Palladium-Catalyzed Amination of Aryl Iodides

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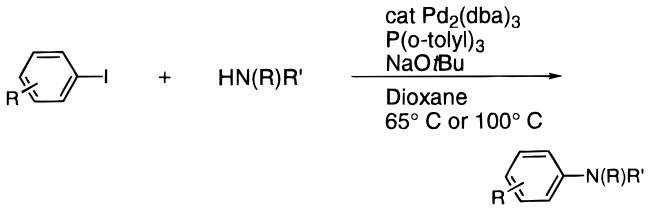
Aromatic amines play a central role in many areas of modern day organic chemistry, including such diverse areas as polymers,[1a] photography,[1b] and medicine.[1c] Although a large number of synthetic methods for the preparation of aniline derivatives have been reported,[2] general techniques which are applicable for the preparation of a wide variety of structurally different anilines are still of great interest. Over a decade ago, Migita and co-workers reported the first examples of the palladium-catalyzed transformation of aryl bromides to aryl amines via the use of aminostannanes.[3] We subsequently developed methodology in which the Migita process was generalized and greatly simplified.[4a] In addition, we recently reported the palladium-catalyzed conversion of aryl bromides and simple amines to the corresponding aniline derivatives.[4b,c,5] It was curious to us that in Migita's paper aryl iodides were reported not to be successfully transformed. Our original attempts to utilize aryl iodides as substrates were also largely unsuccessful. However, after some effort we have now developed a reasonably efficient protocol with which to convert aryl iodides to anilines, thus significantly expanding our amination process. Our results are described in this note.

After a great deal of experimentation, it was found that running the reaction in dioxane was key to the success of this technique. The reaction proceeds to completion in toluene, DMF, DME, and THF as well, albeit with substantially decreased yields.



As can be seen from the results presented in Table 1, the method works with both electron-rich and electron-deficient aryl iodides. In general, secondary amines are more efficiently handled. In the case of primary amines, including aniline, acceptable yields are only realized when there is a substituent ortho to the iodide. This contrasts with the results we have observed with aryl bromides, particularly for anilines. Additionally, while a para-substituted electron-deficient bromide provided the corresponding aniline in ∼70% yield,[4b] attempts to effect a similar transformation with an iodide were not efficient (entry 16).

We currently have no definitive explanation for the differences we observe in the reactions which employ aryl iodides and bromides. Ongoing mechanistic experiments in our laboratory indicate that the coordination chemistry involving the oxidative addition products of aryl iodides and bromides, with respect to their stoichiometry and reactivity, is substantially different.[7]

In summary, we have achieved for the first time the palladium-catalyzed cross coupling of amines with aryl iodides to provide the corresponding aniline derivatives. This process, which is of good generality and efficiency, significantly expands the range of substrates which can be handled using our palladium-catalyzed amination procedure.

Experimental Section

General. All reactions were carried out under an argon atmosphere in oven-dried glassware. Elemental analyses were performed by E&R Microanalytical Laboratory Inc., Corona, NY. Anhydrous dioxane was purchased form Aldrich Chemical Co. and was used without further purification. Dichloromethane was dried by distillation from CaH_2 under nitrogen. All amines were obtained from commercial sources and were purified either by distillation from CaH_2 or by passing through a short column of alumina. Aryl iodides (except substrates 11 and 16) were obtained from commercial sources and were used without further purification. Tris(dibenzylideneacetone)dipalladium(0) and sodium *tert*-butoxide were obtained from Aldrich Chemical Co. and used without further purification. Tri-*o*-tolylphosphine was purchased from Strem Chemical Co. and was used without further purification. Preparative flash chromatography was performed on ICN Biomedicals Silitech 32-63d Silica Gel. Yields refer to isolated yields (average of two runs) of compounds estimated to be ≥95% pure as determined by ^{1}H NMR and either capillary GC (known compounds) or combustion analysis (new compounds).

