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Synthesis of 2-Aryl-1-arylmethyl-1*H*-1,3-benzimidazole Derivatives Using Silica-bonded Propyl-*S*-sulfonic Acid as Recyclable Solid Acid Catalyst

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A highly selective synthesis of 2-aryl-1-arylmethyl-1*H*-1,3-benzimidazoles from the reaction of *o*-phenylenediamine and aromatic aldehydes in the presence of silica-bonded propyl-*S*-sulfonic acid (SBSSA) at 80 ℃ in water in good to excellent yields was developed.

Keywords silica-bonded propyl-*S*-sulfonic acid, aldehydes, 2-aryl-1-arylmethyl-1*H*-1,3-benzimidazoles, *o*-phenylenediamine, water

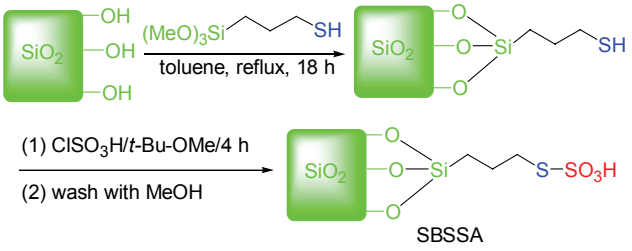
Introduction

The benzimidazole nucleus is of significant importance to medicinal chemistry. Benzimidazoles are components of vitamin B12 and are related to the DNA base purine and the stimulant caffeine.[1] Interest in benzimidazole-containing structures stems from their widespread occurrence in molecules that exhibit significant activity.[2,3] In light of the affinity they display towards a variety of enzymes and protein receptors, medicinal chemists would certainly classify them as 'privileged sub-structures' for drug design.[4,5]

The traditional synthesis of benzimidazoles involves the reaction between *o*-phenylenediamines and carboxylic acid or its derivatives (nitriles, amidates and orthoesters) under harsh dehydrating conditions.[6,7] Benzimidazoles have also been prepared on solid-phase to provide a combinatorial approach.[8] The most popular strategies for their synthesis utilize *o*-nitroanilines as intermediates or resort to direct *N*-alkylation of an unsubstituted benzimidazole.[9] A number of synthetic protocols that involve intermediate *o*-nitroanilines have evolved to include the synthesis of benzimidazoles on solid support.[10] Another method for the synthesis of these compounds is the reaction of *o*-phenylenediamines with aldehydes in the presence of acidic catalysts such as sulfamic acid,[11] silica sulfuric acis,[12] Zn-proline,[13] [Hbim]BF_4,[14] $Fe(ClO_4)_3$,[15] $M(HSO_4)_n$,[16] 1-heptanesulfonic acid sodium salt,[17] poly(*N*,*N*-dibromo-*N*-ethylbenzene-1,3-disulfonamide),[18] $[(CH_2)_4SO_3HMIM][HSO_4]$,[19] acetic acid[20] and sulfuric acid ([3-(3-silicapropyl)sulfanyl]propyl)ester[21] under various reaction conditions.

Recently, silica-bonded propyl-*S*-sulfonic acid (SBSSA) was prepared by reaction of 3-mercaptopropylsilica (MPS) and chlorosulfonic acid (Scheme 1). This solid acid was used as a catalyst in the synthesis of acylals,[22] silyl ethers,[23] quinoxalines,[24] coumarins,[25] bis-pyrazol-5-ols,[26] acridines,[27] trisubstituted imidazoles,[28] *α*-amino nitriles,[29] bis-indolylmethanes,[30] and 2,3-dihydroquinazolin-4(1*H*)-ones.[31]

Scheme 1 Preparation of silica-bonded propyl-*S*-sulfonic acid (SBSSA)



Water emerged as a useful alternative solvent for several organic reactions owing to many of its potential advantages such as safety, economy, easy availability, nontoxicity and environmental concern.[32-34] Reactions in water have been very useful to the synthetic chemist for many years, and their utility is reflected by the many studies to discover new processes with which they can be performed catalytically and chemoselectively.

