REMARKABLE FAST *N*-ALKYLATION OF AZAHETEROCYCLES UNDER MICROWAVE IRRADIATION IN DRY MEDIA

Dariusz Bogdal*, Jan Pielichowski, and Krzysztof Jaskot

Institute of Organic Chemistry, Politechnika Krakowska
ul. Warszawska 24, 31-155 Krakow, Poland

Abstract - Under microwave irradiation a number of azaheterocycles (i.e., pyrrole, imidazole, pyrazole, indole, and carbazole) react remarkably fast with alkyl halides to give exclusively *N*-alkyl derivatives.

In the last few years there has been an interest in the use of microwave heating in organic synthesis.[1] The use of such nonconventional reaction conditions reveals several features like: a short reaction time compared to conventional heating, ease of work-up after a reaction, and reduction in the usual thermal degradation and better selectivity.[2]

Reactions under "dry" conditions (i.e., in the absence of a solvent, on a solid support with or without catalysts) were originally developed in the late1980s.[3] Synthesis without solvents under microwave irradiation offers several advantages.[4] The absence of solvent reduces the risk of explosions when the reaction takes place in a closed vessel in an oven. Moreover, aprotic dipolar solvents with high boiling points are expensive and difficult to remove from the reaction mixtures. During microwave induction of reactions under dry conditions, the reactants adsorbed on the surface of alumina, silica gel, clay, and others absorb the microwaves whereas the support does not, nor does it restrict the transmission of microwaves. Consequently, such supported reagents efficiently induce reactions under safe and simple conditions with domestic microwave ovens instead of specialized expensive commercial microwave systems.

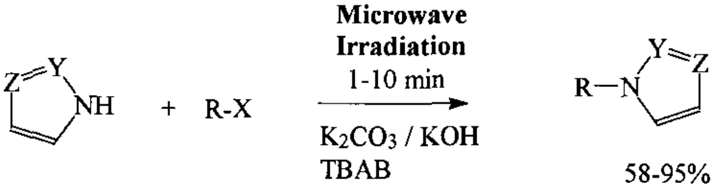
The *N*-alkylation of heterocyclic compounds bearing an acidic hydrogen atom attached to nitrogen is generally accomplished by treatment of these compounds with an appropriate base (sodium hydride or amide, or metaloorganic compounds) followed by treatment of the resulting salts with an alkylating agent.[5] An alternative is to perform the reaction of such heterocyclic compounds under phase transfer catalysis (PTC) conditions, in which halides react directly with heterocyclic compounds without converting them to salts in preliminary steps.[6]

Since a number of heterocycles (e.g., pyrrole and indole) exhibit ambident behavior as nucleophiles, alkylation can occur at carbon as well as at nitrogen,[7] and, in many instances, the major products are those derived from *C*-alkylation. During the past decades, several new procedures have been developed[5,7] in which *N*-alkylation of heterocycles can be accomplished with little or no interference from *C*-alkylation. The PTC procedures appear to be the most useful in terms of mildness of conditions, yield, and convenience. Under PTC conditions, exclusive *N*-alkylation is usually observed.[8]

We have sought to develop a general method of the *N*-alkylation of heterocycles possessing an acidic hydrogen .[9] Such a procedure should retain the convenience of PTC methods but should be free from some limitations related to PTC systems[8] and much faster. Therefore we decided to explore the use of microwave heating under solvent free PTC conditions for *N*-alkylation of heterocycles.

Recently, the *N*-alkylation of 1,2,4-triazole and benzotriazole was achieved in the absence of solvent.[10] The alkylation was performed either in basic medium under solvent free PTC conditions or in the absence of base by conventional and microwave heating. In the case of triazole alkylation, Loupy *at al.* claim that microwave irradiation produces specific (non-thermal) effects both on reactivity and selectivity.[10] More recently, the 'dry' microwave *N*-alkylation of indole was attempted by Abramovitch *et al.*,[11] but the yield of the reaction was unsatisfactory low.

We now report here the remarkable fast method of synthesis of *N*-alkylazaheterocycles in 'dry' media under microwave irradiation Fig. 1.



