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DOWEX-50W PROMOTED FRIEDLÄNDER SYNTHESIS OFSUBSTITUTED QUINOLINES UNDER SOLVENT-FREE CONDITIONS

Huey-Min Wang,*[a] Rei-Sheu Hou,[a] Hau-Dung Du,[b] Iou-Jiun Kang,[c] and Ling-Ching Chen*[b]

[a] Chung Hwa University of Medical Technology, Tainan 717, Taiwan, R.O.C.

[b] Graduate Institute of Pharmaceutical Sciences, College of Pharmacy, Kaohsiung Medical University, Kaohsiung 807, Taiwan, R.O.C.
E-mail: m715003@cc.kmu.edu.tw

[c] Division of Biotechnology and Pharmaceutical Research, National Health Research Institutes, Miaoli County 350, Taiwan, R.O.C.

Abstract - An efficient method for the synthesis of substituted quinolines using Dowex-50W ion exchange resin as reusable eco-friendly catalyst vai Friedländer annulation under solvent-free conditions is described.

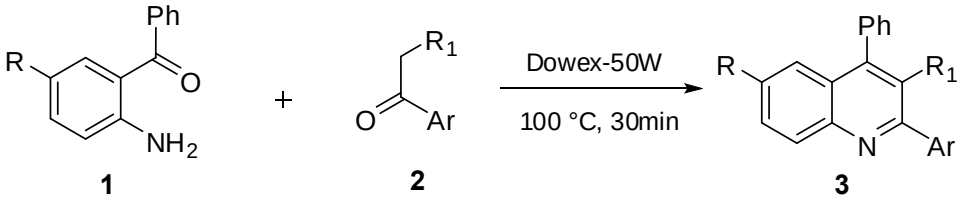
Quinoline derivatives have been well known not only in medicinal chemistry, because of their wide occurrence in natural products[1] and drugs,[2] but also in polymer chemistry, electronics and optoelectronics for their excellent mechanical properties.[3] Versatile methods for the synthesis of the quinoline ring system have been developed.[4] Friedländer annulation is one of the most simple and straightforward approaches for the synthesis of quinoline derivatives.

Homogeneous acidic catalysts such as H_2SO_4, HCl, $AlCl_3$, and BF_3, among others, are commonly used for organic synthesis carried out in laboratories and industries. However, the above-mentioned catalysts have several disadvantages because they are corrosive, toxic or volatile, and generate large amount of waste. In order to overcome all the drawbacks in the use of environmentally hazardous homogeneouse catalysts, new solid acids such as: zeolites, clays, alumina, heteropolyacids, and acidic resins have been tested.[5] Each catalyst has its own advantages and disadvantages. It is always interesting to develop a new environmental benign catalyst for organic transformation.

Recently, the use of ion exchange resins in organic synthesis has received great attention.[6] It is easy to measure, safe to use, and readily removed at the end of the reaction. We report here using Dowex-50 ion

exchange resin as catalyst for the first time to synthesize substituted quinolines via Friedländer annulation under solvent-free conditions.[7]

Treatment of 2-aminobenzophenone (**1**) and aryl ketones (**2**) with Dowex-50 as catalyst at 100 °C for 0.5 h caused cyclodehydration to give substituted quinolines (**3**) in good yields (Scheme 1). The results are given in Table 1. When the reaction is conducted in a conventional solvent, such as acetonitrile, the preparation of 2,4-dephenylquinoline (**3a**) needs refluxing for 10 h.