General Procedure for the Catalytic Amination of Aryl Iodides with Secondary Amines or Secondary Anilines. To a solution of aryl iodide (1 mmol), amine (2.4 mmol), and sodium *tert*-butoxide (2.8 mmol) in dioxane (4 mL) were added tris(dibenzylideneacetone)dipalladium(0) (0.005 mmol, 1 mol % Pd) and tri-*o*-tolylphosphine (0.02 mmol). The solution was heated to 100 °C with stirring until the aryl halide had been completely consumed as judged by GC analysis. The solution was then cooled to room temperature, taken up in ether (30 mL), and washed with brine (15 mL). The organic layer was dried over anhydrous magnesium sulfate, filtered, and concentrated. The crude product was then purified further by flash chromatography on silica gel. Fractions containing product were concentrated to give the pure compound.

***N*-Benzyl-*N*-methyl-*p*-methylaniline (1).**[9] The general procedure gave 164 mg (78%) of a pale yellow oil: ^{1}H NMR ($CDCl_3$, 300 MHz) δ 2.25 (s, 3H), 2.97 (s, 3H), 4.49 (s, 2H), 6.68 (d, 2H, J = 8.67 Hz), 7.03 (d, 2H, J = 8.73 Hz) 7.22−7.30 (m, 5H); ^{13}C NMR ($CDCl_3$, 300 MHz) δ 147.7, 139.2, 129.6, 128.4, 126.8, 126.7, 125.6, 112.6, 56.9, 38.5, 20.2; IR (neat, cm^{-1}) 3084, 3062, 3026, 2918, 1619, 1569, 1523, 1494, 1475, 1453, 1369, 1354, 1325, 1295, 1250, 1210, 1192, 1115, 1028, 947, 803, 733, 696.

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(4) (a) Guram, A.; Buchwald, S. L. *J. Am. Chem. Soc.* **1994**, *116*, 7901−7902. (b) Guram, A. S.; Rennels, R. A.; Buchwald, S. L. *Angew. Chem., Int. Ed. Engl.* **1995**, *34*, 1348−1350.

(5) Hartwig has reported related results: Louie, J.; Hartwig, J.F. *Tetrahedron Lett.* **1995**, *36*, 3609−3612.

(6) Typical reation times are 3−6 h for coupling secondary amines with electron-rich halides, 6−15 h for electron-poor halides, and 6−15 h for coupling reactions with primary amines.

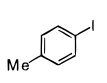
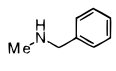
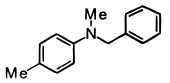
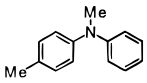
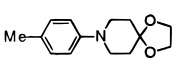
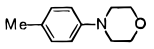
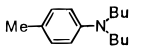
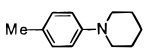
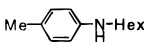
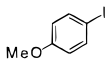
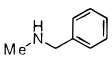
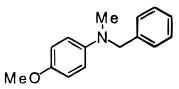
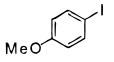
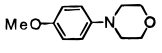
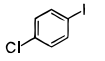
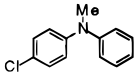
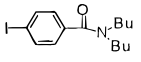
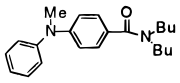
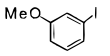
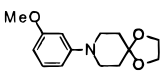
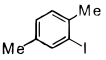
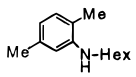
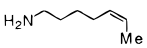
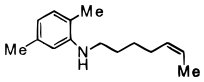
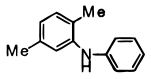
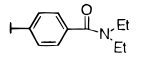
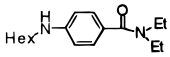
(7) Widenhoefer, R.; Zhong, H.; Buchwald, S. L. Unpublished results.

(8) Required 1.5% $Pd_2(dba)_3$ to achieve 100% conversion.

(9) Horner, L.; Meisel, K. *Werkst. Korros.* **1978**, *29*, 654−664.

