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Studies on the O/C-Arylation of Cyclic β-Diketones with Activated Aryl Fluorides

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Abstract. The reaction of nitrosubstituted aryl fluorides **1** with cyclic β-diketones **2** proceeds at 20 – 80 °C in the presence of bases, such as KOH, KF or NaOEt, leading to the aryl ethers **3a – m**. Depending on the base the reaction of dimedone **2a** or 1,3-cyclohexanedione **2b** with 2,4-dinitrofluorobenzene **1a** or 4-fluoro-3-nitrobenzonitrile **1b** furnishes C- and/or O-arylated products. Upon heating of **3g, e** and **3m** at 40 – 100 °C in DMF/K_2CO_3, the C-arylated ketones **4a – c** are formed in good yields. Starting from **3a** we obtained the chromenedione **5** under the same conditions.

Introduction

The enolates of 1,3-dicarbonyl compounds react as ambident anions leading to C- and/or O-functionalized products[1]. Thus, the acylation of dimedone furnishes O-acylated products. Upon heating in the presence of sodium acetate, C-acylation has been observed [2]. The reaction of dimedone with phenyl isothiocyanate leads exclusively to thiocarbamoylation at the carbon atom [3]. A C-cyclopropanation in 90 % yield succeeds when dimedone is treated with dimorpholinobicyclo[n.1.0.]alkanes [4].

The phenylation of the dimedone anion with diphenyliodonium salts, however, affords C-phenyl, C,C-diphenyl, O,C-diphenyl, and traces of O-phenyl derivatives [5]. In contrast to this, dimedone and aryllead triacetate react in chloroform containing pyridine to give 2,2-bisaryl-5,5-dimethylcyclohexane-1,3-dione in good yields. 2-Methylcyclohexane-1,3-dione and 2-methylcyclopentane-1,3-dione react under similar conditions leading to the mono-C-arylated compounds [6]. The reaction of dimedone with-4-nitrophthalodinitrile in DMF/K_2CO_3 furnishes 2-(2-nitro-4,5-dicyanophenyl)-5,5-dimethylcyclohexane-1,3-dione [7]. A well-known method leading to C-arylated products involves photostimulated or electrochemically induced $S_{RN}1$ reactions between aryl halides and β-dicarbonyl compounds (cf. e. g. [8,9]).

Within the framework of our studies on the chemistry of activated benzene derivatives (cf. [10] and lit. cited therein) we could show recently that nitrosubstituted aryl fluorides **1** are easily prepared from chloronitrobenzenes and KF in DMSO/polyethylene glycol 5090 [11]. In the present paper we report on our studies on arylations of the cyclic β-diketones **2** with **1**.

Results and Discussion

Nitrosubstituted aryl fluorides **1** react with cyclic 1,3-diketones, such as dimedone **2a**, 1,3-cyclohexanedione **2b**, 1,3-cyclopentanedione **2c**, 2-methyl-1,3-cyclohexanedione **2d** , or 2-methyl-1,3-cyclopentanedione **2e** leading to the aryl ethers **3** and, in some cases, also to the corresponding C-arylated compounds **4** as by-products[2]. The reaction was carried out under the following reaction conditions:

Method A: in H_2O/CH_2Cl_2 or in $H_2O/ClCH_2CH_2Cl$ in the presence of potassium hydroxide at 38, 70 or 80 °C using tetrabutylammonium bromide as phase transfer catalyst.

Method B: in dimethyl sulfoxide in the presence of potassium fluoride at 20 to 80 °C.

Method C: in dimethyl sulfoxide in the presence of potassium carbonate at 20 to 80 °C.

Method D: in ethanol/sodium ethoxide at 70 to 75 °C.

Results of the preparation of the aryl ethers **3a – m** are compiled in Table 1. The best yields have been obtained using method A or B. With exception of **3b** (method A), the yields of purified aryl ethers are in the

[1] The reactions of 1,3-cyclopentanediones are reviewed in ref. [1]

[2] The corresponding nitrosubstituted aryl chlorides undergo no reaction with **2** under these conditions.