a, R=H

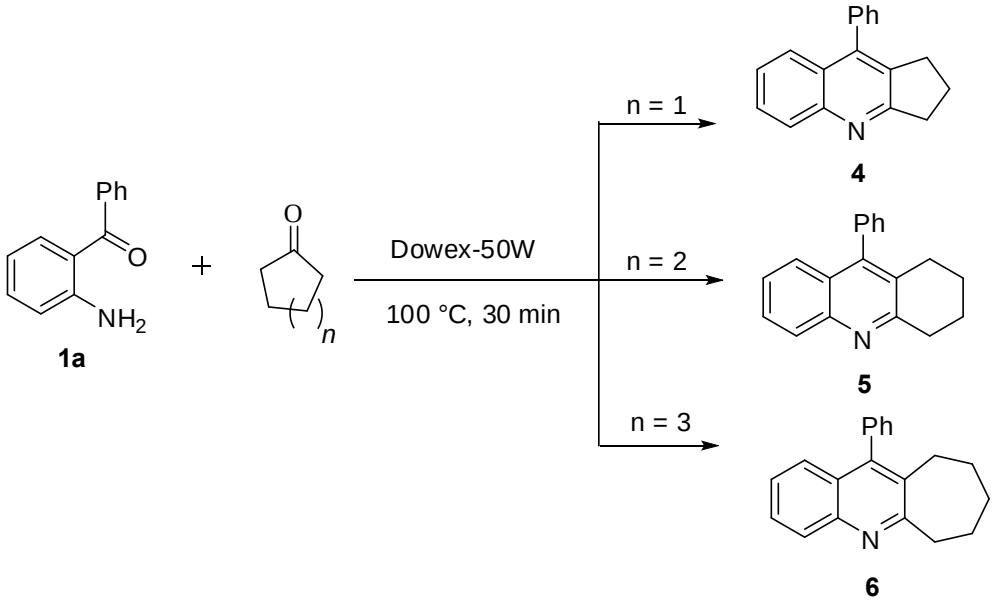
b, R=Cl

Scheme 1

Table 1. Synthesis of substituted quinolines (**3a-o**)

Entry	R	Ar	R_1	Product	Yield (%)
1	H	Ph	H	**3a**	80
2	H	4-MeC_6H_4	H	**3b**	75
3	H	4-$MeOC_6H_4$	H	**3c**	76
4	H	4-FC_6H_4	H	**3d**	74
5	H	4-ClC_6H_4	H	**3e**	82
6	H	4-BrC_6H_4	H	**3f**	84
7	H	2-Furyl	H	**3g**	73
8	H	2-Thienyl	H	**3h**	75
9	H	4-$NO_2C_6H_4$	H	**3i**	85
10	H	Ph	Me	**3j**	80
11	H	Ph	Ph	**3k**	78
12	Cl	4-BrC_6H_4	H	**3l**	84
13	Cl	4-ClC_6H_4	H	**3m**	85
14	Cl	4-MeC_6H_4	H	**3n**	83
15	Cl	4-$NO_2C_6H_4$	H	**3o**	90

This method is equally effective for cyclic ketone. Cyclopentanone, cyclohexanone and cycloheptanone were chosen to react with 2-aminobenzophenone (**1a**) to give the corresponding substituted quinolines (**4, 5** and **6**) in 81, 80 and 83% yields, respectively (Scheme 2).



Scheme 2

In conclusion, we have demonstrated a simple and efficient procedure for the Friedländer synthesis of substituted quinolines using Dowex-50W as catalyst under solvent-free conditions. The significant features of this method include operational simplicity, safe to use, and readily recovered at the end of the reaction.

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EXPERIMENTAL

All melting points are uncorrected. The IR spectra were recorded on a Shimadzu IR-27 G spectrophotometer. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Varian Unity Plus 400 MHz. Chemical shifts (δ) were measured in ppm with respect to TMS. MS were obtained on a JEOL JMS D-300 instrument.

Typical procedure for the synthesis of 2,4-diphenylquinoline (3a)

A mixture of acetophenone (**2a**) (240mg, 2.0mmol), 2-aminobenzophenone (**1a**) (197mg, 1.0mmol), and

Dowex-50W (100mg) was heated for 0.5 h at 100 °C. After cooling to room terperature, the reaction was dissolved in ethyl acetate and Dowex-50W was recovred by filtration. The filtrate was concentrated under reduced pressure and the residue was chromatographed on silica gel eluting with EtOAc-hexane (1:5) to give **3a**.

2,4-Diphenylquinoline (3a)

Mp 110-111 °C (Lit.,[8] mp 112-113 °C). IR (KBr) ν: 3050, 1584, 1541 cm^{-1}. ^1H NMR ($CDCl_3$) δ: 7.45-7.59 (m, 9H), 7.74 (ddd, *J* = 1.2, 6.8, 15.2 Hz, 1H), 7.83 (s, 1H), 7.92 (dd, *J* = 0.8, 8.4 Hz, 1H), 8.20 (d, *J* = 8.4 Hz, 2H), 8.26 (d, *J* = 8.4 Hz, 1H). ^{13}C NMR ($CDCl_3$,100 MHz) δ: 119.3, 125.6, 125.7, 126.3, 127.5, 128.4, 128.5, 128.8, 129.3, 129.4, 129.5 130.0, 138.3, 139.6, 148.7, 149.1, 156.8. MS (EI) *m/z*: 281 (M^+), 280, 202, 176, 139, 125.

