Synthesis of bis-spiropiperidines using nano-$CuFe_2O_4$@chitosan as a robust and retrievable heterogeneous catalyst

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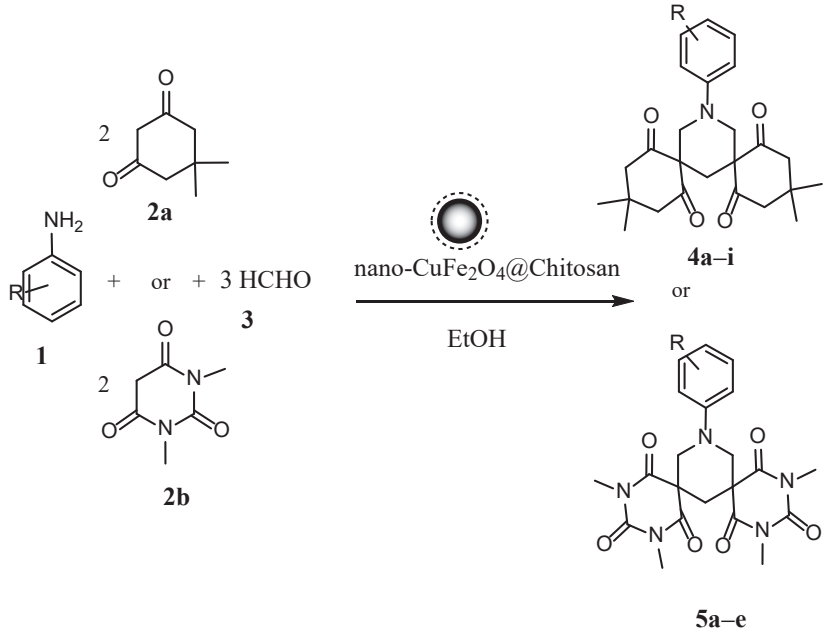
An efficient pseudo six-component synthesis of bis-spiropiperidines is described by one-pot condensation of formaldehyde, aromatic amine and dimedone or *N,N*-dimethyl-barbituric acid using nano-$CuFe_2O_4$@chitosan at room temperature. This method provides several advantages including mild reaction conditions, applicability to wide range of substrates, reusability of the catalyst and little catalyst loading.

Keywords: six-component synthesis, $CuFe_2O_4$@chitosan, bis-spiropiperidines, nanocatalyst, one-pot synthesis

Piperidines and its derivatives are important heterocyclic compounds with wide pharmacological properties, such as antibacterial,[1] antihistaminic,[2] anticonvulsants,[3] anti-HIV,[4] antitumor,[5] acetyl-CoA carboxylase inhibitors,[6] and treatment of influenza virus infection.[7] These activities make piperidines attractive targets in organic synthesis. A number of procedures have been improved for the synthesis of piperidines in the presence of catalysts such as $FeCl_3$,[8] tartaric acid,[9] $ZrCl_4$,[10] acetic acid,[11,12] silica supported tungstic acid (STA),[13] iron(III) trifluoroacetate,[14] cerium supported on chitosan,[15] Dy/chitosan,[16] and silica-supported copper.[17] Despite the availability of these ways, there remains enough choice for a capable and reusable catalyst with high catalytic activity for the preparation of piperidines. The combination of multicomponent reactions with a suitable catalyst could expand their effectiveness from operating cost and conservation points of view. Chitosan is a biopolymer that can be used as a green catalyst in many reactions.[18–20] Presence of both hydroxyl and amino groups in chitosan make it useful as an efficient catalyst. The chitosan can be utilised as a support for the preparation of heterogeneous catalysts.[21–23] Ideally, utilising environmental and green catalysts which can be simply recycled at the end of reactions has obtained significant attention in recent years. Magnetic materials have emerged as a suitable group of heterogeneous catalysts owing to their numerous applications in synthesis and catalysis. To overcome the separation problems of the nano-catalysts, magnetic materials have emerged as recoverable catalysts. The surface of MNPs can be functionalised easily through appropriate surface modifications to enable the loading of a diversity of required functionalities.[24–27] Nano-$CuFe_2O_4$@chitosan was prepared easily by crosslinking method. The $CuFe_2O_4$@chitosan nanoparticles were obtained by crosslinking the amino groups on the chitosan using glutaraldehyde. Herein we report the use of $CuFe_2O_4$@chitosan nanoparticles as an efficient catalyst for the preparation of bis-spiropiperidines by one-pot condensation of formaldehyde, aromatic amine and dimedone or *N,N*-dimethyl-barbituric acid (Scheme 1).

Results and discussion

Initially, we had explored and optimised different reaction parameters for the synthesis of bis-spiropiperidines by the MCR of formaldehyde, 4-chloroaniline and dimedone as a model reaction (Table 1). These reactions were carried out in the presence of various catalysts, such as $(NH_4)_2Ce(NO_3)_6$, $BF_3{\cdot}SiO_2$, $FeCl_3$, chitosan, nano-Fe_3O_4, nano-Fe_3O_4@chitosan, nano-$CuFe_2O_4$ and nano-$CuFe_2O_4$@chitosan. The best results



Scheme 1 Synthesis of bis-spiropiperidines.