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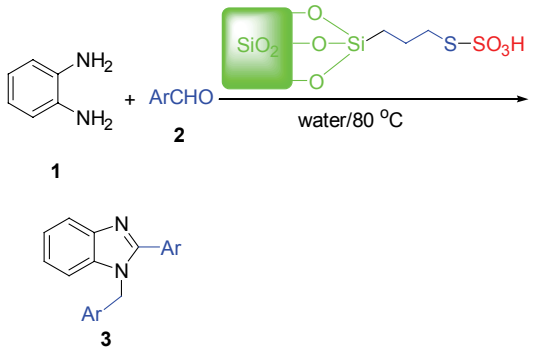
Results and Discussion

During the course of our studies towards the development of new routes to the synthesis of highly substituted heterocycles using solid acid catalysts,[21-31] we found a valid and an efficient procedure for the synthesis of 2-aryl-1-arylmethyl-1*H*-1,3-benzimidazoles via one-pot condensation reaction between *o*-phenylenediamine and aldehydes in the presence of SBSSA as an inexpensive solid acid catalyst (Eq. 1).



(1)

To study the effect of catalyst loading on the synthesis of 2-aryl-1-arylmethyl-1*H*-1,3-benzimidazoles, the reaction between *o*-phenylenediamine and 4-methylbenzaldehyde was chosen as the model reaction in water under reflux conditions (Table 1).

Table 1 The reaction of *o*-phenylenediamine with 4-methoxybenzaldehyde in the presence of different amounts of SBSSA at 80 ℃ in water[a]

Entry	Amount of catalyst/g	Product	Time/min	Yield[b]/%
1	—	—	180	—
2	0.01	**3**	35	87
3	0.03	**3**	20	90
3	0.05	**3**	10	93
4	0.1	**3**	15	92

[a] Reaction conditions: 1,2-phenylenediamine (1 mmol), aldehyde (2 mmol) at 80 ℃ in water (5 mL). [b] Isolated yield.

The results show clearly that SBSSA is an effective catalyst for this transformation and in the absence of SBSSA the reaction did not take place even after 3 h (Table 1, Entriy 1). As indicated in Table 1, the best result has been obtained with amount of 0.05 g (0.16 mol%) SBSSA in terms of reaction time and isolated yield. The higher the catalyst loading (0.1 g), the longer time the condensation to accomplish (Table 1, Entry 4). This is may be due to the decrease of the nucleophilicity of *o*-phenylenediamine when the acidic catalyst loading was increased.

Several aromatic aldehydes with different substituents on the aromatic ring were subjected to the condensation reaction. As shown in Table 2, arylaldehydes without substituents gave the desired benzimidazoles in good yields (**3a**, **3k**). Aldehydes bearing electron-donating substituents gave the desired benzimidazoles **3b**—**3e** in a short reaction times (10 to 30 min) and very good to excellent yields (Table 2). Arylaldehydes with electron-withdrawing substituents such as 4-fluoro- and 4-cyano-benzaldehyde gave the corresponding benzimidazoles **3h**, and **3j** in very good yields.

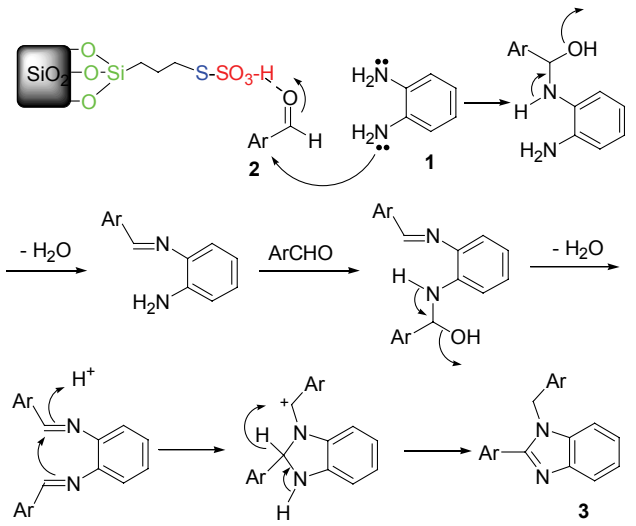
Table 2 Synthesis of 2-aryl-1-arylmethyl-1*H*-1,3-benzimidazoles catalyzed by SBSSA at 80 ℃ in water[a]