Table 1 Preparation of Products **3a – m**

Product	Method	Temp. (°C)	Time (h)	Yield (%)[a]
3a	A	38	7.5	58
	B	20	2.5	68
	D	70	5.5	64
3b	A[b]	80	8	0
	B	80	1.5	70
	C	80	1.5	58
	D	70	6.5	0
3c	A	38	8	78
	B	20	3	58
	D	70	6	68
3d	D	70	4	70
3e	A	38	16	80
	D	75	3	59
3f	A	38	8	57
3g	A	38	7.5	52
3h	A	38	8	54
	B	20	2.5	81
3i	B	80	1.5	56
3k	A[b]	80	7.5	43
3l	A	38	8	56[c]
3m	A[b]	70	7	54
	D	70	6.5	54

[a] Referred to isolated and purified products
[b] $H_2O/Cl(CH_2)_2Cl$
[c] Compound **4a** is formed as by-product (11 %)

range of 43 to 81 %. In some cases (**3a**, **3c – e**, **3m**) also method D leads to good yields (54 to 70 %) of aryl ethers. With the exception of **3b**, the reaction in DMSO in the presence of potassium carbonate, however, furnishes only small amounts of aryl ethers.

The formation of the corresponding C-arylated compounds **4** as by-products has been observed in the reaction of **1a** with **2a** leading to **3l** (56 %) and **4a** (11 %). Our results in Table 2 show that in these cases the ratio of O-/C-arylated products is strongly dependent on the reaction conditions. As expected from the results of the O-arylation, C-arylation dominates when potassium carbonate in DMSO is used as a base.

Furthermore, we have shown that the C-arylated products **4a – c** can also be prepared by rearrangement of the corresponding aryl ethers upon heating in DMF in the presence of potassium carbonate at 40 – 100 °C (40 – 78 %). The rearrangement of **3l** with sodium fluoride under the same conditions, however, leads to **4a** in a yield of only 42 %. The C-arylated compound formed by the rearrangement of **3a** undergoes cyclization forming the 7, 8, 9, 10-tetrahydro-9,9-dimethyl-4-nitrobenzo[c]chromene-2,7-dione **5**.

The reaction pathway may be explained by a partial analogy with the Smiles rearrangement. This rearrangement comprises the conversion of an Ar-X-C-C-YH into an Ar-Y-C-C-XH fragment in the presence of a base. The reaction proceeds via a nucleophilic attack of the resulting -y^- function on the C-atom bearing the X function [12, 13]. We assume that the rearrangement of **3** to **4** also proceeds via an intramolecular nucleophilic substitution. The possibility of this rearrangement could also explain the mixture of products formed by the reaction of **1a, b** with **2a, b** (Table 2).

Table 2 Comparative Studies on the Reactions of **1a** + **2a** + **1b** + **2a**, and **1a** + **2b**

Products (Reactants)	Method	Temp. (°C)	Time (h)	Yields (%)[a] O-Aryl	C-Aryl
3l, 4a (1a + 2a)	B	20	5	48	21
	B	40	5	57	28
	B	70	5	49	29
	C	20	6	28	55
	C	70	5	–	86
	D	70	6.5	60	14
3m, 4b (1b + 2a)	B	60	3	63	21
	C	70	5	–	68
3g, 4c (1a + 2b)	B	40	5	53	26
	C	60	5	–	56

[a] Referred to isolated and purified products

Experimental

Melting points were measured on a Boëtius apparatus and are corrected. ^{1}H-NMR spectra were recorded on a Tesla BS 567 (100 MHz) instrument in $CDCl_3$ or DMSO-d_6 with HMDS as internal standard. ^{13}C-NMR spectra were obtained on a Varian GEMINI 300 spectrometer in $CDCl_3$ or DMSO-d_6/TMS (internal standard: HMDS). Mass spectra were obtained on an HP 5985 B (Hewlett Packard) at 70 eV. IR spectra were taken on a UR 20 spectrometer (Carl Zeiss, Jena). All β-dicarbonyl compounds used are commercially available. The aryl fluorides were prepared according to [11]. The preparation of compounds **3** and **4** is compiled in Tables 1 and 2. Analytical and spectroscopic data of compounds **3**, **4** and **5** are listed in Table 3.