0022-3263/96/1961-1133$12.00/0

Table 1. Conversion of Aryl Iodides to Aniline Derivatives

Entry	Halide	Amine	Product	Method[a]	Yield (%)
1				A	78
				B	79
2				A	74
3				A	66
4				A	73
5				A	68
6				A	59
				B	65
7		nHexNH$_2$		A	18
8				A	63
				B	63
9				A	65
10				A	61
11				A	59
12				A	59
13		nHexNH$_2$		A	69
14				A	68
15				A	64
16		nHexNH$_2$		A[b]	19

***N*-Benzyl-*N*-methyl-*p*-methoxyaniline (8).**[10] The general procedure gave 143 mg (63%) of a pale yellow oil: ^{1}H NMR ($CDCl_3$, 300 MHz) δ 2.91 (s, 3H), 3.74 (s, 3H), 4.42 (s, 2H), 6.72−6.83 (m, 4H), 7.23−7.33 (m, 5H); ^{13}C NMR ($CDCl_3$, 300 MHz) δ 151.7, 144.7, 139.2, 128.4, 127.0, 126.8, 114.6, 114.4, 57.9, 55.6, 38.9; IR (neat, cm^{-1}) 3061, 3027, 2992, 2933, 2903, 2830, 1514, 1494, 1453, 1355, 1295, 1244, 1213, 1181, 1117, 1040, 947, 814, 734, 697.

***N*-Methyl-*N*-phenyl-*p*-methylaniline (2).** The general procedure gave 146 mg (74%) of a pale yellow oil: ^{1}H NMR ($CDCl_3$, 300 MHz) δ 2.32 (s, 3H), 3.28 (s, 3H), 6.86−7.25 (m,9H); ^{13}C NMR ($CDCl_3$, 300 MHz) δ 149.3, 146.5, 131.9, 129.9, 129.0, 122.5, 119.7, 118.1, 40.2, 20.7; IR (neat, cm^{-1}) 3025, 2919, 2874, 1596, 1572, 1510, 1498, 1451, 1342, 1296, 1268, 1253, 1131, 992, 868, 820, 751, 696; GC−MS (*m*/*z*) 197, 196, 180, 167, 152, 118, 104, 91, 77, 51. Anal. Calcd for $C_{14}H_{15}N$: C, 85.24; H, 7.66. Found: C, 84.99; H, 7.63.

***N*-(*p*-Methylphenyl)-1,4-dioxa-8-azaspiro[4.5]decane (3).** The general procedure gave 154 mg (66%) of a white solid, mp = 64.8−65.6 °C: ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.85 (t, 4H, J = 5.7 Hz), 2.26 (s, 3H), 3.26 (t, 4H, J = 5.7 Hz), 3.99 (s, 4H), 6.86, (d, 2H, J = 8.7 Hz), 7.06 (d, 2H, J = 8.4 Hz); ^{13}C NMR ($CDCl_3$, 300 MHz) δ 148.9, 129.5, 128.9, 117.0, 64.2, 48.3, 34.5, 20.3; IR (KBr, cm^{-1}) 2961, 2885, 2840, 1616, 1518, 1464, 1368, 1333, 1232, 1209, 1142, 1098, 1036, 962, 945, 925, 894, 823; GC−MS (*m*/*z*) 233, 188, 172, 146, 119, 91, 65. Anal. Calcd for $C_{14}H_{19}NO_2$: C, 72.06; H, 8.21. Found: C, 72.26; H, 8.10.

4-(4-Methylphenyl) morpholine (4).[11] The general procedure gave 130 mg (73%) of a yellow solid, mp = 49.8−50.4 °C (lit. mp = 48 °C): ^{1}H NMR ($CDCl_3$, 300 MHz) δ 2.27 (s, 3H), 3.10 (m, 4H), 3.85 (m, 4H), 6.82, (d, 2H, J = 8.7 Hz), 7.08 (d, 2H, J = 8.7 Hz); ^{13}C NMR ($CDCl_3$, 300 MHz) δ 149.2, 129.7, 129.5, 116.0, 66.9, 49.9, 20.4; IR (KBr, cm^{-1}) 2957, 2853, 2830, 1517, 1452, 1380, 1298, 1260, 1235, 1118, 928, 819.

