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A SIMPLE DIRECT APPROACH TO 1-SUBSTITUTED 3-ARYLISOQUINOLINES FROM DEOXYBENZOINS AND NITRILES

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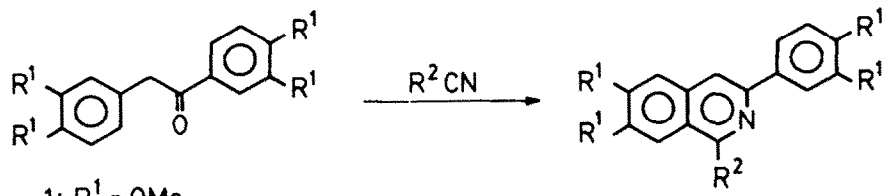
Abstract — A new one-pot synthesis of 3-arylisoquinolines was accomplished by reaction of deoxybenzoins with an excess of nitriles and phosphorus pentoxide at room temperature. The usefulness of this synthesis was demonstrated by the facile preparation of 1-alkyl, 1-alkenyl, and 1-aryl substituted 3-arylisoquinolines 3 and 4 on a preparative scale. The effect of nitrile type and deoxybenzoin substitution pattern on the feasibility of ring construction were studied. In most cases, naphtalene 6 and pyrimidines 7 or 8 were isolated as side products. When the reaction was similarly carried out with phosphorus oxychloride, instead of phosphorus pentoxide, the major product was the chlorostilbene derivative 5.

Nitriles are versatile reagents which have proved to be valuable precursors to a number of polysubstituted heterocycles.[1-4] Formation of imidoyl compounds at the first stage of the reaction of ketones with nitriles in the presence of both protic and Lewis acids has already been described[5,6] and their cyclization reactions have been discussed. It was found that the ketone-nitrile reaction, a Ritter-type reaction,[1] provided several heterocyclic compounds, such as pyrimidines, pyridines, isoquinolines, and 1,3-oxazines.[7] When amides were used instead of nitriles analogous results were observed.[8] The methods, however, are not free from shortcomings. Alkyl aryl ketones always gave mixtures of isoquinolines and pyrimidines in low to moderate yields; furthermore, the reaction has not been applied to aryl benzyl ketones, deoxybenzoins.

In our continuing studies on the synthetic utility of deoxybenzoins, which have produced several heterocycles via the Leuckart reaction,[9] we have developed a simple direct approach to the synthesis of 1-substituted 3-arylisoquinolines. It is the purpose of this paper to describe the reaction of deoxybenzoins 1 and 2 with a series of nitriles: acetonitrile, propionitrile, phenylacetonitrile, benzonitrile, and acrylonitrile, in the presence of $POCl_3$, PCl_5, and P_2O_5.

We began our study by examining the reaction of deoxybenzoin 1, prepared via the Friedel-Crafts reaction,[10] with acetonitrile in the presence of $POCl_3$ under reflux, following the procedure applied by Zieliński to other benzyl ketones.[7] It was found, however, that this reaction generated chlorostilbene 5 in a 80% yield. (see Experimental Section). The chlorostilbene genesis could be explained by assuming the formation of a chlorocarbocation intermediate by the action of $POCl_3$ on deoxybenzoin 1. Similar results have been previously observed in the Bischler-Napieralski cyclization of 1,2-diarylethylamides.[11,12]