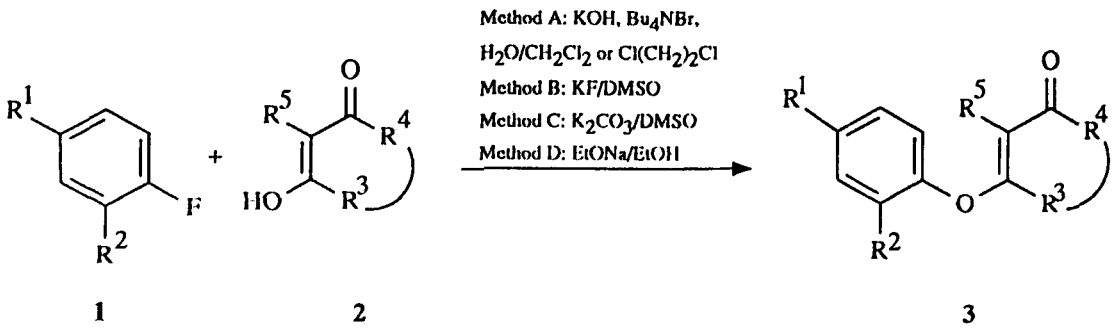
Aryl Ethers 3
(General Procedure)

Method A

1.12 g (0.02 mol) of potassium hydroxide are dissolved in 20 ml of water. Then 0.02 mol of **2** and 0.6 g (ca. 0.002 mol) of tetrabutylammonium bromide are added. A solution of 0.02 mol of **1** in 30 ml of dichloromethane or dichloroethane is added and the mixture is heated for 7 – 16 h under vigorous stirring in a water bath (cf. Table 1). After cooling the phases are separated, the aqueous is washed twice with 10 ml of the appropriate organic solvent, and the combined organic extracts are washed twice with 10 ml of water each. The dried (Na_2SO_4) and filtered organic solution is evaporated to dryness under reduced pressure. A solid residue is obtained.

Methods B, C

0.02 mol of **1** and 0.02 mol of **2** are dissolved in 16 ml of anhydrous dimethyl sulfoxide and then 3.48 g (0.06 mol) of



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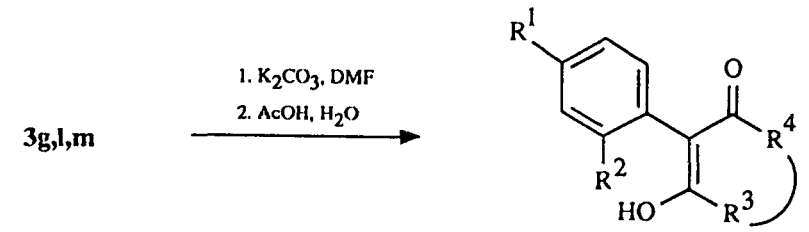


3g,l,m

4a-c

1	R^1	R^2	2	R^3	R^4	R^5
a	NO_2	NO_2	a	CH_2-$C(CH_3)_2$-CH_2-		H
b	CN	NO_2	b	-CH_2-CH_2-CH_2-		H
c	NO_2	CN	c	-CH_2-CH_2-		H
d	NO_2	Cl	d	-CH_2-CH_2-CH_2-		CH_3
			e	-CH_2-CH_2-		CH_3