Entry	Ar (**3**)	Product	Time/min	Yield[b]/%	m.p./℃	Ref.
1	C_6H_5-	**3a**	20	87	130—132	[11]
2	4-MeO-C_6H_4-	**3b**	10	93	129—131	[19]
3	2-MeO-C_6H_4-	**3c**	20	92	150—151	[19]
4	4-Me-C_6H_4-	**3d**	20	88	128—130	[19]
5	2-Me-C_6H_4-	**3e**	30	90	139—140	[13]
6	4-Cl-C_6H_4-	**3f**	25	92	135—137	[19]
7	2-Cl-C_6H_4-	**3g**	35	90	161—163	[11]
8	4-F-C_6H_4-	**3h**	30	84	82—84	[13]
9	4-Br-C_6H_4-	**3i**	30	91	154—156	[13]
10	4-NC-C_6H_4-	**3j**	35	92	190—192	[20]
11	2-Furyl	**3k**	25	75	92—94	[19]

[a] Reaction conditions: 1,2-phenylenediamine (1 mmol), aldehyde (2 mmol), and SBSSA (0.05 g) at 80 ℃ in water (5 mL). [b] Isolated yield.

Some possible mechanisms of this condensation reaction had been reported before by Ravi *et al.*[13] and Jadhav and co-workers.[17] In the proposed mechanism (Scheme 2), aromatic aldehyde was activated by SBSSA during the reaction. Initially Schiff's base formation was formed between arylenediamine and substituted aromatic aldehydes followed by cyclization, and 1,3-hydride transfer.

Scheme 2 Plausible mechanism for the synthesis of 2-aryl-1-arylmethyl-1-benzo[*d*]imidazoles



The possibility of recycling of the catalyst was examined. For this reason, the reaction of *o*-phenylene-

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diamine with 4-methylbenzaldehyde in the presence of SBSSA was studied at 80 ℃ in water. Upon completion, the reaction mixture was filtered and washed with warm ethanol. The product was recrystallized from hot water-ethanol (V∶V=1∶1). The recycled catalyst was reused four times without any treatment. No observation of appreciable loss in its catalytic activities was shown (Figure 1).

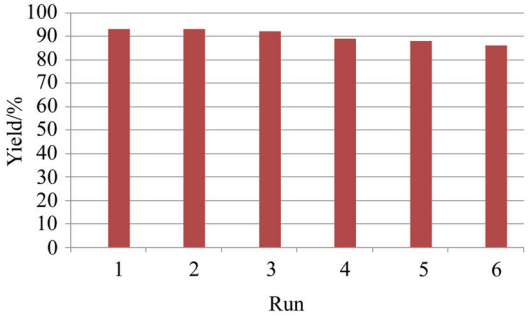

Figure 1 Recyclability of silica bonded propyl-*S*-sulfonic acid (0.05 g) as catalyst in the condensation reaction of *o*-phenylenediamine (1 mmol) and 4-methoxybenzaldehyde (2 mmol) in water (5 mL) at 80 ℃. Reaction time=10 min.

In conclusion, heterogeneous conditions, green solvent, easy and clean work-up, high yields and recovery of the catalyst makes this method practical for the synthesis of benzimidazole derivatives.

Experimental

General

Chemicals were purchased from Fluka, Merck and Aldrich Chemical Companies. All the products were characterized by comparison of their IR, ^{1}H NMR and ^{13}C NMR spectroscopic data and their melting points with the reported values.[11-20] Silica-bonded propyl-*S*-sulfonic acid (SBSSA) was prepared according to previously reported procedure.[22,23]

Catalyst perparation

To a magnetically stirred mixture of 3-mercaptopropylsilica (5 g) in *tert*-buylmethyl ether (20 mL), chlorosulfonic acid (1.0 g, 9 mmol) was added dropwise at 0 ℃ during 2 h. After addition was complete, the mixture was stirred for 2 h until HCl gas evolution was stopped. Then, the mixture was filtered and washed with methanol (30 mL) and dried at room temperature to obtain silica-bonded propyl-*S*-sulfonic acid (SBSSA) as cream powder (5.22 g). Sulfur content of the samples by conventional elemental analysis was 16.12%. The FT-IR spectrum of the catalyst is shown in Figure 2. The catalyst is a solid, and the solid state IR spectrum was recorded using the KBr disk technique. For silica (SiO_2), the major peaks are broad anti symmetric Si—O—Si stretching from 1200 to 1000 cm^{-1} and symmetric Si—O—Si stretching near 802 cm^{-1}, and bending modes lie near 470 cm^{-1}. For sulfonic acid functional group, the FT-IR absorption range of the O=S=O asymmetric and symmetric stretching modes lie in 1176.5 and 1072.3 cm^{-1} respectively, the S—O stretching mode lies in 550—700 cm^{-1} and that of S—S stretching mode lies from 400 to 500 cm^{-1}. FT-IR spectrum shows the overlap asymmetric and symmetric stretching bands of SO_2 with Si—O—Si stretching bands in the silica functionalized *S*-sulfonic acid. The spectrum also shows a broad OH stretching absorption from 3600 to 2491.9 cm^{-1} (Figure 2).

