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An efficient synthesis of some 3-substituted-isoquinolin(2H)-ones

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ABSTRACT

A number of 3-aryl-isoquinolin-1(2H)-ones were efficiently prepared from the corresponding 3-arylisocoumarins by refluxing with methanamide.

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KEYWORDS

Isocoumarin;
Isoquinolin-1(2H)-ones;
Methanamide.

INTRODUCTION

1(2H)-isoquinolones (isocarbostyrils) are the nitrogen analogues of isocoumarins (1H-2-benzopyran-1-ones). Various 1(2H)-isoquinolone derivatives are found in several bioactive natural products such as thalifoline, doryphorine^[1] ruprechstyryl^[2] narciclasine^[3], pancratistatin, lycoricidine^[4], the alkaloids coryaldine^[5], dorianine^[6] hydroxyhydrastinine and thalflavine^[7]. Isoquinolone nucleus is also an integral part of complex isoquinoline alkaloids and is a useful building block in organic synthesis.

The isoquinolone skeleton biogenetically derived from amino acid phenylalanine, exhibits biomimetic characteristics^[8]. Substituted isoquinolones are orally effective antagonists of receptors 5-HT₃, which have shown highly efficacy in the control of cancer models^[9], thymidylate synthase (TS) inhibitors^[10], human Tumor Necrosis Factor (TNF) inhibitors, and tachykinin receptors^[11]. Substituted isocarbostyrils exhibiting antidepressant^[12], anti-inflammatory^[13], analgesic^[14], hypolipidemic^[15] and analeptic^[16] activities have also been reported.

In view of the great therapeutic value of such mo-

tifs in various bioactive molecules, a number of synthetic routes have been developed. These include the Gabriel–Coleman synthesis^[17], ring enlargement of phthalimides^[18], condensation of amines with homophthalic anhydrides^[19], reaction of 2-methoxy carbonylstyrene oxide with ammonia or methyl amine^[20], reaction of coumarin and isocoumarin derivatives^[21] with ammonia and amines. The latter method has been used in the synthesis of (±)-licoricidine^[22], narciclasine^[23], (+)-deoxypancratistatin^[24], and the benzophenanthridine alkaloid nitidine^[25]. In addition, the [4+2]-cycloaddition of the ketenes to cyano ketones^[26], treatment of indanones with sodium azide^[27] and recently solid-phase synthesis of isoquinolinones using Bischler–Napieralski cyclization^[28], and palladium mediated synthesis of isoquinolinones^[29] syntheses via Curtius arrangement of cinnamic acids or via an isoquinolone N-oxide have been reported^[30].

The substitution of the oxygen by nitrogen atom is still one of the most important methods. A number of reagents have been used including ammonia, ammonium acetate or amines, but the results are never satisfactory^[31-33] except for a recently reported method^[34].

Majority of the natural isocoumarins being of

polyketide origin possess a C-3 alkyl/aryl substituent consequently, a variety of methodologies are available for rapid access to these bioactive heterocycles^[35]. Therefore the conversion of 3-substituted isocoumarins into corresponding isoquinolin-1(2*H*)-ones could prove a synthetically feasible procedure. As a continuation of our interest towards synthesis of naturally occurring isocoumarins^[36] and their synthetic analogues in this article, we wish to describe the conversion of a number of 3-substituted isocoumarins into their nitrogen analogues.

RESULTS AND DISCUSSION

The isocoumarins (**1a-j**) were synthesized according to method reported earlier^[36]. An equimolar mixture of the isocoumarin and methanamide was refluxed for 2-4 hours to afford corresponding 1(2*H*)-isoquinolones (**2a-j**). The products were obtained in 76-85 % yields in high purity (TABLE 1). The progress of the reaction was followed by TLC. The successful substitution was initially indicated by appearance of a fluorescent blue spot under longer wave length of UV lamp, having R_f values lower than that of the parent isocoumarin. The products were further characterized by comparison of their mp, IR, ¹H NMR and mass spectral data with those of the corresponding isocoumarins. Thus, a shift of lactonic carbonyl absorption from 1710-1730 cm^{-1} to 1630-1650 cm^{-1} and appearance of absorption at 3220-3380 cm^{-1} for NH was noted in the IR spectra. In the ¹H NMR a downfield shift of the characteristic H-4 proton of the isocoumarins at δ 6.0-6.2 to δ 6.6-6.9 in isoquinolones was observed besides, appearance of NH absorption at δ 9.4-10.8. A variety of substituents on the aryl ring are well-tolerated, and the reaction leads to completion in all the cases. The generality of the conversion was indicated by substrates bearing an aralkyl group (**2c**), heterocyclyl (**2i**) or a long aliphatic chain (**2j**) at C-3 position.