3	R^1	R^2	R^3 R^4	R^5	4	R^1	R^2	R^3 R^4
a	NO_2	CN	-CH_2-$C(CH_3)_2$-CH_2-	H	a	NO_2	NO_2	-CH_2-$C(CH_3)_2$-CH-
b	NO_2	Cl	-CH_2-$C(CH_3)_2$-CH_2-	H	b	CN	NO_2	-CH_2-$C(CH_3)_2$-CH_2-
c	NO_2	CN	-CH_2-CH_2-	CH_3	c	NO_2	NO_2	-CH_2-CH_2-CH_2-
d	CN	NO_2	-CH_2-CH_2-	CH_3				
e	NO_2	NO_2	-CH_2-CH_2-	CH_3				
f	NO_2	CN	-CH_2-CH_2-CH_2-	CH_3				
g	NO_2	NO_2	-CH_2-CH_2-CH_2-	H				
h	NO_2	CN	-CH_2-CH_2-CH_2-	H				
i	NO_2	Cl	-CH_2-CH_2-CH_2-	H				
k	NO_2	CN	-CH_2-CH_2	H				
l	NO_2	NO_2	-CH_2-$C(CH_3)_2$-CH_2-	H				
m	CN	NO_2	-CH_2-$C(CH_3)_2$-CH_2-	H				