2-(4-Methylphenyl)-4-phenylquinoline (3b)

Mp 95-96 °C (Lit.,[9] mp 116-117 °C). IR (KBr) ν: 3049, 1624, 1548 cm^{-1}. ^1H NMR ($CDCl_3$) δ: 2.43 (s, 3H), 7.34 (d, *J* = 8.0 Hz, 2H), 7.44-7.58 (m, 6H), 7.71-7.75 (m, 1H), 7.81 (s, 1H), 7.89 (dd, *J* = 1.0, 8.0 Hz, 1H), 8.10 (d, *J* = 8.0 Hz, 2H), 8.23 (d, *J* = 8.0 Hz, 1H). ^{13}C NMR ($CDCl_3$,100 MHz) δ: 21.3, 119.1, 125.5, 125.6, 126.0, 127.3, 128.3, 128.5, 129.3, 129.5, 129.9, 136.7, 138.4, 139.3, 148.7, 148.9, 156.7. MS (EI) *m/z*: 295 (M^+), 294, 202, 145, 139.

2-(4-Methoxyphenyl)-4-phenylquinoline (3c)

Mp 78-79 °C (Lit.,[8] mp 77-79 °C). IR (KBr) ν: 1736 cm^{-1}. ^1H NMR ($CDCl_3$) δ: 3.89 (s, 3H), 7.06 (ddd, *J* = 2.6, 2.6, 9.2 Hz, 2H), 7.44-7.58 (m, 6H), 7.69-7.75 (m, 1H), 7.79 (s, 1H), 7.89 (ddd, *J* = 0.8, 0.8, 8.4 Hz, 1H), 8.19 (ddd, *J* = 2.4, 2.4, 9.2 Hz, 2H), 8.24 (ddd, *J* = 0.6, 0.8, 8.4 Hz, 1H). ^{13}C NMR ($CDCl_3$,100 MHz) δ: 55.3, 114.1, 118.8, 125.4, 125.5, 125.9, 128.3, 128.5, 128.8, 129.4, 129.5, 129.8, 130.1, 132.0, 137.4, 138.4, 148.7, 149.0, 156.3, 160.8. MS (EI) *m/z*: 311 (M^+), 310, 268, 267, 140, 139, 132.

2-(4-Fluorophenyl)-4-phenylquinoline (3d)

Mp 63-64 °C. IR (KBr) ν: 3056, 1593, 1546 cm^{-1}. ^1H NMR ($CDCl_3$) δ: 7.19-7.24 (m, 2H), 7.47-7.57 (m, 6H), 7.72-7.76 (m, 1H), 7.78 (s, 1H), 7.91 (dd, *J* = 1.0, 8.4 Hz, 1H), 8.18-8.24 (m, 3H). ^{13}C NMR ($CDCl_3$,100 MHz) δ: 115.4, 115.7, 118.7, 125.5, 126.2, 128.3, 128.4, 129.2, 129.3, 129.4, 129.5, 129.8, 135.5, 135.6, 138.1, 148.6, 149.1, 155.5, 162.4. MS (EI) *m/z*: 299 (M^+), 298, 220, 202, 201, 139, 125. Anal. Calcd for $C_{12}H_{14}NF$: C, 84.26, H, 4.71, N, 4.68. Found: C, 84.39, H, 4.54, N, 4.53.

2-(4-Chlorophenyl)-4-phenylquinoline (3e)

Mp 104-105 °C (Lit.,[10] mp 106 °C). IR (KBr) ν: 3055, 1589, 1542 cm^{-1}. ^1H NMR ($CDCl_3$) δ: 7.47-7.57 (m, 8H), 7.74-7.77 (m, 1H), 7.79 (s, 1H), 7.91 (d, *J* = 8.0 Hz, 1H), 8.14-8.18 (m, 2H), 8.23 (d, *J* = 8.4 Hz, 1H). ^{13}C NMR ($CDCl_3$,100 MHz) δ: 118.6, 125.6, 125.7, 126.5, 128.4, 128.6, 128.8, 128.9, 129.5, 129.6, 130.0, 136.5, 137.9, 138.2, 148.7, 149.4, 155.4. MS (EI) *m/z*: 317 (M^++2), 315 (M^+), 314, 220, 202, 176, 139.