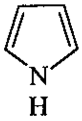
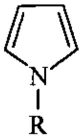
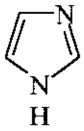
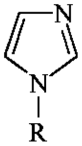
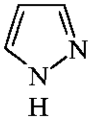
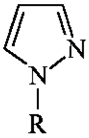
Z,Y = C: pyrrole; Z = N, Y = C: imidazole; Z = C, Y = N:
X = Cl, Br, I; TBAB: tetrabutylammonium

Figure 1: The reactions of five-membered azaheterocycles with alkyl halides under microwave irradiation.

The reactions were carried out by simply mixing of an azaheterocycle compound with 50% excess of an alkyl halide and a catalytic amount of tetrabutylammonium bromide (TBAB). The mixtures were adsorbed either on the mixture of potassium carbonate and potassium hydroxide or potassium carbonate and then irradiated in an open vessel in a domestic microwave oven for 1-10 min. For five-membered azaaromatic compounds the results are summarized in Table 1, while the results for the reactions of azaaromatic compounds fused with benzene rings are given in Table 2 (Fig. 2).

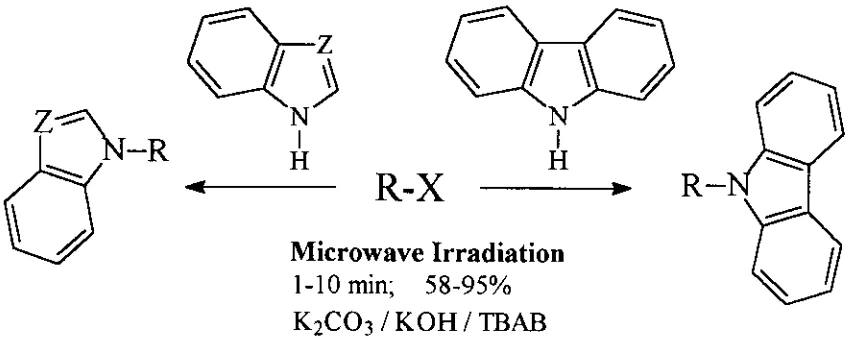
Since the shape and size of the reaction vessel are important factors for the heating of dielectrics in a microwave oven, the preferred reaction vessel is a tall beaker of much larger capacity than the volume of

Table 1. The reactions of five-membered azaheterocycles with alkyl halides under irradiation in a microwave oven.

Compound	R-X	Product	Time [s]	Power [W]	Yield [%]	mp/bp [°C]/[°C/Torr]	References
	Benzyl chloride		50	300	77	120-2/10	14
	1-Chlorodecane		60	300	74	142-5/10	
	1-Bromopentane		34	300	58	75-87/10	
	Benzyl chloride		40	300	89	71-72	15
	1-Chlorodecane		40	300	85	148-51/10	17
	1-Bromopentane		28	300	73	91-93/10	
	Benzyl chloride		35	300	89	oil	16
	1-Chlorodecane		50	300	84	142-4/10	16
	1-Bromopentane		25	300	61	100-2/30	16

Reagents ratio: heterocyclic compound (5 mmol), alkyl halide (7.5 mmol), tetrabutylammonium bromide (0.5 mmol), K_2CO_3 (20 mmol), KOH (20 mmol).

the reaction mixture, and bearing a loose cover. A large Erlenmeyer flask with a funnel as a loose top cap can be used in place of the beaker. Superheating of liquids is common under microwave irradiation, thus the strategy of the reactions is to keep the reaction temperature substantially below the boiling point of each compound used for the reaction. Since it is difficult to measure temperature in a household microwave oven, one of the best solutions is to repeat an experiment several times increasing the power slowly so that vapours do not escape outside of the flask.