Table 3 Aryl Ethers **3a – m** and C-arylated Compounds **4a – c, 5** Prepared

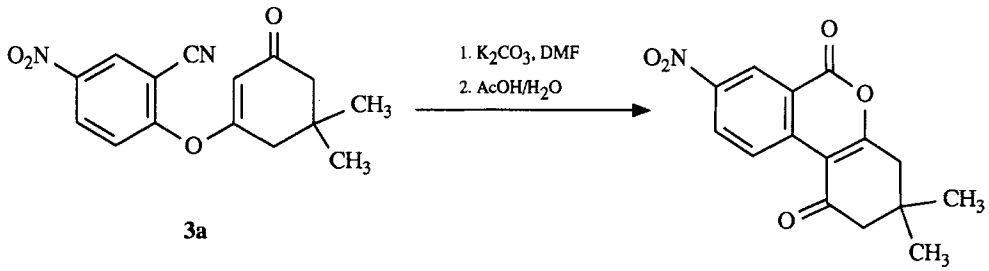
Product	m.p. (°C) (solvent)	Molecular Formula[a] (Mol. wt.)	MS m/e (%)	IR (KBr) ν (cm^{-1})	^{1}H-NMR[b] δ (ppm)	^{13}C-NMR[b] δ (ppm)
3a	125 – 127 (toluene/n-hexane 1:2)	$C_{15}H_{14}N_2O_4$ (286.28)	286 (M^+, 23), 67 (100)	2955, 2870, 2235, 1680, 1640, 1610	1.12 (s, 6H, $2CH_3$), 2.23 (s, 2H, CH_2), 2.57 (s, 2H, CH_2), 5.06 (s, 1H, CH), 7.35 – 8.53 ($3H_{arom.}$)	28.16, 32.83, 41.66, 50.53, 107.28, 108.12, 112.82, 123.25, 129.71, 144.94, 159.05, 173.89, 198.19
3b	114 – 116 (ethanol)	$C_{14}H_{14}ClNO_4$ (295.71)	295 (M^+, 13), 297 ($M^+ + 2$), 67 (100)	2960, 2870, 1660, 1625	1.10 (s, 6H, $2CH_3$), 2.20 (s, 2H, CH_2), 2.52 (s, 2H, CH_2), 4.92 (s, 1H, CH), 7.25 – 8.32 ($3H_{arom.}$)	28.26, 32.82, 41.76, 50.66, 105.80, 123.69, 123.92, 126.65, 128.29, 145.87, 153.83, 173.90, 198.40
3c	128 – 130 (ethanol)	$C_{13}H_{10}N_2O_4$ (258.23)	258 (M^+, 24), 83 (100)	2240, 1715, 1675, 1620	1.62 (s, 3H, CH_3), 2.63 (m, 4H, $2CH_2$), 7.25 – 8.60 ($3H_{arom.}$)	6.95, 25.98, 34.17, 105.94, 113.00, 119.44, 124.80, 129.71, 129.84, 143.85, 160.19, 176.81, 204.73
3d	176 – 178 (DMF/ ethanol 1:2)	$C_{13}H_{10}N_2O_4$ (258.23)	258 (M^+, 16), 147 (100)	2240, 1700, 1675, 1610	1.55 (s, 3H, CH_3), 2.50 (m, 4H, $2CH_2$), 7.31 – 8.28 ($3H_{arom.}$)	6.60, 25.82, 34.01, 110.15, 115.75, 122.59, 123.85, 130.02, 137.51, 150.57, 177.69, 204.48
3e	84 – 86 (ethanol)	$C_{12}H_{10}N_2O_6$ (278.22)	278 (M^+, 22), 167 (100)	3110, 3045, 1710, 1680, 1615	1.56 (s, 3H, CH_3), 2.56 (m, 4H, $2CH_2$), 7.47 – 8.84 ($3H_{arom.}$)	6.62, 25.86, 34.10, 122.02, 122.90, 123.38, 129.10, 141.25, 144.04, 151.88, 177.75, 204.56
3f	132 – 134 (ethanol)	$C_{14}H_{12}N_2O_4$ (272.26)	272 (M^+, 22), 53 (100)	2225, 1680, 1655, 1610	1.66 (s, 3H, CH_3), 2.07 (m, 2H, CH_2), 2.46 (m, 4H, $2CH_2$), 6.95 – 8.52 ($3H_{arom.}$)	8.50, 20.74, 27.57, 36.91, 104.40, 113.57, 116.20, 126.01, 130.18, 130.40, 142.97, 161.26, 164.93, 198.82
3g	122 – 124 (ethanol)	$C_{12}H_{10}N_2O_6$ (278.22)	278 (M^+, 4), 67 (100)	3100, 3045, 2935, 1665, 1610, 1600	2.08 (m, 2H, CH_2), 2.35 (m, 2H, CH_2), 2.70 (m, 2H, CH_2), 5.00 (s, 1H, CH), 7.44 – 8.90 ($3H_{arom.}$)	20.82, 27.96, 36.47, 107.46, 122.58, 126.34, 129.74, 141.68, 145.18, 150.77, 176.48, 198.74
3h	163 – 165 (DMF/ ethanol 1:2)	$C_{13}H_{10}N_2O_4$ (258.23)	258 (M^+, 34), 67 (100)	3095, 3075, 2225, 1680, 1655, 1610	2.10 (m, 2H, CH_2), 2.39 (m, 2H, CH_2), 2.73 (m, 2H, CH_2), 5.08 (s, 1H, CH), 7.39 – 8.54 ($3H_{arom.}$)	20.82, 27.98, 36.48, 108.12, 108.74, 112.78, 123.11, 129.64, 129.73, 145.00, 159.06, 175.25, 198.13
3i	104 – 106 (methanol)	$C_{12}H_{10}ClNO_4$ (267.66)	267 (M^+, 82), 269 ($M^+ + 2$), 67 (100)	3095, 1675, 1655, 1605	2.08 (m, 2H, CH_2), 2.34 (m, 2H, CH_2), 2.67 (m, 2H, CH_2), 4.92 (s, 1H, CH), 7.26 – 8.32 ($3H_{arom.}$)	20.99, 27.87, 36.56, 107.12, 123.94, 124.14, 126.82, 128.42, 146.01, 154.00, 175.80, 198.95
3k	142 – 144 (acetone/ n-hexane 1:2)	$C_{12}H_8N_2O_4$ (244.20)	244 (M^+, 44), 69 (100)	3095, 3060, 3025, 2230, 1705, 1685, 1615, 1605	2.58 (m, 2H, CH_2), 2.92 (m, 2H, CH_2), 5.17 (s, 1H, CH), 7.49 – 8.56 ($3H_{arom.}$)	28.09, 34.82, 107.66, 109.70, 112.57, 122.41, 129.77, 129.86, 145.22, 159.79, 186.26, 203.82
3l	133 – 135 (ethanol)	$C_{14}H_{14}N_2O_6$ (306.27)	306 (M^+, 16), 83 (100)	2960, 2925, 2870, 1660, 1630, 1610	1.13 (s, 6H, $2CH_3$), 2.23 (s, 2H, CH_2), 2.56 (s, 2H, CH_2), 5.03 (s, 1H, CH), 7.43 – 8.90 ($3H_{arom.}$)	28.20, 32.87, 41.70, 50.55, 106.07, 122.34, 126.16, 129.38, 141.61, 145.00, 150.51, 174.62, 198.23