2-(4-Bromophenyl)-4-phenylquinoline (3f)

Mp 111-112 °C (Lit.,[11] mp 128-129 °C). IR (KBr) ν: 3029, 1584, 1539 cm^{-1}. ^1H NMR ($CDCl_3$) δ: 7.48-7.57 (m, 6H), 7.64-7.67 (m, 2H), 7.73-7.87 (m, 2H), 7.91 (dd, *J* = 1.0, 8.4 Hz, 1H), 8.08-8.11 (m, 2H), 8.24 (d, *J* = 8.4 Hz, 1H). ^{13}C NMR ($CDCl_3$,100 MHz) δ: 118.7, 123.8, 125.5, 125.7, 126.4, 128.4, 128.5, 129.0, 129.4, 129.6, 130.0, 131.8, 138.1, 138.3, 148.6, 149.3, 155.3. MS (EI) *m/z*: 361 (M^++2), 360 (M^++1), 359 (M^+), 279, 278, 202, 139.

2-(2-Furyl)-4-phenylquinoline (3g)

Mp 98-99 °C (Lit.,[9] mp 109-111 °C). IR (KBr) ν: 3060, 1594, 1546 cm^{-1}. ^1H NMR ($CDCl_3$) δ: 6.60 (dd, *J* = 1.6, 3.6 Hz, 1H), 7.25 (m, 1H), 7.43-7.57 (m, 6H), 7.63 (dd, *J* = 0.6, 1.8 Hz, 1H), 7.74 (ddd, *J* = 1.0, 6.8, 8.4 Hz, 1H), 7.78 (s, 1H), 7.87 (dd, *J* = 1.2, 8.4 Hz, 1H), 8.21 (d, *J* = 8.4 Hz, 1H). ^{13}C NMR ($CDCl_3$,100 MHz) δ: 110.1, 112.1, 117.6, 125.6, 125.7, 126.1, 128.3, 128.4, 129.4, 129.5, 129.8, 138.0, 144.0, 148.4, 148.5, 148.9, 153.6. MS (EI) *m/z*: 271 (M^+), 270, 243, 242, 241, 120.

4-Phenyl-2-(2-thienyl)quinoline (3h)

Mp 92-93 °C (Lit.,[9] mp 89-92 °C). IR (KBr) ν: 3054, 1585, 1543 cm^{-1}. ^1H NMR ($CDCl_3$) δ: 7.16 (dd, *J* = 3.8, 5.0 Hz, 1H), 7.42-7.57 (m, 7H), 7.68-7.74 (m, 3H), 7.84 (dd, *J* = 0.8, 8.4 Hz, 1H), 8.16 (d, *J* = 8.8 Hz, 1H). ^{13}C NMR ($CDCl_3$,100 MHz) δ: 117.8, 125.6, 125.7, 125.8, 126.0, 128.0, 128.4, 128.5, 128.6, 129.4, 129.5, 129.6, 138.0, 145.3, 148.5, 148.7, 151.7. MS (EI) *m/z*: 287 (M^+), 286, 253, 202, 200.

2-(4-Nitrophenyl)-4-phenylquinoline (3i)

Mp 156-157 °C (Lit.,[11] mp 162-163 °C). IR (KBr) ν: 3055, 1588, 1547 cm^{-1}. ^1H NMR ($CDCl_3$) δ: 7.52-7.58 (m, 6H), 7.77-7.81 (m, 1H), 7.86 (s, 1H), 7.95 (d, *J* = 8.0 Hz, 1H), 8.27 (d, *J* = 8.4 Hz, 1H), 8.37-8.42 (m, 4H). ^{13}C NMR ($CDCl_3$,100 MHz) δ: 119.0, 120.8, 123.3, 123.9, 125.7, 126.0, 127.2, 128.1,

128.2, 128.6, 129.3, 129.4, 130.0, 130.2, 130.9, 137.8, 145.3, 148.2, 148.7, 149.8, 153.9. MS (EI) *m/z*: 326 (M^+), 325, 280, 278, 202, 176, 139.