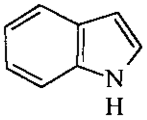
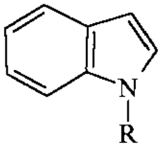
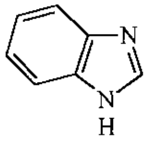
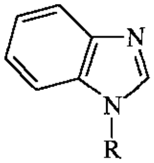
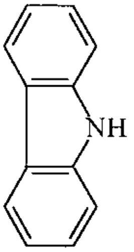
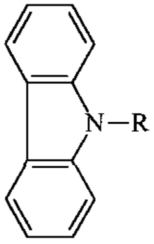


Z = C: indole; Z = N: benzimidazole; TBAB: tetrabutylammonium bromide

Figure 2: The reactions between five-membered azaheterocycles fused with benzene rings and alkyl halides under microwave irradiation.

After the reaction, the work-up procedure is reduced a to treatment with an appropriate solvent (e.g., *THF* or *CH_2Cl_2*), purification by distillation in a Kugelrohr apparatus or recrystallization. If necessary before recrystallization, compounds can be separated from starting materials by means of flash chromatography. Melting and boiling points of all the compounds are in good agreement with literature data. The reaction procedures are not optimised yet.

Table 2. The reactions of five-membered azaheterocycles fused with benzene rings with alkyl halides under irradiation in a microwave oven.

Compound	**R-X**	**Product**	**Time** [s]	**Power** [W]	**Yield** [%]	**mp/bp** [°C]/[°C/Torr]	**References**
	Benzyl chloride		30	300	94	43-45	12
	1-Chlorodecane		45	300	88	oil	
	1-Bromopentane		27	300	86	138-40/10	12
	3-Chloropropene		38	300	83	119-21/10	13
	Benzyl chloride		55	300	78	115-117	14
	1-Chlorodecane		65	300	64	oil	
	1-Bromopentane		35	300	75	oil	14
	Benzyl chloride		240*	450	95	116-120	18, 19
	1-Chlorodecane		600*	450	82	18-20	
	1-Bromobutane		420*	450	85	57-59	18, 19
	1-Bromopentane		300*	450	79	51-53	18

Reagents ratio: heterocyclic compound (5 mmol), alkyl halide (7.5 mmol), tetrabutylammonium bromide (0.5 mmol), K_2CO_3 (20 mmol), KOH (20 mmol); * heterocyclic compound (5 mmol), alkyl halide (7.5 mmol), tetrabutylammonium bromide (0.5 mmol), K_2CO_3 (20 mmol).

In conclusion, we have developed a simple and economical method for the *N*-alkylation of azaheterocycles that occurs remarkable fast under mild conditions using inexpensive reagents and a household microwave oven as the irradiation source. Moreover, the procedure is alternative to those which rely on the use of dipolar aprotic solvents, thallium salts of heterocycles, and several procedure that rely on "standard" PTC methods.

EXPERIMENTAL

Elemental analyses were performed on a Perkin-Elmer 240 microanalyzer. Melting points, measured on a Boetius-PHMK 05 microscope plates, are uncorrected. The reactions were monitored by means of TLC. ^{1}H- and ^{13}C-NMR spectra were recorded with a TESLA 487 C spectrometer, TMS being used as a internal standard; the chemical shifts are expressed in δ values downfield from TMS. MS spectra were recorded on a Hewllet-Packard 5985 spectrometer. IR spectra were performed on a Bio-Rad spectrophotometer, and the wave numbers were given with a precision of 2 cm^{-1}.

General Procedure for *N*-Alkylation of Azaheterocycle: A mixture of an azaheterocyclic compound (5.0 mmol), alkyl halide (7.5 mmol), tetrabutylammonium bromide - TBAB (0.17 g, 0.50 mmol), and potassium carbonate (2.8 g, 20 mmol) or mixture of potassium carbonate (2.8 g, 20 mmol) and potassium hydroxide (1.1 g, 20 mmol) was heated in a domestic microwave oven in an open Erlenmeyer flask for an appropriate time (see Table 1 or Table 2). After being cooled down, the reaction mixture was extracted with methylene chloride or THF (2 x 25 mL). Then the extract was dried with $MgSO_4$, filtered, and the solvent was evaporated to dryness. Liquid compounds were purified on Kugelrohr distillation apparatus, while solid compounds were purified by means of flash chromatography to afford desired *N*-alkyl derivatives of azaheterocycle compound, yield: 58-95%.