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were obtained in ethanol and found that the reaction gave satisfying results in the presence of nano-$CuFe_2O_4$–chitosan. Nano-$CuFe_2O_4$ structure contains Fe^{3+}–O^{2-} in the tetrahedral sites and Cu^{2+}–O^{2-} in the octahedral sites.[24] Therefore, copper plays an important role in the catalytic activity. In order to optimise the reaction conditions, we have performed the reaction using varied quantities of the catalyst. There was no difference in yield and reaction time when catalyst loading was enhanced to 30 mg.

To investigate the scope and limitation of this catalytic process, formaldehyde, aromatic amines and dimedone or *N,N*-dimethyl-barbituric acid were chosen as substrates (Table 2). Investigations of the reaction scope revealed that various aromatic amines (bearing electron-withdrawing and electron-donating groups) can be utilised in this protocol.

Reusability of the catalysts is one of the significant properties of every catalyst used in large scale production. Therefore, the reusability of the heterogeneous catalyst was explored using the model reaction system under the optimised conditions. After completion of the reaction, the magnetic nanocatalyst is easily and efficiently separated from the product by an external magnetic field. The nano-$CuFe_2O_4$–chitosan was washed three to four times with ethanol and dried at room temperature for 5 h. The reusability of the nano-$CuFe_2O_4$–chitosan catalyst was examined and it was found that product yields decreased to a small extent on each reuse (run 1, 88%; run 2, 88%; run 3, 87%; run 4, 87%; run 5, 86%, run 6, 85%).

Table 1 Optimisation of reaction conditions using different catalysts[a]

Entry	Solvent	Catalyst	Time (h)	Yield (%)[b]
1	–	–	20	trace
2	CH_3CN	$(NH_4)_2Ce(NO_3)_6$ (5 mol%)	12	30
3	$CHCl_3$	$BF_3{\cdot}SiO_2$ (0.04 g)	12	37
4	EtOH	$FeCl_3$ (5 mol%)	7	56
5	EtOH	chitosan (0.03 g)	5	60
6	EtOH	nano-$CuFe_2O_4$ (0.04 g)	3	67
7	H_2O/EtOH (5:5)	nano-$CuFe_2O_4$@chitosan (0.02 g)	2	74
8	$CHCl_3$	nano-$CuFe_2O_4$@chitosan (0.02 g)	2	52
9	CH_3CN	nano-$CuFe_2O_4$@chitosan (0.02 g)	2	78
10	EtOH	nano-$CuFe_2O_4$@chitosan (0.01 g)	2	82
11	EtOH	nano-$CuFe_2O_4$@chitosan (0.02 g)	2	88
12	EtOH	nano-$CuFe_2O_4$@chitosan (0.03 g)	2	88
13	EtOH	nano-Fe_3O_4@chitosan (0.02 g)	2	80
14	EtOH	nano-Fe_3O_4	3	61

[a]Formaldehyde (3 mmol), 4-chloroaniline (1 mmol) and dimedone (2 mmol); [b]Isolated yield

Conclusion

In conclusion, we have developed a straightforward method for the synthesis of bis-spiropiperidines by one-pot condensation of formaldehyde, aromatic amine and dimedone or *N,N*-dimethyl-barbituric acid using nano-$CuFe_2O_4$@chitosan at room temperature. The procedure offers several advantages including cleaner reaction profiles, use of easily available, cheap, high yields, shorter reaction time and simple method, reusability of the catalyst and little catalyst loading.

Experimental

The products were isolated and characterised by physical and spectral data. ^{1}H NMR and ^{13}C NMR spectra were recorded on Bruker Avance-400 MHz spectrometers in the presence of tetramethylsilane as internal standard. The IR spectra were recorded on FTIR Magna 550 apparatus using with KBr plates. Melting points were determined on Electro thermal 9200, and are not corrected. The elemental analyses (C, H, N) were obtained from a Carlo ERBA Model EA 1108 analyser. Powder X-ray diffraction (XRD) was carried out on a Philips diffractometer of X'pert Company with monochromatised Cu Kα radiation (λ= 1.5406 Å). Microscopic morphology of products was visualised by SEM (QBSD). The thermogravimetric analysis (TGA) curves are recorded using a V5.1A DUPONT 2000. The magnetic property of magnetite nanoparticle has been measured with a vibrating sample magnetometer (VSM) (Meghnatis Daghigh Kavir Co.; Kashan Kavir; Iran) at room temperature.