3-Methyl-2,4-diphenylquinoline (3j)

Mp 134-135 °C (Lit.,[12] mp 145-146 °C). IR (KBr) ν: 3048, 1609, 1568 cm^{-1}. ^1H NMR ($CDCl_3$) δ: 2.17 (s, 3H), 7.32-7.34 (m, 2H), 7.40-7.58 (m, 8H), 7.62-7.68 (m, 3H), 8.20 (d, *J* = 8.4 Hz, 1H).^{13}C NMR ($CDCl_3$,100 MHz) δ: 18.5, 115.8, 126.1, 126.5, 126.9, 127.7, 127.9, 128.0, 128.2, 128.4, 128.6, 128.8, 129.2, 129.3, 137.5, 141.3, 146.1, 147.6, 160.6. MS (EI) *m/z*: 295 (M^+), 294, 217, 189, 139.

2,3,4-Triphenylquinoline (3k)

Mp 190-191 °C (Lit.,[13] mp 189-190 °C). IR (KBr) ν: 3052, 1603, 1547 cm^{-1}. ^1H NMR ($CDCl_3$) δ: 6.88-6.91 (m, 2H), 6.99-7.02 (m, 3H), 7.14-7.16 (m, 2H), 7.21-7.23 (m, 3H), 7.26-7.31 (m, 3H), 7.38-7.40 (m, 2H), 7.46 (ddd, *J* = 1.2, 6.8, 8.4 Hz, 1H), 7.59 (dd, *J* = 1.2, 8.4 Hz, 1H), 7.74 (ddd, *J* = 1.2, 6.8, 8.4 Hz, 1H), 8.28 (d, *J* = 8.4 Hz, 1H). ^{13}C NMR ($CDCl_3$,100 MHz) δ: 126.2, 126.4, 126.5, 127.1, 127.2, 127.4, 127.5, 127.6, 129.2, 129.5, 129.8, 130.1, 131.2, 132.8, 136.8, 138.2, 141.0, 147.2, 147.5, 158.8. MS (EI) *m/z*: 357 (M^+), 356, 278, 176, 171.

6-Chloro-2-(4-bromophenyl)-4-phenylquinoline (3l)

Mp 173-175 °C (Lit.,[14] mp 174-176 °C). IR(KBr) ν: 3060, 1062, 1591 cm^{-1}. ^1H NMR ($CDCl_3$) δ: 7.54-7.58 (m, 5H), 7.64-7.70 (m, 3H), 7.80 (s, 1H), 7.86 (d, *J* = 2.2 Hz, 1H), 8.08 (d, *J* = 11.2 Hz, 2H), 8.15 (d, *J* = 12.4Hz, 1H). MS (EI) *m/z*: 397 (M^++4), 395 (M^++2), 393 (M^+), 358. Anal. Calcd for $C_{21}H_{13}BrClN$: C, 63.90, H, 3.32, N, 3.55. Found: C, 63.87, H, 3.14, N, 3.58.

6-Chloro-2-(4-chlorophenyl)-4-phenylquinoline (3m)

Mp 162-162 °C (Lit.,[14] mp 164-166 °C). IR(KBr) ν: 3056, 1066, 1585 cm^{-1}. ^1H NMR ($CDCl_3$) δ: 7.49-7.58 (m, 7H), 7.68 (dd, *J* = 3.2, 12.0 Hz, 1H), 7.80 (s, 1H), 7.86 (d, *J* = 2.8 Hz, 1H), 8.13 (d, *J* = 2.0 Hz, 2H), 8.16 (d, *J* = 3.2 Hz, 1H). MS (EI) m/z: 353 (M^++4), 351 (M^++2), 349 (M^+), 314. Anal. Calcd for $C_{21}H_{13}Cl_2N$: C, 72.02, H, 3.74, N, 4.00. Found: C, 72.06, H, 3.78, N, 4.23.