***N*-Benzylpyrrole** - **MS** m/z = 157 (M$^+$, 42), 91 (100). **IR** (film) ν = 3100(m), 3064(s), 3031(s), 2923(m), 2861(m), 1606(w), 1553(w), 1497(s), 1453(s), 1357(s), 1284(s), 1088(s), 1069(s), 1029(m), 968(m), 724(s) cm^{-1}. **^{1}H NMR** (80 MHz, $CDCl_3$) δ(ppm) = 5.04 (s, 2H (-CH_2-)), 6.18 (t, J = 2.2 Hz, 2H) and 6.67 (t, J = 2.2 Hz, 2H) and 7.02~7.34 (m, 5H) aromatic protons. ***N*-Decylpyrrole** - **MS** m/z = 207 (M$^+$, 35), 81 (100), 67 (11). **IR** (film) ν = 3053(w), 2966(s), 2855(s), 1506(m), 1465(m), 1456(m), 1281(m), 1112(s), 1080(s), 728(s) cm^{-1}. **^{1}H NMR** (80 MHz, $CDCl_3$) δ(ppm) = 0.88 (t, J = 6.1 Hz, 3H (-CH_3)), 1.26 (s, 14H, 7x (-CH_2-)), 1.38~1.89 (m, 2H (-CH_2-)), 3.85 (t, J = 7.3 Hz, 2H, (-CH_2-)), 6.12 (t, J = 2.2 Hz, 2H) and 6.63(t, J = 2.2 Hz, 2H) aromatic protons. Anal. Calcd for $C_{14}H_{25}N$: C 81.09, H 12.15, N 6.75. Found: C 80.98, H 12.04, N 6.86. ***N*-Pentylpyrrole** - **MS** m/z = 137 (M$^+$, 51), 81 (100), 67 (13). **IR** (film) ν = 3053(w), 3030(w), 2965(s), 2854(s), 1507(m), 1469(m), 1456(m), 1279(m), 1109(s), 1080(s), 728(s) cm^{-1}. **^{1}H NMR** (80 MHz, $CDCl_3$) δ(ppm) = 0.86 (t, J = 6.1 Hz, 3H (-CH_3)), 1.28 (m, 4H, 2x (-CH_2-)), 1.46~1.89 (m, 2H (-CH_2-)), 3.80 (t, J = 7.3 Hz, 2H, (-CH_2-)), 6.16 (t, J = 2.2 Hz, 2H) and 6.65 (t, J = 2.2 Hz, 2H) aromatic protons. Anal. Calcd for $C_9H_{15}N$: C 78.78, H 11.02, N 10.21. Found: C 78.59, H 10.01, N 11.35.