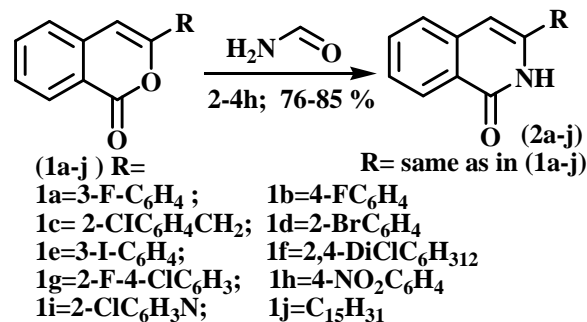
In conclusion, one pot, conversion of a number of 3-substituted isocoumarins to the corresponding isoquinolones has been achieved by refluxing with methanamide.

EXPERIMENTAL

TABLE 1: Physicochemical and spectral data of 1(2*H*)-isoquinolones (**2a-j**)

Entry	Comp.	R	Mp (°C)	Yield (%)	Reflux time (h)	¹ H NMR δ (ppm)		
						H-4(1)	H-4(2)	NH(2)
1	2a	3-FC ₆ H ₄	216-218	84	2.5	6.1	6.83	10.25
2	2b	4-FC ₆ H ₄	222-224	82	3.0	6.4	6.80	10.54
3	2c	2-ClCH ₂ C ₆ H ₄	188-190	85	3.5	6.24	6.66	9.53
4	2d	2-BrC ₆ H ₄	170-172	82	2.0	6.2	6.63	9.4
5	2e	3-IC ₆ H ₄	226-228	84	2.5	6.34	6.8	9.8
6	2f	2,4-DiClC ₆ H ₃	221-222	78	3.0	6.21	6.66	10.89
7	2g	2-Cl-4-FC ₆ H ₃	213	80	3.5	6.35	6.65	10.05
8	2h	4-NO ₂ C ₆ H ₄	230-232	84	4.0	6.26	6.94	8.79
9	2j	2-Cl-C ₆ H ₃ N	210-212	82	4.0	6.3	6.84	10.2
10	2k	C ₁₅ H ₃₁	71-73	76	4.0	6.24	6.89	6.62

Recrystallization solvent: Ethyl acetate



SCHEME 1 : Conversion of isocoumarins into 1(2*H*)-isoquinolones

Melting points were recorded using a digital Gallenkamp(SANYO) model MPD BM 3.5 apparatus and are uncorrected. ¹H NMR spectra were determined as CDCl₃ solutions at 300MHz on a Bruker AM-300 spectrophotometer. FT IR spectra were recorded using an FTS 3000 MX spectrophotometer; mass spectra(EI, 70eV) on a GC-MS instrument and elemental analyses with a LECO-183 CHNS analyzer. All compounds were purified by thick layer chromatography using silica gel from Merck.

General procedure for the conversion of isocoumarins into isoquinolin-1(2*H*)-ones (**2a-j**)

A mixture of isocoumarins (**1a-j**)(10mmol) and formamide (10mmol) was refluxed for 2-4h(TABLE 1). On completion of the reaction, followed by TLC the solution was poured into water (300ml). The resulting precipitates were filtered out and recrystallized

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from ethyl acetate to afford 1(2H)-isoquinolones (2a-j).

3-(3-Fluorophenyl)isocoumarin (1a)

$R_f=0.65$. IR(KBr):=1730, 2980, 1615 cm^{-1} . $^1\text{H NMR}$ δ 6.87(1H, s, H-4), 8.14(1H, s, H-2'), 7.56-7.70(2H, m, H-4', H-5'), 7.4(1H, d, $J=2.1$, H-6'), 7.3(2H, d, $J=7.8$, H-5, H-8), 7.22 (1H, dd, $J=1.8$, 2.1, H-6), 7.15(1H, dd, $J=2-4$, 2-4, H-7). EIMS(70eV): $m/z(\%)=240(43)$, 160(100%), 118(93).