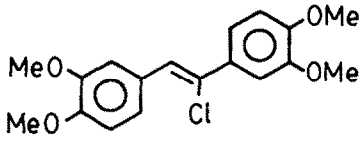


1: R^1 = OMe
2: R^1 = H

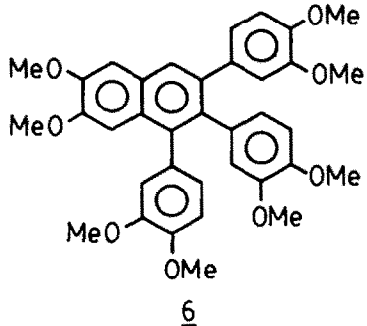
3: R^1 = OMe
4: R^1 = H

a: R^2=Me ; b: R^2=Et ; c: R^2=CH_2Ph ; d: R^2= Ph ; e: R^2=CH=CH_2

Therefore, a variety of reaction conditions were investigated in order to obtain the isoquinoline derivative. Thus, the use of acetonitrile as solvent[12] did not preclude stilbene formation, but did allow to obtain moderate yield of the desired 1-methyl substituted 3-arylisoquinoline 3a,[13] though always accompanied by naphtalene 6 and pyrimidine 7a as side products. The results are summarized in Table 1. The structure of naphtalene 6 was assigned on the basis of spectral data (see Experimental Section) and confirmed by comparison with an authentic sample, obtained by DDQ oxidation[14] of the corresponding tetraline derivative.[12]



5



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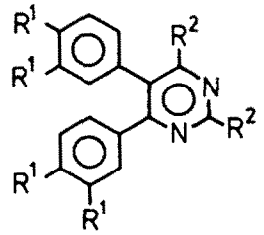

7: R^1 = OMe
8: R^1 = H

a: R^2=Me ; b: R^2=Et ; d: R^2=Ph

The pyrimidine 7a was characterized by spectral studies (see Table 2). The IR and UV spectra were similar to those of other diaryl substituted pyrimidines.[9] Thus, the IR spectrum showed bands at 1610, 1580, 1550, and 1520 cm^{-1} (C=C and C=N). The UV spectrum had three maxima at 229, 287, and 375 nm. The ^{1}H NMR spectrum exhibited, in addition to the resonances of the methoxy groups and aromatic protons, the characteristic singlets at 2.40 and 2.78 ppm, due to the methyl groups attached to C-6 and C-2, respectively.

Attempts to run reactions at lower temperatures in the hope of improving isoquinoline yield failed. In fact, at room temperature the starting material was recovered and in other cases (25°C to 60°C) only small conversions were observed. Nevertheless, it is noteworthy that in the reported examples the 3-arylisoquinoline yields were generally similar to the overall yields obtained via the classical Bischler-Napieralski[12] or Pictet-Spengler[15] cyclizations and subsequent dehydrogenation.[13] Besides, our method has the advantage of reducing the number of steps and is experimentally less complex. The mechanistic pathway to explain the formation of isoquinolines and pyrimidines in this kind of reactions has been previously demonstrated.[6,7] In our case, the presence of naphtalene 6 could be

Table 1. Reactions of Ketones 1 and 2 with Nitriles

Entry	Ketone	Conditions Nitrile	Method[a]	Isoquinoline	Yield(%) of Stilbene	Naphtalene	Pyrimidine
1[b]	1	MeCN	A	3a (24)	5 (60)	6 (5)	-
2[c]	1	MeCN	A	3a (23)	5 (57)	6 (7)	7a (4)
3[d]	1	MeCN	A	3a (20)	5 (57)	6 (8)	7a (10)
4[e]	1	MeCN	A	3a (20)	5 (57)	6 (8)	7a (10)
5[f]	1	MeCN	B	3a (75)	-	6 (7)	7a (5)
6[f]	1	EtCN	B	3b (70)	-	-	7b (4)
7[f]	1	$PhCH_2CN$	B	3c (45)	-	6 (15)	-
8[f]	1	PhCN	B	3d (35)	-	6 (24)	7d (7)
9[f]	1	$CH_2=CHCN$	B	3e (31)	-	-	-
10[d]	2	MeCN	A	4a (56)	-	-	8a (28)
11[d]	2	PhCN	A	4d (10)	-	-	8d (43)
12[f]	2	MeCN	B	4a (42)	-	-	8a (50)
13[f]	2	PhCN	B	4d (4)	-	-	8d (78)

[a] Method A: $POCl_3$, reflux; method B: P_2O_5, room temperature. [b] Ketone (1 mmol), nitrile (1 ml), $POCl_3$(1 mmol). [c] Ketone (1 mmol), nitrile (10 ml), $POCl_3$(1 mmol). [d] Ketone (1 mmol), nitrile (10 ml), $POCl_3$(4 mmol). [e] Ketone (1 mmol), nitrile (10 (10 ml), $POCl_3$(5 mmol). [f] Ketone (1 mmol), nitrile (10 ml), P_2O_5(4 mmol).

rationalized by assuming that a dimerization-type process has taken place between the imidoyl intermediate and the deoxybenzoin.