***N*,*N*-Dibutyl-4-methylbenzenamine (5).**[12] The general procedure gave 149 mg (68%) of a pale yellow oil. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 0.94 (t, 6H, J = 7.2 Hz), 1.33 (sx, 4H, J = 7.8 Hz), 1.53 (p, 4H, J = 7.5 Hz), 2.23 (s, 3H), 3.22 (t, 4H, J = 7.8 Hz), 6.56−6.60 (m, 2H), 7.01 (d, 2H, J = 8.1 Hz); ^{13}C NMR ($CDCl_3$, 300 MHz) δ 146.1, 129.6, 124.3, 112.2, 51.0, 29.4, 20.3, 20.1, 14.0; IR (neat, cm^{-1}) 2957, 2931, 2871, 1619, 1520, 1464, 1394, 1368, 1283, 1216, 1189, 800.

***N*-Methyl-*N*-phenyl-*p*-chloroaniline (10).** The general procedure gave 133 mg (61%) of a colorless oil: ^{1}H NMR ($CDCl_3$, 300 MHz) δ 3.28 (s, 3H), 6.88−7.32 (m, 9H); ^{13}C NMR ($CDCl_3$, 300 MHz) δ 148.6, 147.6, 129.3, 129.0, 125.5, 122.2, 121.4, 120.5, 40.3; IR (neat, cm^{-1}) 3061, 3037, 2943, 2880, 2815, 1601, 1588, 1568, 1494, 1452, 1342, 1304, 1253, 1184, 1156, 1133, 1098, 1085, 1066, 817, 744, 696; GC−MS (*m*/*z*) 219, 218, 217, 216, 201, 181, 167, 138, 125, 113, 104, 90, 77, 51. Anal. Calcd for $C_{13}H_{12}ClN$: C, 71.87; H, 5.57. Found: C, 71.95; H, 5.81.

1-(4-Methylphenyl)piperidine (6).[11] The general procedure gave 107 mg of a yellow oil which was Kugelrohr distilled to give 103 mg (59%) of a colorless oil: ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.50−1.73 (m, 6H), 2.26 (s, 3H), 3.09 (t, 4H, J = 5.4 Hz), 6.85 (d, 2H, J =8.4 Hz), 7.05 (d, 2H, J = 8.4 Hz); ^{13}C NMR ($CDCl_3$, 300 MHz) δ 150.3, 129.5, 128.7, 116.9, 51.3, 25.9, 24.3, 20.4; IR (neat, cm^{-1}) 2933, 2854, 2803, 1619, 1514, 1464, 1452, 1443, 1383, 1333, 1237, 1131, 1027, 920, 810.

4-(4-Methoxyphenyl)morpholine (9).[11] The general procedure gave 128 mg (66%) of a tan solid, mp = 73.3 °C (lit. mp = 71 °C). ^{1}H NMR ($CDCl_3$, 300 MHz) δ 3.06 (t, 4H, J = 4.5 Hz), 3.78 (s, 3H), 3.86 (t, 4H, J = 4.8 Hz), 6.84−6.91 (m, 4H); ^{13}C NMR ($CDCl_3$, 300 MHz) δ 153.9, 145.5, 117.7, 114.4, 66.9, 55.4, 50.7; IR (KBr, cm^{-1}) 2971, 2854, 2816, 1514, 1452, 1294, 1266, 1247, 1229, 1185, 1121, 1030, 928, 818.

***N*,*N*-Dibutyl-*p*-(*N*-methylaniline)benzamide (11).** The general procedure gave 198 mg (59%) of a colorless oil: ^{1}H NMR

(10) Kanner, C. B.; Pandit, U. K. *Tetrahedron* **1982**, *38*, 3597−3604.