3-(4-Fluorophenyl)isocoumarin (1b)

$R_f=0.55$, IR(KBr):=1725, 3020, 1590 cm^{-1} . $^1\text{H NMR}$ δ 6.95(1H, s, H-4), 8.70(2H, d, $J=7.8$ -H-3', H-5'), 7.77 (2H, d, $J=3$, H-2', H-6'), 7.72(1H, d, $J=1.2$, H-5), 7.51(3H, m, H-6, H-7, H-8). MS(70eV): $m/z(\%)=240(43)$, 160(100%), 118(93).

3-(2-Chlorobenzyl)isocoumarin (1c)

IR(KBr):=1738(C=O), 2860(C-H), 1558(C=C) cm^{-1} ; $^1\text{H NMR}$ δ :2.0(2H, s, CH_2), 6.52(1H, s, H-4), 7.48-7.51(2H, m, H-6, H-7), 7.58(1H, d, $J=1.6$, H-5), 7.65(1H, d, $J=1.5$, H-8), 7.81(1H, dd, $J=1.5$, $J=1.5$, H-5'), 7.85(1H, d, $J=1.5$, H-6'), 8.1(1H, dd, $J=1.5$, $J=1.8$, H-4'), 8.9(1H, d, $J=8.1$); MS(70eV): $m/z(\%)=272$, 270(43), 160(100%), 118(93).

3-(2-Bromophenyl)isocoumarin (1d)

IR(KBr):=1710, 3025, 1590 cm^{-1} ; $^1\text{H NMR}$ δ 6.75(1H, s, H-4), 7.81(1H, d, $J=2.4$, H-3'), 7.55-7.65 (3H, m, H-4', 5', 6'), 7.2-7.3(4H, m, H-5, 6, 7, 8) ppm; MS (70eV): $m/z(\%)$ 300(M^+ , 100), 226(40) 109(37), 145(60).

3-(3-Iodophenyl)isocoumarin (1e)

IR(KBr):=1725, 3010, 1580 cm^{-1} ; $^1\text{H NMR}$ δ 6.96(1H, s, H-4), 8.15(1H, s, H-2'), 7.96(1H, d, $J=9$, H-4'), 7.67(1H, d, $J=8.1$, H-6'), 7.61(1H, dd, $J=4.8$, 3.3, H-5'), 7.51-7.55(4H, m, H-5-H-8). MS(70eV): $m/z(\%)=347.9(100.0\%)$, 348.9(16.4%)

3-(2,4-Dichlorophenyl)isocoumarin (1f)

IR(KBr):=1705, 2970, 1620 cm^{-1} . $^1\text{H NMR}$ δ =6.95 (1H, s, H-4), 7.6(1H, d, $J=0.9$, H-3'), 7.5(1H, d, $J=13.2$, H-5'), 7.4(1H, d, $J=8.5$, H-6'), 7.1-7.35(4H, m, H-5-H-8). MS(70eV): $m/z(\%)=291.9(64.3\%)$, 290.9 (16.3%)

3-(2-Chloro-4-fluorophenyl)isocoumarin (1g)

IR(KBr):= 2990, 1705, 1595 cm^{-1} ; $^1\text{H NMR}$ δ 6.77 (1H, s, H-4), 7.80(1H, s, H-3'), 7.78(1H, d, $J=2.7$ H-5'), 7.19(1H, d, $J=2.4$, H-6'), 7.15(4H, m, H-5, 6, 7, 8). MS(70eV): $m/z(\%)=274.5(\text{M}^+, 100)$, 226(44) 109.5(37), 145(62). MS(70eV): $m/z(\%)=274.02$ (100.0%), 276.02(32.4%)

3(3-Nitrophenyl)isocoumarin (1h)

IR(KBr):=1734(C=O), 2893(C-H), 1512(C=C) cm^{-1} ; $^1\text{H NMR}$ δ : 7.1(1H, s, H-4), 7.2-7.4 4H, m, H-5, 6, 7, H-8), 7.7-7.5(2H, m, H-5', H-6'), 8.2(1H, d, $J=8.2$, H-4'), 8.4(1H, s, H-2')); MS(70eV): $m/z(\%)=267[\text{M}^+](97.7)$, 336(34.2), 314(63.7), 160(100%).