In order to improve the yields on isoquinoline, we decided to try PCl_5 and P_2O_5 as condensing agents, as they have proved to be useful in other cyclization reactions.[11,12] However, the use of PCl_5 always led to complex mixtures of unrecognizable products. On the other hand, it has been shown that treatment of a solution of deoxybenzoin 1, in the corresponding nitrile as solvent, with P_2O_5 at room temperature promoted the condensation to isoquinoline derivatives in high yields, minimizing the competitive side reactions. As it can be seen from Table 1, the reaction is sensitive to the nature of the nitrile.

To define the scope and limitations of our procedure to synthesize 3-arylisoquinolines, the method was applied to deoxybenzoin 2. Thus, the reaction of 2 with acetonitrile and benzonitrile in the presence of $POCl_3$ or P_2O_5 furnished the corresponding mixtures of isoquinolines 4 and pyrimidines 8 (Table 1). The structures of isoquinoline 4d and pyrimidine 8d could not be deduced from ^1H NMR data (Table 2), but they were unambiguously proved by the ^{13}C NMR spectra.[16] The longer reaction times required, the low isoquinoline yields, and the fact that no naphtalene derivative was isolated could be explained by the absence of alkoxylated substituent in the benzylic ring.

In conclusion, the method described in this paper, treatment of deoxybenzoins with nitriles and P_2O_5 at room temperature, allows the direct one-pot synthesis of 1-substituted 3-arylisoquinolines on a preparative scale and its advantages are that it does not required the tedious preparation of intermediate compounds[12,13,15] or the use of sophisticated reagents. It is noteworthy, however, that activating substituents in the benzylic ring are necessary for the cyclization to take place; otherwise tetrasubstituted pyrimidines are obtained as major products.