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($CDCl_3$, 300 MHz) δ 0.8−1.6 (m, br, 14H), 3.33−3.5 (m, br, 4H), 6.87−7.4 (m, 9H); ^{13}C NMR ($CDCl_3$, 300 MHz) δ171.7, 149.5, 148.1, 129.3, 127.9, 127.7, 123.1, 116.6, 40.0, 30.1 (br), 19.1, 13.6; IR (neat, cm^{-1}) 3036, 2957, 2930, 2871, 1627, 1593, 1561, 1514, 1496, 1465, 1422, 1345, 1295, 1256, 1191, 1132, 1103, 830, 762, 700. Anal. Calcd for $C_{22}H_{30}N_2O$: C, 78.06; H, 8.93. Found: C, 77.87; H, 9.16.

***N*-(3-Methoxyphenyl)-1,4-dioxa-8-azaspiro[4.5]decane (12).**[4b] The general procedure gave 147 mg (59%) of a colorless oil: ^1H NMR ($CDCl_3$, 300 MHz) δ 1.83 (t, 4H, J = 6 Hz), 3.32 (t, 4H, J = 6 Hz), 3.79 (s, 3H), 3.99 (s, 4H), 6.37−6.58 (m, 3H), 7.15 (t, 1H, J = 8.1 Hz); ^{13}C NMR ($CDCl_3$, 300 MHz) δ 160.5, 152.1, 129.6, 109.2, 107.1, 104.0, 102.8, 64.2, 55.0, 47.5, 34.3; IR (neat, cm^{-1}) 2956, 2884, 2833, 1602, 1579, 1496, 1466, 1438, 1365, 1341, 1294, 1251, 1228, 1202, 1168, 1143, 1103, 1053, 965, 946, 912, 833, 761, 690.

General Procedure for the Catalytic Amination of Aryl Iodides with Primary Amines or Anilines. To a solution of aryl iodide (1 mmol), amine (1.1 mmol), and sodium *tert*-butoxide (2.8 mmol) in dioxane (9 mL) were added tris(dibenzylideneacetone)dipalladium (0) (0.005 mmol, 1 mol% Pd) and tri-*o*-tolylphosphine (0.02 mmol). The solution was heated to 100 °C with stirring until the aryl halide had been completely consumed as judged by GC analysis. The solution was then cooled to room temperature, taken up in ether (30 mL), and washed with brine (15 mL). The organic layer was dried over anhydrous magnesium sulfate, filtered, and concentrated. The crude product was then purified further by flash chromatography on silica gel. Fractions containing product were concentrated to give the pure compound.

***N*-(2,5-Xylyl)-hexylamine (13).** The general procedure gave 141 mg (69%) of a pale yellow oil: ^1H NMR ($CDCl_3$, 300 MHz) δ 0.91 (m, 3H), 1.30−1.45 (m, 6H), 1.66 (p, 2H, J = 7.8 Hz), 2.09 (s, 3H), 2.29 (s, 3H), 3.13 (t, 2H, J = 7.2 Hz), 3.38 (s, 1H, br), 6.43−6.47 (m, 2H), 6.92 (d, 1H, J = 7.5 Hz); ^{13}C NMR ($CDCl_3$, 300 MHz) δ 146.2, 136.6, 129.8, 118.6, 117.2, 110.5, 43.9, 31.6, 29.6, 26.9, 22.6, 21.5, 16.9, 14.0; IR (neat, cm^{-1}) 3428, 3014, 2956, 2926, 2856, 1615, 1584, 1523, 1466, 1426, 1376, 1312, 1298, 1266, 792; GC−MS (*m*/*z*) 205, 134. Anal. Calcd for $C_{14}H_{23}N$: C, 81.88; H, 11.30. Found: C, 82.07; H, 11.22.

