aromatics); 7.66 (ddd, 1H, J = 1.4 Hz, 6.9 Hz, 8.5 Hz, aromatic); 7.75 (d, 1H, J = 8.3 Hz, aromatic); 7.83 (d, 1H, J = 8.2 Hz, aromatic); 7.93 (d, 1H, J = 7.5 Hz, aromatic); 7.96 (d, 1H, J = 8.5 Hz, aromatic); 7.97 (d, 1H, J = 8.2 Hz, aromatic); 8.07 (d, 1H, J = 8.5 Hz, aromatic); 8.54 (d, 1H, J = 8.7 Hz, aromatic); 8.85 (d, 1H, J = 8.2 Hz, aromatic) (cf. Ref. [20])	214, 250, 256, 278, 286, 334, 350 (cf. Ref. [23])
9e	6 h	40	0.71	161–163.5° (*n*-C_3H_7OH)	159–159.5°[8]	266	3.90 (s, 2H, CH_2); 7.07 (t, 1H, J = 7.5 Hz, aromatic); 7.30 (t, 1H, J = 7.5 Hz, aromatic); 7.32–7.49 (m, 5H, aromatic); 7.80 (dd, 1H, J = 7.3 Hz, aromatic); 8.11 (d, 1H, J = 6 Hz, aromatic); 8.45 (dd, 1H, J = 7.3 Hz, aromatic); 8.52 (dd, 1H, J = 7.5 Hz, 0.5 Hz, aromatic); 8.58 (dd, 1H, J = 7.5 Hz, 0.5 Hz, aromatic)	206, 244, 264, 320, 334 (cf. Ref. [8])
9f	2 h	88	0.67	230–232° (C_2H_5OH)	228–229°[9]	316	4.51 (s, 2H, CH_2); 7.48 (t, 1H, J = 6.9 Hz, aromatic); 7.58 (t, 1H, J = 6.9 Hz, aromatic); 7.61–7.79 (m, 4H, aromatic); 7.93 (d, 1H, J = 6.3 Hz, aromatic); 7.95 (d, 1H, J = 7.8 Hz, aromatic); 8.13–8.22 (m, 2H, aromatic); 8.54 (d, 1H, J = 8.7 Hz, aromatic); 8.73 (dd, 1H, J = 2.2 Hz, 7.3 Hz, aromatic); 8.81 (d, 1H, J = 8.0 Hz, aromatic); 8.93 (dd, 1H, J = 1.3 Hz, 7.8 Hz, aromatic)	212, 248, 278, 328, 346

[a] Yield of pure product after preparative thin-layer chromatography.

Packard 8451A Diode Array spectrophotometer was used to obtain the U.V. spectra. The ^{1}H-N.M.R. spectra were recorded on a Varian CF-20 or a Brucker AM300 spectrometer. Mass spectra were recorded on a V.G. Mikromass 16F spectrometer. Silica gel (CAMAG DF-5) was employed for preparative thin layer chromatography. A 2-mm layer of absorbent on plates 20 × 20 cm was used and 50–100 mg of material was applied to each plate. Bands were detected by exposure to short-wavelength uv light (254 nm). Analytical T.L.C. was performed on Merck plastic sheets type $60F_{254}$ (silica gel).

Diarylhydroxyacetic Acid Esters 6; General Procedure:
Arylmagnesium bromide[13–16] or aryllithium[17] reagents **4** (10 mmol) are prepared in dry ether or tetrahydrofuran (50 ml) and added, under an atmosphere of dry nitrogen, to a cold (0 °C) solution of the ethyl or methyl α-oxoarylacetate[17] **5** (10 mmol) in the same solvent (50 ml). The resulting mixture is heated under reflux for 2 h and allowed to stir overnight. After addition of dilute (10% v/v), hydrochloric acid (50 ml) (saturated aqueous ammonium chloride was used with tetrahydrofuran as solvent), the organic layer is separated, combined with ether washings (2 × 50 ml) of the aqueous layer, washed with water (2 × 50 ml), dried with magnesium sulphate, and the solvent was removed under vacuum. Pure hydroxy esters are either obtained directly from crystallisation of the crude material from ethanol (**6a–c** and **g** or by flash chromatography[18] (silica gel grade 60, Merck 9385 mesh 60A) followed by recrystallisation from ethanol (**6d–f**) (Table 1).

Benzofluorene-carboxylic Esters 8: General Procedure:
A solution of the diarylhydroxyacetic acid ester **6** (1.0 mmol) in glacial acetic acid (25 ml), cooled in cold tap water (< 10 °C), is treated dropwise with concentrated sulphuric acid (96%) until no more colouration due to the intermediate carbenium ion is observed (~ 25 ml). The viscous mixture is poured onto crushed ice (~ 100 g), extracted with ethyl acetate (2 × 50 ml), the organic extracts are washed successivley with water (50 ml), aqueous sodium hydrogen carbonate (10%) until neutral, water (50 ml) again, and dried with magnesium sulphate. The solvent is removed under vacuum and the product is purified by preparatory thin-layer chromatography (silica gel/benzene) followed by recrystallisation from ethanol (Table 2).

Benzofluorenes 9: General Procedure:
Benzofluorene-carboxylic ester **8** (100 mg) is heated with an alkaline aqueous methanolic solution (20 ml methanol + 1 ml 40% potassium hydroxide in water) under an atmosphere of nitrogen until all the starting material is consumed as indicated by T.L.C. (silica gel/benzene) (2 h–4 days). The reaction mixture is made acidic with hydrochloric acid, extracted with chloroform (5 × 10 ml), the organic extracts are combined and washed with water (2 × 50 ml) and dried with magnesium sulphate. The solvent was removed under vacuum and the product was purified by preparatory thin-layer chromatography (silica gel/1:1 benzene: hexane) followed by recrystallisation from an appropriate solvent (Table 3).

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