6-Chloro-2-(4-methylphenyl)-4-phenylquinoline (3n)

Mp 132-134 °C (Lit.,[14] mp 132-134 °C). IR(KBr) ν: 3046, 1602, 1581 cm^{-1}. ^1H NMR ($CDCl_3$) δ: 2.44 (s, 3H), 7.33 (d, *J* = 10.8 Hz, 2H), 7.52-7.57 (m, 5H), 7.66 (dd, *J* = 2.8, 12.0 Hz), 7.82 (s, 1H), 7.85 (d, *J* = 2.8 Hz, 1H), 8.09 (d, *J* = 11.2 Hz, 2H), 8.16 (d, *J* = 12.0 Hz, 1H). MS (EI) *m/z*: 331 (M^++2), 329 (M^+),

294. Anal. Calcd for $C_{22}H_{16}ClN$: C, 80.11, H, 4.89, N, 4.25. Found: C, 80.18, H, 4.77, N, 4.35.

6-Chloro-2-(4-nitrophenyl)-4-phenylquinoline (3o)

Mp 219-220 °C (Lit.,[14] mp 218-220 °C). IR(KBr) ν: 3040, 1611, 1524 cm^{-1}. ^1H NMR ($CDCl_3$) δ: 7.53-7.58 (s, 5H), 7.72 (dd, *J* = 3.2, 12.0 Hz, 1H), 7.88 (s, 1H), 7.90 (d, *J* = 3.2 Hz, 1H), 8.20 (d, *J* = 12.4 Hz, 1H). MS (EI) *m/z*: 362 (M^++2), 360 (M^+), 314, 278. Anal. Calcd for $C_{21}H_{13}ClN_2O_3$: C, 69.91, H, 3.63, N, 7.76. Found: C, 69.95, H, 3.72, N, 7.62.

2,3-Dihydro-9-phenyl-1*H*-cyclopenta[*b*]quinoline (4)

Mp 131-132 °C (Lit.,[15] mp 130 °C). IR (KBr) ν: 3058, 2923, 1569, 1485, 831 cm^{-1}. ^1H NMR ($CDCl_3$, 400 MHz) δ: 2.15-2.19 (m, 2H), 2.91 (t, *J* = 7.2 Hz, 2H), 3.24 (t, *J* = 7.6 Hz, 2H), 7.35-7.41 (m, 3H), 7.47-7.54 (m, 3H), 7.60-7.64 (m, 2H), 8.06-8.08 (m, 1H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 23.5, 30.3, 35.1, 125.4, 125.6, 126.2, 128.2, 128.4, 129.2, 133.6, 136.7, 142.6, 147.9, 167.4. EI-MS *m/z*: 245 (M^+), 244, 217, 168.

9-Pheyl-1,2,3,4-tetrahydroacridine (5)

Mp 137 °C (Lit.,[15] mp 138 °C). IR (KBr) ν: 3061, 2940, 2860, 1570, 1485, 1440, 1220, 765, 705 cm^{-1}. ^1H NMR ($CDCl_3$, 400 MHz) δ: 1.75-1.81 (m, 2H), 1.93-1.99 (m, 2H), 2.59 (t, *J* = 6.8 Hz, 2H), 3.18 (t, *J* = 6.8 Hz, 2H), 7.20-7.35 (m, 4H), 7.46-7.55 (m, 3H), 7.63-7.65 (m, 1H), 8.17 (d, *J* = 8.4 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 22.4, 22.7, 27.8, 33.3, 125.8, 125.8, 126.6, 127.1, 127.9, 128.6, 128.8, 129.0, 136.5, 144.6, 147.9, 158.5; EI-MS *m/z*: 259 (M^+), 244, 230, 217, 202, 189, 121.

11-Phenyl-7,8,9,10-tetrahydro-6*H*-cyclohepta[*b*]quinoline (6)

Mp 109-110 °C (Lit.,[16] 105-107 °C). IR (KBr) ν: 3054, 2927, 2851, 1571, 1485, 1443, 1196, 762, 708 cm^{-1}. ^1H NMR ($CDCl_3$, 400 MHz) δ: 1.61 (s, 2H), 1.86 (d, *J* = 2.4 Hz, 4H), 2.72 (t, *J* = 5.6 Hz, 2H), 3.38 (d, *J* = 3.6 Hz, 2H), 7.22-7.23 (m, 2H), 7.23-7.38 (m, 2H), 7.46-7.52 (m, 3H), 7.63 (t, *J* = 8.0 Hz, 1H), 8.22 (d, *J* = 8.4 Hz, 1H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 26.7, 28.1, 30.5, 31.6, 38.9, 126.0, 126.3, 126.8, 127.2, 128.4, 128.8, 129.1, 134.1, 136.9, 143.9, 146.9, 164.1. EI-MS *m/z*: 273 (M^+), 272, 258, 244, 231, 230.

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