(*Z*)-*N*-(5-Hexenyl)-2,5-xylidine (14). The general procedure gave 155 mg of a yellow oil which was Kugelrohr distilled to give 147 mg (68%) of a colorless oil: ^1H NMR ($CDCl_3$, 300 MHz) δ 1.49 (p, 2H, J = 7.2 Hz), 1.6−1.72 (m, 5H) 2.07−2.29 (m, 5H), 2.29 (s, 3H), 3.14 (t, 2H, J = 6.9 Hz), 3.38 (s, 1H, br), 5.35−5.51 (m, 2H), 6.42−6.47 (m, 2H), 6.92 (d, 1H, J =7.5 Hz); ^{13}C NMR ($CDCl_3$, 300 MHz) δ 146.2, 136.7, 130.2, 129.8, 124.2, 118.7, 117.2, 110.5, 43.8, 29.2, 27.1, 26.5, 21.5, 17.0, 12.8; IR (neat, cm^{-1}) 3426, 3012, 2927, 2856, 1615, 1583, 1523, 1444, 1298, 1267, 793, 701; GC−MS (*m*/*z*) 217, 160, 134, 105, 91, 77. Anal. Calcd for $C_{15}H_{23}N$: C, 82.88; H, 10.67. Found: C, 83.10, H, 10.45.

***N*-Phenyl-2,5-xylidine (15).**[13] The general procedure gave 137 mg of a yellow oil which was Kugelrohr distilled to give 128 mg (64%) of a colorless oil: ^1H NMR ($CDCl_3$, 300 MHz) δ 2.19 (s, 3H), 2.26 (s, 3H), 5.31 (s, 1H, br), 6.73−7.26 (m, 8H); ^{13}C NMR ($CDCl_3$, 300 MHz) δ 144.1, 140.9, 136.4, 130.7, 129.2, 125.3, 122.8, 120.2, 119.5, 117.3, 21.1, 17.4; IR (neat, cm^{-1}) 3386, 3048, 3018, 2920, 2858, 1600, 1578, 1518, 1497, 1464, 1412, 1377, 1311, 1000, 805, 747, 694.

***N*-(4-Methylphenyl)hexylamine (7).** The general procedure gave 75 mg (39%) of a yellow oil which was Kugelrohr distilled to give 35 mg (18%) of a white solid, mp = 37.1−37.3 °C: ^1H NMR ($CDCl_3$, 300 MHz) δ 0.89 (m, 3H), 1.26−1.64 (m, 8H), 3.07 (t, 2H, J = 7.5 Hz), 3.45 (s, br, 1H), 6.54 (d, 2H, J = 8.7 Hz), 6.97 (d, 2H, J = 8.89 Hz); ^{13}C NMR ($CDCl_3$, 300 MHz) δ 146.3, 129.7, 126.3, 112.9, 44.4, 31.6, 29.6, 26.8, 22.6, 20.3, 14.0; IR (KBr, cm^{-1}) 3413, 2927, 2858, 1618, 1522, 1458, 806; GC−MS (*m*/*z*) 191, 120. Anal. calcd for $C_{13}H_{21}N$: C, 81.61; H, 11.06. Found: C, 81.82; H, 10.83.