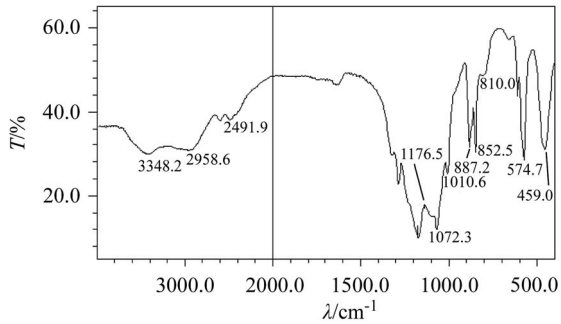

Figure 2 FT-IR spectra of silica-bonded propyl-*S*-sulfonic acid.

pH analysis of the SBSSA

To an aqueous suspension of 0.1 g SBSSA, NaOH (1.8 mL, 0.1 mol•L^{-1}) was added. This is equal to a loading of 1.8 mmol SO_3H/g. So, all of the SH functional groups in 3-mercaptopropylsilica were sulfonated. According to previous reports,[35] the loading of 3-mercaptopropylsilica is 0.33 mmol/g.

General procedure for the synthesis of 2-aryl-1-arylmethyl-1*H*-1,3-benzimidazoles derivatives

To a mixture of *o*-phenylenediamine (1 mmol), aromatic aldehyde (2 mmol) in water (5 mL), SBSSA (0.05 g) were added and the reaction mixture was stirred in an oil bath at 80 ℃ for the appropriate time (see Table 2). The progress of the reaction was monitored by TLC. After completion of the reaction, the mixture was filtered and the precipitates were solved in hot ethanol (10 mL×3) and filtered to afford the desired product. Finally the crude product was recrystallized from water-ethanol (V∶V=1∶1). The recovered catalyst was dried and reused for subsequent runs.

1-Benzyl-2-phenylbenzimidazole (**3a**): m.p. 130—132 ℃ (Lit.[11] 129—130 ℃); ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 5.37 (s, 2H), 7.02 (dd, J_1=8.1 Hz, J_2=1.5 Hz, 2H), 7.11—7.17 (m, 2H), 7.21—7.27 (m, 4H), 7.33—7.39 (m, 3H), 7.59—7.62 (m, 2H), 7.79 (dd, J_1=8.7 Hz, J_2=1.0 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 48.42, 110.59, 120.02, 122.73, 123.09, 126.01, 127.82, 128.80, 129.11, 129.30, 129.97, 130.09, 136.09, 136.42, 143.20, 154.22.

1-(4-Methoxybenzyl)-2-(4-methoxyphenyl)-1*H*-benzimidazole (**3b**): m.p. 129—131 ℃ (Lit.[19] 129—130 ℃); ^{1}H NMR ($CDCl_3$, 500 MHz) δ: 3.81 (s, 3H), 3.88 (s, 3H), 5.41 (s, 2H), 6.89 (dt, J_1=6.9 Hz, J_2=2.4 Hz, 2H), 7.01 (dt, J_1=8.8 Hz, J_2=2.4 Hz, 2H), 7.06 (d, J=8.7

Hz, 2H), 7.24—7.26 (m, 2H), 7.31—7.34 (m, 1H), 7.68 (dt, J_1=8.8 Hz, J_2=2.4 Hz, 2H), 7.88 (d, J=8.0 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ: 48.33, 55.71, 55.80, 110.85, 114.63, 114.86, 120.09, 122.75, 123.00, 123.20, 127.64, 128.86, 131.15, 136.46, 154.47, 159.56, 161.37.