Table 2. Spectral Data for the new Isoquinoline and Pyrimidine Derivatives

Compound	IR, ν cm^{-1}	UV, λ nm (log ε)	^{1}H NMR, δ(ppm), J(Hz)[a]
3b	1625	239(4.42) 257(4.44) 274(4.46) 309(4.33)	1.52 (3H,t,J=7.5,CH_3CH_2), 3.27 (2H,q,J=7.5, CH_3CH_2), 3.90 (3H,s,MeO), 4.00 (9H,s, 3 x MeO), 6.95 (1H,d, J=8.1,H-5')[b], 7.15 (1H,s,H-5)[b], 7.30 (1H,s,H-8), 7.55-7.75 (2H,m,H-4 and H-6'), 7.82 (1H,d,J=1.8,H-2')
3c	1620	241(4.49) 272(4.51) 313(4.35)	3.85 (3H,s,MeO), 3.90 (3H,s,MeO), 3.95 (6H,s, 2 x MeO), 4.62 (2H,s,$PhCH_2$), 6.95 (1H,d,J=8.0, H-5')[b], 7.00 (1H,s,H-5)[b], 7.20-7.42 (6H,m,H-8 and Ph), 7.65-7.90 (3H,m,H-4,H-2', and H-6')
3e	1625	245(s)[c] 255(s)[c] 280(4.62) 310(s)[c]	3.90 (3H,s, MeO), 4.00 (9H,s,3 x MeO), 5.75(1H, dd,J_{AB}=2.4, J_{BX}=10.0, vinylic H_B), 6.75 (1H,dd, J_{AB}= 2.4, J_{AX}= 16.2, vinylic H_A), 6.95-7.10 (2H, m, H-5 and H-5'), 7.25-7.75 (2H,m,H-8 and CH_2=CH), 7.78-8.00 (3H,m,H-4,H-2' and H-6')
4a	1625	211(4.32) 252(4.51) 299(4.09)	3.00 (3H,s,MeC=N), 7.42-8.35 (10H,m,aromatic protons)
4d	1600	251(4.23) 282(3.99) 365(1.45)	7.30-7.78 (9H,m,aromatic protons), 8.51-8.80 (6H,m,aromatic protons)
7a	1610	229(4.42) 287(4.13) 375(3.25)	2.40 (3H,s,6-MeC=N), 2.78 (3H,s,2-MeC=N), 3.60 (3H,s,MeO), 3.70 (3H,s,MeO), 3.80 (3H,s,MeO), 3.85 (3H,s,MeO), 6.62-7.25 (6H,m,aromatic protons)
7b	1600	240(4.45) 287(4.21) 375(3.42)	1.45 (3H,t,J=8.1,6-CH_3CH_2)[b], 1.50 (3H,t,J=8.0, 2-CH_3CH_2)[b], 2.65 (2H,q,J=8.1,6-CH_3CH_2)[b], 3.05 (2H,q,J=8.0,2-CH_3CH_2)[b], 3.60 (3H,s,MeO), 3,70 (3H,s,MeO), 3.80 (3H,s,MeO), 3.85 (3H,s,MeO), 6.60-7.27 (6H,m,aromatic protons)
7d	1600	259(5.05) 287(5.10) 374(4.40)	3.50 (3H,s,MeO), 3.62 (3H,s,MeO), 3.80 (3H,s, MeO), 3.87 (3H,s,MeO), 6.50-7.23 (6H,m,H-2,H-5, and H-6 of 2 x Ar), 7.25-7.70 (8H,m,6-Ph and H-3,H-4, and H-5 of 2-Ph), 8.52-8.80 (2H,m,H-2 and H-6 of 2-Ph)
8a	1600	232(4.18) 274(3.98) 358(1.26)	2.40 (3H,s,6-MeC=N), 2.80 (3H,s,2-MeC=N), 7.05-7.52 (10H,m,2 x Ph)
8d	1600	256(4.30) 287(4.30 350(s)[c]	7.50-7.80 (12H,m,aromatic protons), 8.80-9.00 (8H,m,aromatic protons)

[a] s: singlet, d: doublet, dd: doublet of doublets, t: triplet, q: quartet, m: multiplet. [b] Partially overlapped signals. [c] s: shoulder

EXPERIMENTAL

Melting points were determined on either Electrothermal 1A 6304 or Büchi apparatus and are uncorrected. IR spectra were registered in KBr (solids) or $CHCl_3$ solution (oils) on a Perkin-Elmer 1430 spectrophotometer and only noteworthy absorptions (cm^{-1}) are reported. UV spectra were recorded in CH_2Cl_2 solution on a Beckman 5260 spectrophotometer. ^{1}H NMR and ^{13}C NMR spectra were taken on a Perkin-Elmer R-12B and a Varian XL-200 spectrometers, in $CDCl_3$ solution using TMS as internal standard. Mass spectrum was measured with a Hewlett-Packard 5930 B mass spectrometer. Microanalyses were carried out by the Colegio Universitario de Alava (Spain). TLC was performed on silica gel 60 plates (0.2 mm layer, GF-254, Merck). Visualization was accomplished by UV light or by spraying with Dragendorff's reagent.[17] Flash column chromatographic separations[18] were carried out on silica gel 60 (0.040-0.063 nm, 230-400 mesh, Merck).

Nitriles were refluxed over P_2O_5 and distilled inmediately before use. 3,4-dimethoxybenzyl 3,4-dimethoxyphenyl ketone 1 and benzyl phenyl ketone 2 were prepared as reported.[10,19] When a number of compounds were made by a more or less standard procedure, a typical method is given and the compounds are listed in tables (Tables 1, 2, and 3).

Table 3. Characterization of Isoquinolines 3 and 4 and Pyrimidines 7 and 8.