Table 3 (continued)

Product	m.p. (°C) (solvent)	Molecular Formula[a] (Mol. wt.)	MS m/e (%)	IR (KBr) ν (cm^{-1})	^{1}H-NMR[b] δ (ppm)	^{13}C-NMR[b] δ (ppm)
3m	157 – 158 (ethanol)	$C_{15}H_{14}N_2O_4$ (286.28)	286 (M^+, 2), 83 (100)	2965, 2870, 2235, 1660, 1645, 1610	1.12 (s, 6H, $2CH_3$), 2.21 (s, 2H, CH_2), 2.54 (s, 2H, CH_2), 4.98 (s, 1H, CH), 7.35 – 8.33 ($3H_{arom.}$)	28.20, 32.85, 41.76, 50.60, 106.03, 111.28, 115.74, 126.36, 130.43, 138.03, 142.11, 149.26, 174.62, 198.18
4a	240 – 250 (decomp.) (acetic acid)	$C_{14}H_{14}N_2O_6$ (306.27)	306 (M^+, 1), 83 (100)	2955, 2865, 2550, 1600	1.03 (s, 6H, $2CH_3$), 2.31 (s, 2H CH_2), 2.38 (s, 2H, CH_2), 7.61 – 8.56 ($3H_{arom.}$)	27.06, 28.56, 31.61, 46.35, 110.72, 119.22, 126.36, 135.48, 135.54, 145.95, 149.36, 184.20
4b	242 – 245 (decomp.) (acetic acid)	$C_{15}H_{14}N_2O_4$ (286.28)	240 (M^+-NO_2, 32), 83 (100)	2955, 2875, 2525, 2225, 1605	1.00 (s, 6H, $2CH_3$), 2.30 (s, 2H, CH_2), 2.37 (s, 2H, CH_2), 7.52 – 8.38 ($3H_{arom.}$), 11.30 (s, 1H, OH)	27.03, 28.49, 31.52, 46.30, 110.36, 110.87, 117.28, 127.66, 133.82, 135.14, 135.28, 149.72, 183.50
4c	240 – 247 (decomp.) (acetic acid)	$C_{12}H_{10}N_2O_6$ (278.22)	278 (M^+, 1), 232 (100)	3160, 3100, 2930, 2625, 1615, 1605	1.88 (m, 2H, CH_2), 2.45 (m, 4H, $2CH_2$), 7.64 – 8.58 ($3H_{arom.}$) 11.74 (s, 1H, OH)	19.98, 32.75, 112.01, 119.19, 126.40, 135.50, 135.84, 146.05, 149.43, 184.50
5	188 – 190 (acetic acid)	$C_{15}H_{13}NO_5$ (287.26)	287 (M^+, 91), 231 (100)	3100, 2950, 2865, 1730, 1675, 1605	1.04 (s, 6H, $2CH_3$), 2.47 (s, 2H, CH_2), 2.82 (s, 2H, CH_2), 7.51 – 8.08 ($3H_{arom.}$)	27.46, 31.55, 41.64, 51.92, 109.37, 120.69, 124.15, 127.01, 129.48, 138.94, 146.30, 159.04, 172.13, 196.88