3-(2-Chloropyridyl)isocoumarin (1i)

IR(KBr):=1743(C=O), 2882(C-H), 1543(C=C) cm^{-1} ; $^1\text{H NMR}$ δ : 6.95(1H, s, H-4), 7.2-7.5(3H, m, H-5, H-6, H-7), 7.6(1H, d, $J=1.5$, H-8), 8.0(1H, dd, $J=2.2$, $J=2.4$, H-4'), 8.5(1H, d, $J=2.1$, H-5'), 8.7(1H, d, $J=1.6$, H-3'); MS(70eV): $m/z(\%)=259, 257[\text{M}^+](97.7)$, 336(34.2), 314(63.7), 160(100%), 118(97.7).

3-Pentadecylisocoumarin (1j)

IR(film): 2918, 2849, 1728, 1712, 1656, 1604, 1160, 642 cm^{-1} ; $^1\text{H NMR}$ δ 0.87(t, $J=6.28$ Hz, 3H, H-15'), 1.28(br s, 24H, H-3'-H-14'), 1.70(p, $J=8.4$ Hz, 2H, H-2'), 2.52(t, $J=7.08$ Hz, 2H, H-1'), 6.24(s, H-4), 7.34(d, $J=8.16$ Hz, H-5), 7.49(td, $J=0.88$, 7.28 Hz, H-7), 7.65(m, 1H, H-6) 8.25(d, $J=8.16$ Hz, H-8); MS(70eV): $m/z(\%)=356[\text{M}^+](97.7)$, 336(34.2), 314(63.7), 160(100%), 118(97.7).

3-(3-Fluorophenyl) isoquinolin-1(2H)-one (2a)

IR(KBr):=3332(NH), 1656(C=O), 2825(C-H), 1517(C=C) cm^{-1} ; $^1\text{H NMR}$: 6.83(1H, s, H-4), 7.1-7.2(3H, m, H-5, H-6, H-7), 7.23(1H, d, $J=2.4$, H-8), 7.54(1H, dd, $J=1.8$, $J=1.6$, H-5'), 7.70(1H, d, $J=1.5$, H-6'), 7.72(1H, d, $J=0.9$, H-2'), 8.4(1H, d, $J=1.2$, H-4'), 10.3(1H, s, NH); EIMS(70eV): $m/z(\%)=240$, 239 $[\text{M}^+](82.3)$, 144(53), 117(97.7).

3-(4-Fluorophenyl) isoquinolin-1(2H)-one (2b)

IR(KBr):=3320(NH), 1630(C=O), 2870(C-H), 1527(C=C) cm^{-1} ; $^1\text{H NMR}$ δ : 6.8(1H, s, H-4), 7.4(1H, dd, $J=1.2$, 1.1, H-5) 7.5-7.6(2H, m, H-6, H-7), 7.63

(1H, d, J=1.2, H-8), 7.7(2H, d, J=1.8, H-2', H-6'), 8.4(2H, d, J=8.4, H-3', H-5'), 10.54(1H, s, NH); EIMS(70eV):m/z(%)=240, 239[M⁺](61.3), 144(53), 117(98.1).

3-(2-Chlorobenzyl)isoquinolin-1(2H)-one (2c)

IR(KBr):=3348(NH), 1638(C=O), 2860(C-H), 1558(C=C) cm⁻¹; ¹HNMR δ: 2.19(2H, s, CH₂), 6.66(1H, s, H-4), 7.5-7.55(2H, m, H-6, H-7), 7.6(1H, d, J=1.5, H-5), 7.7(1H, d, J=1.5, H-8), 7.85(1H, dd, J=1.5, J=1.5, H-5'), 7.89(1H, d, J=1.5, H-6'), 8.3(1H, dd, J=1.5, 1.8, H-4'), 9.1(1H, d, J=8.1, H-3'), 9.53(1H, s, NH); EIMS(70eV):m/z(%)=271, 269[M⁺](43.7), 144(53.5), 117(59.2).

3-(2-Bromophenyl)isoquinolin-1(2H)-one (2d)

IR(KBr):=3341(NH), 1635(C=O), 2864(C-H), 1535(C=C) cm⁻¹; ¹HNMR δ: 6.635(1H, s, H-4), 7.34(1H, d, J=1.8, H-5), 7.45-7.55(2H, d, m, H-6, 7), 7.63(1H, d, J=2.7, H-8), 7.66-7.75(2H, m, H-4', H-5'), 8.38(1H, d, J=1.5, H-6'), 9.12(1H, d, J=8.4, H-3'), 9.4(1H, s, NH); EIMS(70eV):m/z(%)=301, 299[M⁺](71.3), 144(62), 117(87.4).