***N*-Benzylimidazol** - **MS** m/z = 158 (M^+, 34), 91 (100), 68 (22). **IR** (KBr) ν = 3113(m), 3066(m), 3031(m), 2941(w), 2861(w), 1634(s), 1604(m), 1587(m), 1507(s), 1453(s), 1391(m), 1365(m), 1279(m), 1232(s), 1202(m), 1074(s), 1029(m), 825(m), 729(s) cm^{-1}. **^{1}H NMR** (80 MHz, $CDCl_3$) δ(ppm) = 5.09 (s, 2H ($-CH_2-$)), 6.88~7.52 (m, 8H (aromatic protons)). ***N*-Decylimidazole** - **MS** m/z = 208 (M^+, 21), 82 (100), 68 (26). **IR** (film)ν = 3111(w), 2926(s), 2855(s), 1634(w), 1511(w), 1507(s), 1465(s), 1376(m), 1283(m), 1230(s), 1208(s), 1078(s), 908(m), 820(m), 728(s) cm^{-1}. **^{1}H NMR** (80 MHz, $CDCl_3$) δ(ppm) = 0.88 (t, J = 6.0 Hz, 3H ($-CH_3$)), 1.26 (s, 14H, 7x ($-CH_2-$)), 1.64~1.91 (m, 2H ($-CH_2-$)), 3.83 (t, J = 7.2 Hz, 2H, ($-CH_2-$)), 6.88~7.45 (m, 3H (aromatic protons)). ***N*-Pentylimidazole** - **MS** m/z = 138 (M^+, 31), 82 (100). **IR** (film) ν = 3054(w), 2956(s), 2929(s), 2870(s), 1634(w), 1511(m), 1485(m), 1463(s), 1399(w), 1315(s), 1234(m), 1193(m), 1113(m), 761(m), 738(s) cm^{-1}. **^{1}H NMR** (80 MHz, $CDCl_3$) δ(ppm) = 0.89 (t, J = 5.9 Hz, 3H ($-CH_3$)), 1.19~1.37 (m, 4H 2× ($-CH_2-$)), 1.66~1.90 (m, 2H ($-CH_2-$)), 4.28 (t, J = 7.0 Hz, 2H ($-CH_2$ -)), 6.42~7.64 (m, 3H (aromatic protons)). Anal. Calcd for $C_8H_{14}N_2$: C 69.52, H 10.21, N 20.27. Found: C 70.34, H 11.14, N 19.39. ***N*-Benzylpyrazole** - **MS** m/z = 158 (M^+, 48), 157 (100), 91 (84). **IR** (film) ν = 3109(m), 3065(m), 3032(s), 2934(m), 2861(w), 1631(w), 1607(w), 1513(s), 1496(s), 1454(s), 1436(m), 1396(s), 1279(m), 1089(s), 1049(s), 1029(w), 970(m), 917(w), 754(s) cm^{-1}. **^{1}H NMR** (80 MHz, $CDCl_3$) δ(ppm) = 5.15 (s, 2H ($-CH_2-$)), 6.22~7.34 (m, 8H (aromatic protons)). ***N*-Decylpyrazole** - **MS** m/z = 208 (M^+, 18), 81 (100), 68 (39). **IR** (film) ν = 3107(w), 2926(s), 2855(s), 1634(w), 1513(m), 1465(s), 1456(s), 1437(m), 1396(s), 1279(m), 1089(s), 1044(s), 996(m), 872(w), 747(s) cm^{-1}. **^{1}H NMR** (80 MHz, $CDCl_3$) δ(ppm) = 0.87 (t, J = 6.0 Hz, 3H ($-CH_3$)), 1.26 (s, 14H, 7x ($-CH_2-$)), 1.75~2.03 (m, 2H ($-CH_2-$)), 4.20 (t, J = 7.2 Hz, 2H, ($-CH_2-$)), 6.45~7.67 (m, 3H (aromatic protons)). ***N*-Pentylpyrazole** - **MS** m/z = 138 (M^+, 23), 81 (100), 68 (21). **IR** (film) ν = 3108(w), 3064(m), 2927(s), 2852(s), 1632(w), 1513(m), 1495(s), 1456(s), 1437(m), 1395(m), 1090(m), 1043(s), 994(m), 876(w), 747(s) cm^{-1}. **^{1}H NMR** (80 MHz, $CDCl_3$) δ(ppm) = 0.86 (t, J = 6.0 Hz, 3H ($-CH_3$)), 1.26 (m, 4H, 2x ($-CH_2-$)), 1.76~1.98 (m, 2H ($-CH_2-$)), 4.22 (t, J = 7.1 Hz, 2H, ($-CH_2-$)), 6.45~7.67 (m, 3H (aromatic protons)). ***N*-Benzylindole** - **MS** m/z = 207 (M^+, 76), 91 (100). **IR** (KBr) ν = 3087(w), 3054(m), 3030(m), 2924(m), 2853(w), 1510(m), 1486(m), 1464(s), 1438(m), 1399(w), 1357(m), 1317(s), 1255(w), 1181(s), 1123(w), 1029(w), 763(m), 740(s) cm^{-1}. **^{1}H NMR** (80 MHz, $CDCl_3$) δ(ppm) = 5.29 (s, 2H ($-CH_2-$)), 6.51~7.64 (m, 11H (aromatic protons)). ***N*-Decylindole** - **MS** m/z = 257 (M^+, 47), 130 (100), 117 (15). **IR** (film) ν = 3054(w), 2926(s), 2854(s), 1612(w), 1556 (w), 1512(m), 1485(m), 1463(s), 1399(w), 1316(m), 1013(w), 761(m), 738(s) cm^{-1}. **^{1}H NMR** (80 MHz, $CDCl_3$) δ(ppm) = 0.87 (t, J = 6.1 Hz,3H ($-CH_3$)), 1.25 (s, 14H, 7x ($-CH_2-$)), 1.68~1.92 (m, 2H ($-CH_2-$)), 4.20 (t, J = 7.2 Hz, 2H, ($-CH_2-$)), 6.45~7.67 (m, 6H (aromatic protons)). Anal. Calcd for $C_9H_{15}N$: C 83.99, H 10.57, N 5.44. Found: C 83.87, H 10.43, N 5.58. ***N*-Pentylindole** - **MS** m/z = 187