[a] Satisfactory microanalyses obtained: C ± 0.25, H ± 0.05, N ± 0.09, Cl ± 0.14

[b] NMR spectra for **3a – m** are measured in $CDCl_3$, for **4a – c** and **5** in DMSO-d_6



3a **5**

dried, finely powdered potassium fluoride (Method B) or 8.28 g (0.06 mol) of dried, finely powdered potassium carbonate (Method C) are added. The thus prepared mixture is heated under stirring for several hours as indicated in Tables 1 and 2. After cooling to room temperature, 8 ml (Method B) or 20 ml (Method C) of acetic acid and 240 ml of water are added dropwise under stirring. Then the precipitate is isolated and dried over $CaCl_2$ under reduced pressure.

Method D

0.46 g (0.02 mol) of sodium are dissolved in 20 ml of absolute ethanol. Then 0.02 mol of **2** is added. After 5 min 0.02 mol of compound **1** is added, and the reaction mixture is then heated for several hours as indicated in Tables 1 and 2. After cooling to room temperature, the solid obtained is isolated by suction. Crude **3c–e** are precipitated from the reaction mixture by addition of water (200 ml).

Purification of Crude Products and Separation of Mixtures Obtained by Methods A – D

3a is precipitated from 50 ml of cold toluene by addition of hot n-hexane (100 ml). **3b** is dissolved in 70 ml of cold ethyl acetate, the residue is filtered off and the filtrate is evaporated to dryness under reduced pressure. The residue is recrystallized from 15 ml of ethanol. **3c, d, e** are recrystallized from 20 ml of ethanol, 50 ml of DMF/ethanol (1 : 2), and 12 ml of ethanol, respectively. **3f, g** are extracted with n-hexane using a Soxhlet apparatus, the n-hexane solution is evaporated to dryness, and the residue is recrystallized from 40 ml of ethanol. In this way **3g** is separated from the unsoluble **4c**. **3h** is recrystallized from 30 ml of DMF/ethanol (1 : 2), **3i** is extracted from unsoluble impurities as described for **3f, g** and recrystallized from 10 ml of methanol. **3k** is precipitated from 15 ml of hot acetone by addition of hot n-hexane (30 ml). **3l, m** are separated from **4a, 4b**, respectively, by dissolution in 100 ml of cold ethyl acetate, the undissolved **4a, b** are filtered off and the filtrate is evaporated to dryness under reduced pressure. **3l, m** are recrystallized from ethanol (15 ml). **4a, b, c** are recrystallized from acetic acid.

Aryl Ketones 4a – c

(General Procedure)

8.28 g (0.06 mol) of dried, finely powdered potassium carbonate are added to 0.02 mol of **3g, l, m** in 20 ml of anhydrous DMF. Then the reaction mixture is heated under stirring for several hours (**4a**: 70 °C, 5 h; **4b**: 100 °C, 1.5 h; **4c**: 40 °C, 5 h). After cooling to room temperature, 20 ml of acetic acid are added portionwise. Then 250 ml of water are added dropwise, the precipitate is isolated and recrystallized from acetic acid (**4a** from 170 ml, **4b** and **4c** from 40 ml). Yields: **4a** (78 %), **4b** (60 %), **4c** (40 %).

7, 8, 9, 10-Tetrahydro-9,9-dimethyl-4-nitrobenzo-[c]chromene-2,7-dione (**5**)

8.28 g (0.06 mol) of dried, finely powdered potassium carbonate are added to 5.72 g (0.02 mol) of **3a** in 20 ml of anhydrous DMF. The reaction mixture is heated under stirring for 5 h at 70 °C. Then 20 ml of acetic acid are added portionwise, the reaction mixture is diluted with 20 ml of water and heated under stirring for 1 h at 90 °C.

After cooling to room temperature, the reaction mixture is poured into 200 ml of water, the precipitate is isolated and recrystallized from 15 ml of acetic acid. Yield: 50 %.

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