Compound	M.P.(°C) (EtOH)	R_f	Formula	Calcd. (Found) (%) C	H	N
3a	168-169[a]	0.19[b]	$C_{20}H_{21}NO_4$	70.78 (71.00)	6.24 (6.22)	4.13 (4.11)
3b	166-167	0.32[b]	$C_{21}H_{23}NO_4$	71.39 (71.57)	6.52 (6.54)	3.97 (3.96)
3c	169-170	0.54[b]	$C_{26}H_{25}NO_4$	75.18 (75.30)	6.02 (6.03)	3.37 (3.36)
3d	156-158[c]	0.52[b]	$C_{25}H_{23}NO_4$	74.80 (74.99)	5.76 (5.76)	3.49 (3.48)
3e	164-165	0.38[b]	$C_{21}H_{21}NO_4$	71.80 (71.64)	5.98 (5.96)	3.99 (3.98)
4a	oil	0.20[b]	$C_{16}H_{13}N$	87.67 (87.84)	5.94 (5.91)	6.39 (6.38)
4d	230-231	0.31[b]	$C_{21}H_{15}N$	89.68 (89.71)	5.34 (5.36)	4.98 (4.98)
7a	105-106	0.11[b]	$C_{22}H_{24}N_2O_4$	69.47 (69.50)	6.32 (6.35)	7.37 (7.35)
7b	oil	0.10[b]	$C_{24}H_{28}N_2O_4$	70.59 (70.41)	6.86 (6.84)	6.86 (6.87)
7d	172-173	0.51[d]	$C_{32}H_{28}N_2O_4$	76.19 (76.35)	5.56 (5.57)	5.56 (5.54)
8a	104-105	0.15[b]	$C_{18}H_{16}N_2$	83.08 (83.28)	6.15 (6.14)	10.77 (10.78)
8d	191-192	0.10[d]	$C_{28}H_{20}N_2$	87.50 (87.41)	5.21 (5.24)	7.29 (7.27)

[a] Lit.[13] M.P. 168-170°C (MeOH). [b] Eluent: dichloromethane/ethyl acetate (9.5:0.5).
[c] Lit.[13] M.P. 156-158°C (EtOH). [d] Eluent: hexane/dichloromethane (7:3).

Typical procedure for the reaction of deoxybenzoins with nitriles in the presence of $POCl_3$. Method A.

To a solution of the deoxybenzoin 1 (0.01 mol) in the appropiate nitrile as solvent (100 ml), $POCl_3$ (0.04 mol) was added dropwise at room temperature (Table 1). The reaction mixture was refluxed for 18-20 h. The reaction was monitored by TLC on silica gel using dichloromethane/ethyl acetate (9.5:0.5) as eluent and stopped when the starting material was consumed. At the end of the reaction, the solvent was removed under reduced pressure, water was added and the resulting suspension neutralized with 10% sodium hydroxide and extracted with dichloromethane. The dried extracts (anhydrous sodium sulphate) were concentrated and the residue separated by flash column chromatography into 3-arylisoquinoline 3, stilbene 5, naphtalene 6, and pyrimidine 7, which were recrystallized from ethanol (Table 3).

Chlorostilbene 5 had M.P. 105-107°C (EtOH), R_f= 0.8 (dichlomethane/ethyl acetate, 9.5:0.5). ^{1}H NMR δ ppm: 3.90 (6H, s, 2 x MeO), 3.92 (6H, s, 2 x MeO), 6.85-7.00 (3H, m, CH=CCl and aromatic protons), 7.25-7.50 (4H, m, aromatic protons). ^{13}C NMR δ ppm: 55.9 and 56.0 (MeO), 109.9, 110.7, 110.8, 112.2, 119.4, 122.8, and 124.5 (CH=CCl and aromatic CH), 128.3, 130.1, and 132.4 (CH=CCl, C-1', and C-1"), 148.5,, 148.6, 148.7, and 149.5 (aromatic C-O). (Found: C, 64.71; H, 5.64; Cl, 10.72. $C_{18}H_{19}ClO_4$ requires C, 64.58; H, 5.72; Cl, 10.59 %).