(M^+, 36), 130 (100), 117 (10). **IR** (film) ν = 3087(w), 3054(m), 2958(s), 2930(s), 2870(s), 1612(w), 1556(w), 1512(s), 1484(m), 1463(s), 1399(m), 1316(s), 1247(m), 1192(m), 762(m), 739(s) cm^{-1}. **^{1}H NMR** (80 MHz, $CDCl_3$) δ(ppm) = 0.86 (t, J = 6.1 Hz, 3H ($-CH_3$)), 1.65~1.89 (m, 4H 2× ($-CH_2-$)), 1.65~1.86 (m, 2H ($-CH_2-$)), 4.28 (t, J = 7.0 Hz, 2H ($-CH_2$ -)), 6.42~7.64 (m, 6H (aromatic protons)). ***N*-Allylindole - MS** m/z = 157 (M^+, 97), 156 (100), 117 (16). **IR** (film) ν = 3084(w), 3055(w), 2984(w), 2918(w), 2857(w), 1510(m), 1485(m), 1462(s), 1414(w), 1316(m), 1258(w), 1196(m), 1023(w), 990(w), 925(m), 762(m), 740(s) cm^{-1}. **^{1}H NMR** (80 MHz, $CDCl_3$) δ(ppm) = 4.64~4.74 (m, 2H ($-CH_2-$)), 5.09~5.24 (m, 2H ($=CH_2$)), 5.75~5.99 (m, 1H (-CH)), 6.48~7.68 (m, 6H (aromatic protons)). ***N*-Benzylbenzimidazole - MS** m/z = 208 (M^+, 32), 91 (100). **IR** (KBr) ν = 3082(m), 3032(m), 2945(w), 2924(w), 2865(w), 1613(m), 1495(s), 1443(s), 1364(s), 1331(m), 1285(m), 1265(s), 1201(s), 972(m), 886(s), 774(s), 731(s)cm^{-1}. **^{1}H NMR** (80 MHz, $CDCl_3$) δ(ppm) = 5.34 (s, 2H ($-CH_2-$)), 7.13~7.93 (m, 10H (aromatic protons)).

***N*-Decylbenzimidazole - MS** m/z = 258 (M, 67), 131 (100), 118 (74). **IR** (film) ν = 3086(m), 3058(m), 3039(m), 2955(s), 2927(s), 2854(s), 1615(m), 1496(s), 1460(s), 1365(m), 1330(m), 1286(s), 1260(m), 1202(m), 1109(w), 890(w), 743(s) cm^{-1}. **^{1}H NMR** (80 MHz, $CDCl_3$) δ(ppm) = 0.87 (t, J = 6.1 Hz, 3H ($-CH_3$)), 1.24 (s, 14H, 7x ($-CH_2-$)), 1.58~1.89 (m, 2H ($-CH_2-$)), 4.34 (t, J = 7.1 Hz, 2H, ($-CH_2-$)), 6.97~7.98 (m, 10H (aromatic protons)). Anal. Calcd for $C_{17}H_{26}N_2$: C 79.02, H 10.14, N 10.84. Found: C 78.88, H 10.02, N 10.97. ***N*-Pentylbenzimidazole - MS** m/z = 188 (M^+, 48), 131 (100), 118 (10). **IR** (film) ν = 3089(w), 3060(w), 2959(s), 2932(s), 2873(m), 1615(w), 1496(s), 1457(s), 1373(m), 1330(m), 1286(m), 1260(m), 1202(s), 1109(s), 1008(w), 891(w), 743(s) cm^{-1}. **^{1}H NMR** (80 MHz, $CDCl_3$) δ(ppm) = 0.87 (t, J = 6.1 Hz, 3H ($-CH_3$)), 1.26 (m, 4H, 2x ($-CH_2-$)), 1.48~1.85 (m, 2H ($-CH_2-$)), 4.30 (t, J = 7.1 Hz, 2H, ($-CH_2-$)), 6.98~8.05 (m, 10H (aromatic protons)).