3-(3-Iodophenyl)isoquinolin-1(2H)-one (2e)

IR(KBr):=3353(NH), 1643(C=O), 2892(C-H), 1533(C=C) cm⁻¹; ¹HNMR δ: 6.8(1H, s, H-4), 7.5-7.54(2H, m, H-6, H-7), 7.5(1H, d, J=1.2, H-5), 7.6(1H, d, J=1.2, H-8), 7.74(1H, d, J=1.2, H-6'), 8.1(1H, t, J=1.8, H-5'), 8.2(1H, d, J=13.8, H-4'), 8.44(1H, s, H-2'), 10.6(1H, s, NH); EIMS(70eV):m/z(%)=348, 347[M⁺](16.5), 144(51), 117(59.6).

3-(2,4-Dichlorophenyl)isoquinolin-1(2H)-one (2f)

IR(KBr):=3330(NH), 1650(C=O), 2862(C-H), 1550(C=C) cm⁻¹; ¹HNMR δ: 6.63(1H, s, H-4), 7.5(1H, d, J=5.7, H-5), 7.61-7.67(2H, m, H-6, H-7), 7.71(1H, d, J=6.9, H-8), 7.88(1H, s, H-3'), 8.457(1H, d, J=7.8, H-6'), 9.06(1H, d, J=8.1, H-5'), 10.89(1H, s, NH); EIMS(70eV):m/z(%)=291, 289[M⁺](5.7), 144(53), 117(97.7).

3-(2-Chloro-4-Fluorophenyl)isoquinolin-1(2H)-one (2g)

IR(KBr):=3381 cm⁻¹(NH), 1655(C=O), 2876(C-H), 1550(C=C); ¹HNMR δ: 6.65(1H, s, H-4), 7.5-7.6(2H, m, H-6, H-7), 7.7(1H, d, J=1.2, H-5), 7.8(1H, d,

J=1.2, H-8), 7.9(1H, d, J=1.2, H-6'), 8.2(1H, s, H-3'), 8.4(1H, d, J=8.1, H-5'), 10.0(1H, s, NH), H-3'); EIMS(70eV):m/z(%)=275, 273[M⁺](72.8), 145(53), 117(84.4).

3-(3-Nitrophenyl)isoquinolin-1(2H)-one (2h)

IR(KBr):=3339(NH), 1634(C=O), 2821(C-H), 1512(C=C) cm⁻¹; ¹HNMR δ: 6.9(1H, s, H-4), 7.4-7.6(4H, m, H-5, 6, 7, 8), 7.7-7.2(2H, m, H-5', H-6'), 8.4(1H, d, J=8.4, H-4'), 8.6(1H, s, H-2'), 8.75(1H, s, NH); EIMS(70eV):m/z(%)=266[M⁺](82.3), 145(12), 117(43.0).

3-(2-Chloropyridyl)isoquinolin-1(2H)-one (2i)

IR(KBr):=3353(NH), 1663(C=O), 2882(C-H), 1543(C=C) cm⁻¹; ¹HNMR δ: 6.84(1H, s, H-4), 7.5-7.7(3H, m, H-5, H-6, H-7), 7.8(1H, d, J=1.5, H-8), 8.1(1H, dd, J=2.4, J=2.3, H-4'), 8.7(1H, d, J=2.2, H-5'), 8.8(1H, d, J=1.8, H-3'), 10.2(1H, s, NH); EIMS(70eV):m/z(%)=258, 256[M⁺](6.9), 145(32), 118(63.8).

3-Pentadecylisoquinolin-1(2H)-one (2j)

IR(KBr):=3323(NH), 1652(C=O), 2876(C-H), 1513(C=C) cm⁻¹; ¹HNMR δ: 0.9(3H, t, J=5.1, H-15'), 1.5-1.7(26H, m, H-2'-H-14'), 2.3(2H, t, J=7.5, H-1'), 6.89(1H, s, H-4), 7.6(1H, d, J=1.2, H-5), 7.7-7.74(2H, m, H-6, H-7), 8.2(1H, d, J=13.5, H-8), 6.62(1H, s, NH); EIMS(70eV):m/z(%)=355[M⁺](82.3), 145(43.6), 118(43.3).

Solvent system R_f values (Hexane: Ethyl acetate 4:1)

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