***N*,*N*-Diethyl-*p*-(hexylamino)benzamide (16).** To a solution of *N*,*N*-diethyl-*p*-iodobenzamide (303 mg, 1.0 mmol), *n*-hexylamine (0.15 mL, 1.1 mmol), and sodium *tert*-butoxide (270 mg, 2.78 mmol) in dioxane (9 mL) were added tris(dibenzylideneacetone)dipalladium(0) (5 mg, 0.005 mmol) and tri-*o*-tolylphosphine (6 mg, 0.02 mmol). The solution was heated to 100 °C with stirring. Additional portions of the palladium complex and phosphine were added after 16 h and after 22 h. Complete consumption of starting material occurred after 40 h (as judged by GC analysis). The solution was then cooled to room temperature, taken up in ether (30 mL), filtered, and concentrated. The crude product was then purified by flash chromatography on silica gel using 4/1 hexane/ethyl acetate as the eluant. Fractions containing product were concentrated to give 52 mg (19%) of a colorless oil: ^1H NMR ($CDCl_3$, 300 MHz) δ 0.90 (m, 3H), 1.18 (t, 6H, J = 6.9 Hz), 1.29−1.42 (m, 6H), 1.61 (p, 2H, J = 6.9 Hz), 3.11 (t, 2H, J = 7.2 Hz), 3.43 (q, 4H, J = 6.9 Hz), 3.87 (s, br, 1H), 6.53−6.56 (m, 2H), 7.23−2.27 (m, 2H); ^{13}C NMR ($CDCl_3$, 300 MHz) δ 172.8, 149.4, 128.4, 125.1, 111.6, 43.6, 31.5, 29.3, 26.7, 22.5, 14.0; IR (neat, cm^{-1}) 3333, 2958, 2930, 2858, 1614, 1531, 1470, 1456, 1425, 1380, 1335, 1314, 1285, 1176, 1098, 831, 764. Anal. calcd for $C_{17}H_{28}N_2O$: C, 73.87; H, 10.21. Found: C, 74.12; H, 10.26.

***N*,*N*-Dibutyl-*p*-iodobenzamide.** To a solution of di-*n*-butylamine (1.7 mL, 10.0 mmol) in dichloromethane (2 mL) in an oven-dried Schlenk tube at 0 °C was added slowly 4-iodobenzoyl chloride (1.066 g, 4.0 mmol) in dichloromethane (2 mL). The solution was stirred at 0 °C for 10 min and then warmed to rt and stirred for 5 h. The reaction mixture was then diluted with ether (30 mL), washed with saturated aqueous $NaHCO_3$ (10 mL), and washed with brine (10 mL). The organic layer was then dried over anhydrous $MgSO_4$, filtered, and concentrated to give 1.29 g (90%) of a colorless oil: ^1H NMR ($CDCl_3$, 300 MHz) δ 0.7−1.7 (m, 14 H), 3.1−3.6 (m, br, 4H), 7.1 (m, 2H), 7.75 (m, 2H); ^{13}C NMR ($CDCl_3$, 300 MHz) δ 170.3, 137.2, 136.5, 128.0, 94.8, 48.5, 44.3, 30.6, 29.3, 20.0, 19.5, 13.6, 13.4; IR (neat, cm^{-1}) 2957, 2930, 2871, 1632, 1586, 1558, 1487, 1465, 1425, 1379, 1296, 1265, 1234, 1182, 1101, 1007, 830, 754; GC−MS (*m*/*z*) 359, 358, 316, 231, 203, 104, 76. Anal. Calcd for $C_{15}H_{22}INO$: C, 50.15; H, 6.17. Found: C, 50.38; H, 6.07.

***N*,*N*-Diethyl-*p*-iodobenzamide.**[14] To a solution of diethylamine (1.05 mL, 10.0 mmol) in dichloromethane (2 mL) in an oven-dried flask at 0 °C was added slowly 4-iodobenzoyl chloride (1.066 g, 4.0 mmol) in dichloromethane (2 mL). The solution was stirred at 0 °C for 10 min and then warmed to rt and stirred for 15 h. The reaction mixture was then diluted with ether (30 mL), washed with saturated aqueous $NaHCO_3$ (10 mL), and washed with brine (10 mL). The organic layer was then dried over anhydrous $MgSO_4$, filtered, and concentrated to give 1.190 g (98%) of a colorless oil: ^1H NMR ($CDCl_3$, 300 MHz) δ 1.0−1.35 (m, br, 6H), 3.1−3.6 (m, br, 4H), 7.08−7.15 (m, 2H), 7.7−7.8 (m, 2H); ^{13}C NMR ($CDCl_3$, 250 MHz) δ 170.2, 137.5, 136.6, 128.0; IR (neat, cm^{-1}) 2972, 2933, 1632, 1586, 1488, 1470, 1458, 1427, 1383, 1363, 1347, 1315, 1288, 1095, 1008.

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