Naphtalene 6 had M.P. 208-210°C (EtOH), R_f= 0.3 (dichloromethane/ethyl 9.5:0.5), UV λ nm (log ε): 261 (4.92), 245 (4.86). ^{1}H NMR δ ppm: 3.50 (3H, s, MeO), 3.60 (3H, s, MeO), 3.65 (3H, s, MeO), 3.70 (6H, s, 2 x MeO), 3.85 (3H, s, MeO), 3.90 (3H,s, MeO), 4.05 (3H, s, MeO), 6.50-7.30 (14H, m, aromatic protons), 7.80 (1H, s, H-4). ^{13}C NMR δppm: 55.6, 55.65, 55.7, and 55.9 (MeO), 105.8, 106.2, 110.1, 110.5, 113.9, 114.9, 115.5, 121.1, and 124.0 (aromatic CH), 127.0, 127.8, and 128.7 (C-4a, C-8a, and/or C-4), 132.5, 133.3, and 135.5 (C-1', C-1", and/or C-1"), 136.1 (C-2), 137.6 (C-3), 138.1 (C-1), 146.9, 147.4, 147.5, and 147.8 (aromatic C-O). MS m/e(%): 596(100, M^+), 597(39), 598(10).(Found: C, 72.69; H, 6.11. $C_{36}H_{36}O_8$ requires C, 72.48; H, 6.04%).Besides, its structure was confirmed by comparison with authentic naphtalene obtained by oxidation of the corresponding tetraline (see below).

3-arylisoquinolines 3 and pyrimidines 7 were identified on the basis of spectral data (Table 2).

Typical procedure for the reaction of deoxybenzoins with nitriles in the presence of P_2O_5. Method B.

To a magnetically stirred solution of the deoxybenzoin 1 (0.01 mol) in the corresponding nitrile as solvent (100 ml), anhydrous P_2O_5 (0.04 mol) was added. The addition was carried out in portions under nitrogen atmosphere at room temperature. The progress of the reaction could be followed by TLC on silica gel (eluent: dichloromethane/ethyl acetate, 9.5:0.5). When the reaction was completed (16-18 h) and after work-up and separation as described above, the 3-arylisoquinolines 3, naphtalene 6, and pyrimidines 7 were isolated (Tables 1, 2, and 3).

Oxidation of 1,2,3-tris(3,4-dimethoxyphenyl)-6,7-dimethoxy-1,2,3,4-tetrahydronaphtalene with 2,3-dichloro-5,6-dicyanobenzoquinone (DDQ)

A solution of 1 g (5 mmol) of DDQ in 10 ml of toluene was added to 1 g (2 mmol) of the tetrahydronaphtalene[12] in 10 ml of toluene. The solution turned green and then to a yellow slurry over 1 h. After refluxing for 5 h, the solution was filtered to remove the dihydroxydicyanoquinone. Concentration and recrystallization from ethanol gave 0.8 g (yield 80%) of the corresponding naphtalene 6 as colorless crystals of M.P. 208-209°C.

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16. Selected ^{13}C NMR data (assignments made using off-resonance experiemnts):
    7d. δ ppm: 55.6, 55.7, and 55.8 (MeO), 110.4,111.0, 113.1, 114.3, 123.1, and 123.6 (aromatic CH of aryl groups), 127.8, 128.3, 128.4, 128.5, 129.2, 129.7, and 130.5 (aromatic CH of Ph groups), 128.2, 128.3, 131.3, 137.8, 139.0 (C-5, C-1', C-1", C-1" , and/or C-1'), 143.0, 148.2, 148.9, and 149.5 (aromatic C-O), 165.5, 164.5, and 162.5 (C-2, C-4, and/or C-6).
    8a. δ ppm: 21.4 (6-MeC=N), 24.0 (2-MeC=N), 125.4, 125.3, 126.4, 127.5, 127.6, and 128.0 (aromatic CH), 134.6, 134.7, and 136.3 (C-5, C-1', and/or C-1"), 161.6 (C-6), 163.7 and 164.0 (C-2 and/or C-4).
    8d. δ ppm: 127.7, 128.3, 128.4, 128.6, 129.9, and 131.1 (aromatic CH), 127.3, 130.6, 136.6, 137.8, and 138.8 (aromatic C and C-5), 162.8, 165.3, and 165.4 (C-2, C-4, and/or C-6).
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