1-(2-Methoxybenzyl)-2-(2-methoxyphenyl)-1*H*-benzimidazole (**3c**): m.p. 150—151 ℃ (Lit.[19] 149—152 ℃); ^{1}H NMR ($CDCl_3$, 500 MHz) δ: 3.59 (s, 3H), 3.77 (s, 3H), 5.24 (s, 2H), 6.70 (d, J=6.8 Hz, 1H), 6.76 (t, J=7.4 Hz, 1H), 6.83 (d, J=8.2 Hz, 1H), 6.96 (d, J=8.3 Hz, 1H), 7.05 (t, J=7.5 Hz, 1H), 7.17—7.28 (m, 4H), 7.45 (dt, J_1=7.9 Hz, J_2=1.6 Hz, 1H), 7.54 (dd, J_1=7.5 Hz, J_2=1.6 Hz, 1H), 7.86 (d, J=8.0 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ: 44.07, 55.59, 55.63, 110.40, 111.25, 111.31, 120.07, 120.83, 121.25, 122.56, 123.03, 124.83, 127.26, 128.23, 128.89, 131.98, 132.82, 156.96, 158.04.

1-(4-Methylbenzyl)-2-(4-methylphenyl)-1*H*-benzimidazole (**3d**): m.p. 128—130 ℃ (Lit.[19] 128—130 ℃); ^{1}H NMR ($CDCl_3$, 500 MHz) δ: 2.33 (s, 3H), 2.40 (s, 3H), 5.41 (s, 2H), 6.99 (d, J=7.8 Hz, 2H), 7.13 (d, J=7.8 Hz, 2H), 7.19—7.26 (m, 4H), 7.30 (t, J=7.3 Hz, 1H), 7.59 (d, J=7.8 Hz, 2H), 7.86 (d, J=8.0 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ: 21.50, 21.85, 48.65, 110.96, 120.18, 123.06, 123.33, 126.32, 127.44, 129.61, 129.88, 130.13, 133.81, 136.45, 137.90, 140.55, 154.68.

1-(2-Methylbenzyl)-2-(2-methylphenyl)-1*H*-benzimidazole (**3e**): m.p. 139—140 ℃; ^{1}H NMR ($CDCl_3$, 500 MHz) δ: 2.15 (s, 3H), 2.24 (s, 3H), 5.18 (s, 2H), 6.64 (d, J=7.6 Hz, 1H), 7.01—7.03 (m, 1H), 7.13—7.35 (m, 10H), 7.88 (d, J=8.0 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ: 19.48, 20.24, 46.21, 110.98, 120.41, 122.86, 123.32, 126.07, 126.46, 126.80, 127.98, 130.09, 130.30, 130.81, 131.02, 134.41, 135.26, 135.42, 138.80, 154.27; IR (KBr) v: 3050, 2950, 1600, 1475, 1450, 1390, 1350, 1280, 1160, 840, 685 cm^{-1}. Anal. calcd for $C_{22}H_{20}N_2$: C 84.58, H 6.45, N 8.97; found C 84.41, H 6.42, N 8.82.

1-(4-Chlorobenzyl)-2-(4-chlorophenyl)-1*H*-benzimidazole (**3f**): m.p. 135—137 ℃ (Lit.[19] 136—138 ℃); ^{1}H NMR ($CDCl_3$, 500 MHz) δ: 5.39 (s, 2H), 7.01 (d, J=8.5 Hz, 2H), 7.19 (d, J=8.0 Hz, 1H), 7.25—7.35 (m, 4H), 7.43 (dd, J_1=6.8 Hz, J_2=1.7 Hz, 2H), 7.59 (dd, J_1=6.8 Hz, J_2=1.7 Hz, 2H), 7.86 (d, J=8.0 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ: 48.23, 110.74, 120.58, 123.52, 123.94, 127.69, 128.73, 129.59, 129.82, 130.87, 134.32, 135.04, 136.33, 136.83, 143.42, 153.27.

1-(2-Chlorobenzyl)-2-(2-chlorophenyl)-1*H*-benzimidazole (**3g**): m.p. 161—163 ℃ (Lit.[11] 160—162 ℃); ^{1}H NMR ($CDCl_3$, 500 MHz) δ: 5.36 (s, 2H), 6.64 (d, J=6.9 Hz, 1H), 7.07 (dt, J_1=7.1 Hz, J_2=0.5 Hz, 1H), 7.14—7.22 (m, 2H), 7.25—7.34 (m, 4H), 7.42—7.45 (m, 2H), 7.51 (d, J=8.0 Hz, 1H), 7.89 (d, J=8.0 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ: 46.16, 110.98, 120.76, 123.19, 123.85, 127.40, 127.55, 128.20, 129.43, 130.03, 130.35, 131.88, 132.56, 132.82, 133.68, 134.79, 135.21, 143.37, 151.90.