***N*-Benzylcarbazole - MS**: m/z = 237 (M^+, 72), 180 (7), 91 (100). **IR** (KBr): 3064(w), 3055(w), 3038(w), 2939(w), 1625(w), 1607(m), 1496(m), 1487(s), 1473(s), 1349(m), 1335(s), 1321(w), 1220(m), 1150(w), 849(w), 755(s) cm^{-1}. **^{1}H NMR** (80 MHz, $CDCl_3$) δ(ppm) = 5.49 (s, 2H ($-CH_2-$)), 7.12~8.17 (m, 13H (aromatic protons)). ***N*-Decylcarbazole - MS**: m/z = 307 (M^+, 36), 180 (100), 166 (11). **IR** (film): 3053(w), 2926(s), 2853(s), 1634(w), 1597(m), 1542(w), 1486(s), 1463(m), 1456(s), 1375(m), 1347(s), 1326(s), 1231(w), 1152(w), 748(s) cm^{-1}. **^{1}H NMR** (80 MHz, $CDCl_3$) δ(ppm) = 0.87 (t, J = 5.9 Hz, 3H ($-CH_3$)), 1.23 (s, 14H, 7× ($-CH_2-$)), 1.41~1.52 (m, 2H ($-CH_2-$)), 4.26 (t, J = 7.5 Hz, 2H ($-CH_2$ -)), 7.17~8.15 (m, 8H (aromatic protons)). Anal. Calcd for $C_{22}H_{29}N$: C 85.94, H 9.51, N 4.56. Found: C 85.76, H 9.38, N 4.74. ***N*-Butylcarbazole - MS**: m/z = 223 (M^+, 30), 180 (100), 166 (10). **IR** (KBr): 3045(m), 2953(s), 2928(s), 2869(s), 2858(s), 1625(w), 1592(w), 1485(s), 1452(s), 1378(m), 1349(s), 1326(s), 1240(w), 1212(s), 1150(s), 1118(w), 875(w), 749(s), 724(s) cm^{-1}. **^{1}H NMR** (80 MHz, $CDCl_3$) δ(ppm) =

0.93 (t, J = 6.4 Hz, 3H (-CH_3)), 1.26~1.48 (m, 2H (-CH_2-)), 1.77~1.85 (m, 2H (-CH_2-)), 4.29 (t, J = 7.1 Hz, 2H (-CH_2 -)), 7.17~8.15 (m, 8H (aromatic protons)). ***N*** **-Pentylcarbazole** - **MS**: m/z = 237 (M^+, 31), 180 (100), 166 (16). **IR** (KBr): 3051(m), 2951(s), 2931(s), 2866(s), 2856(s), 1595(s), 1485(s), 1456(s), 1373(m), 1349(s), 1328(s), 1234(s), 1196(m), 1151(m), 1120(w), 873(w), 752(s) cm^{-1}. **^{1}H NMR** (80 MHz, $CDCl_3$) δ(ppm) = 0.87 (t, J = 6.1 Hz, 3H (-CH_3)), 1.28~1.48 (m, 4H 2× (-CH_2-)), 1.78~1.84 (m, 2H (-CH_2-)), 4.28 (t, J = 7.2 Hz, 2H (-CH_2 -)), 7.17~8.15 (m, 8H (aromatic protons)).

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