1-(4-Fluorobenzyl)-2-(4-fluorophenyl)-1*H*-benzimidazole (**3h**):[13] m.p. 82—84 ℃; ^{1}H NMR ($CDCl_3$, 500 MHz) δ: 5.41 (s, 2H), 7.00—7.06 (m, 4H), 7.16 (dt, J_1=7.6 Hz, J_2=2.0 Hz, 2H), 7.22 (d, J=8.0 Hz, 1H), 7.28 (dt, J_1=7.6 Hz, J_2=0.9 Hz, 1H), 7.34 (dt, J_1=7.6 Hz, J_2=0.9 Hz, 1H), 7.63—7.67 (m, 2H), 7.88 (d, J=8.0 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ: 48.21, 110.83, 116.45, 116.52, 116.69, 120.31, 123.61, 123.95, 128.02, 128.08, 131.69, 131.75, 132.15, 136.10, 154.00.

1-(4-Bromobenzyl)-2-(4-bromophenyl)-1*H*-benzimidazole (**3i**):[13] m.p. 154—156 ℃; ^{1}H NMR ($CDCl_3$, 500 MHz) δ: 5.38 (s, 2H), 6.95 (d, J=8.36 Hz, 2H), 7.20 (d, J=8.0 Hz, 1H), 7.28 (t, J=7.8 Hz, 1H), 7.34 (t, J=7.6 Hz, 1H), 7.47 (d, J=8.4 Hz, 2H), 7.52 (d, J=8.1 Hz, 2H), 7.60 (d, J=8.4 Hz, 2H), 7.87 (d, J=8.0 Hz, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ: 48.31, 110.77, 120.55, 123.61, 124.03, 127.99, 131.08, 132.58, 132.79, 135.50, 152.50.

4-{[2-(4-Cyanophenyl)-1*H*-benzimidazol-1-yl]methyl}benzonitrile (**3j**): m.p. 190—192 ℃ (Lit.[20] 190—191 ℃); ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 5.44 (s, 2H), 7.12 (t, J=7.5 Hz, 3H), 7.25 (dt, J_1=7.5 Hz, J_2=1.0 Hz, 1H), 7.32 (dt, J_1=7.5 Hz, J_2=1.3 Hz, 1H), 7.59 (d, J=8.3 Hz, 2H), 7.67—7.69 (m, 4H), 7.83 (d, J=8.1 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 48.16, 110.24, 112.42, 113.89, 118.07, 120.76, 123.71, 124.37, 126.59, 129.28, 129.69, 132.71, 133.19, 134.09, 135.93, 141.07, 143.17, 151.72.

2-(2-Furyl)-1-(2-furylmethyl)-1*H*-benzimidazole (**3k**): m.p. 92—94 ℃ (Lit.[19] 92—95 ℃); ^{1}H NMR ($CDCl_3$, 500 MHz) δ: 5.66 (s, 2H), 6.26 (dd, J_1=16.7 Hz, J_2=1.7 Hz, 2H), 6.62—6.63 (m, 1H), 7.30—7.33 (m, 4H), 7.50—7.52 (m, 1H), 7.65—7.67 (m, 1H), 7.79—7.81 (m, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ: 42.19, 108.92, 110.51, 110.98, 112.62, 114.03, 120.00, 123.62, 123.88, 143.16, 144.63, 149.82.

1,4-Diphenylene-4,4'-(methylene)bis(3-methyl-1-phenyl-1*H*-pyrazol-5-ol) (**4**): m.p. 213—216 ℃; ^{1}H NMR (500 MHz, DMSO-d_6) δ: 2.29 (s, 12H), 5.05 (s, 2H), 7.17 (s, 4H), 7.22 (t, J=7.1 Hz, 4H), 7.41 (t, J=7.8 Hz, 8H), 7.69 (d, J=7.9 Hz, 8H), 12.41 (brs, 2H) 14.11 (s, 2H); ^{13}C NMR (125 MHz, DMSO-d_6) δ: 12.51, 33.69, 121.50, 127.84, 129.72, 140.93, 147.07, 155.01; IR (KBr) v: 3410, 3020, 1590, 1490, 1410, 1350, 1290, 1120, 1020, 850, 745, 690 cm^{-1}. Anal. calcd for $C_{48}H_{42}N_8O_4$: C 72.52, H 5.32, N 14.09; found C 72.35, H 5.29, N 13.85.

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