Preparation of nano-$CuFe_2O_4$@chitosan

Nano-$CuFe_2O_4$@chitosan was prepared according to the procedure reported in the literature with some modification.[25] For the preparation of nano-$CuFe_2O_4$@chitosan, chitosan (0.5 g) in 100 mL of 2.0 wt% acetic acid solution, 1.5 g oleic acid modified $CuFe_2O_4$ NPs were added into a three-necked flask. The mixture was subjected to ultrasonic wavea for 10 min and stirred for 20 min. Then 2 mL of glutaraldehyde solution (25 wt%) was added into the mixture at 40 ºC and crosslinking reaction was kept for 180 min. After the reaction the composites were gathered through magnetic separation and were washed with deionised water and ethanol several times. The products were dried under vacuum conditions.

Synthesis of bis-spiropiperidines; general procedure

A mixture of formaldehyde (3 mmol), dimedone or *N,N*-dimethyl-barbituric (2 mmol), aniline derivatives (1 mmol) and $CuFe_2O_4$@chitosan nanoparticles (0.02 g) in ethanol (15 mL) was stirred at room

Table 2 Synthesis of bis-spiropiperidines using nano-$CuFe_2O_4$@chitosan[a]

Entry	R	Dicarbonyl compound	Product	Time (min)	Yield (%)[a]	m.p. °C (Found)	Lit. m.p. °C[ref]
1	4-Cl	Dimedone	**4a**	120	88	216–218	216–218[12]
2	4-Br	Dimedone	**4b**	120	88	200–202	200–202[14]
3	4-NO_2	Dimedone	**4c**	120	84	224–226	220–222[14]
4	3-NO_2	Dimedone	**4d**	120	86	187–189	187–189[12]
5	2,3-Cl	Dimedone	**4e**	120	85	250–252	–
6	4-CH_3	Dimedone	**4f**	120	90	198–200	199–201[15]
7	4-F	Dimedone	**4g**	120	88	144–146	142–144[12]
8	3,4-Me	Dimedone	**4h**	120	92	190–192	186–188[17]
9	3,4-Cl	Dimedone	**4i**	120	85	240–242	238–240[12]
10	4-NO_2	*N,N*-Dimethyl-barbituric acid	**5a**	140	80	270–272	–
11	4-Cl	*N,N*-Dimethyl-barbituric acid	**5b**	140	82	230–232	–
12	2-NO_2	*N,N*-Dimethyl-barbituric acid	**5c**	145	81	261–263	–
13	4-Br	*N,N*-Dimethyl-barbituric acid	**5d**	140	83	215–217	–
14	4-CH_3	*N,N*-Dimethyl-barbituric acid	**5e**	140	85	247–249	–

[a]Isolated yield

temperature. The reaction was monitored by TLC. After completion of the reaction, the nanocatalyst was easily separated using an external magnet. The solid product was recrystallised with *n*-hexane/ethyl acetate to get pure product.

15-(4-Chlorophenyl)-3,3,11,11-tetramethyl-15-azadispiro [5.1.5.3] hexadecane-1,5,9,13-tetrone (**4a**): White solid; yield 88%; m.p. 216–218 °C; IR (ν_{max}/cm^{-1}) KBr: 2964, 2926, 2865, 2834, 2795, 1725, 1735, 1706, 1694, 1598, 1496, 1432, 1340, 1249, 825, 674, 516; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.03 (s, 12H, CH_3), 2.51 (s, 2H, CH_2), 2.65 (d, *J* = 12.0 Hz, 4H, $COCH_2$), 2.84 (d, *J* = 12.0 Hz, 4H, $COCH_2$), 3.43 (s, 4H, NCH_2), 7.04 (d, *J* = 8.4 Hz, 2H, ArH), 7.25 (d, *J* = 8.4 Hz, 2H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$) δ 28.4, 28.6, 30.7, 32.3, 51.3, 54.7, 65.6, 120.2, 126.5, 129.1, 150.2, 205.8; Anal. calcd for $C_{25}H_{30}ClNO_4$: C, 67.63; H, 6.81; N, 3.15; found: C, 67.71; H, 6.86; N, 3.23%.

15-(4-Bromophenyl)-3,3,11,11-tetramethyl-15-azadispiro [5.1.5.3] hexadecane-1,5,9,13-tetrone (**4b**): Yellow solid; yield 88%; m.p. 200–202 ºC; IR (ν_{max}/cm^{-1}) KBr: 2956, 1730, 1720, 1705, 1690, 1586, 1492, 1247, 1223, 1149, 1075, 823, 661, 515; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.03 (s, 6H, CH_3), 1.04 (s, 6H, CH_3), 2.52 (s, 2H, CH_2), 2.67 (d, *J* = 13.6 Hz, 4H, $COCH_2$), 2.86 (d, *J* = 13.6 Hz, 4H, $COCH_2$), 3.45 (s, 4H, NCH_2), 7.00 (d, *J* = 7.2 Hz, 2H, ArH), 7.35 (d, *J* = 7.2 Hz, 2H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$) δ 28.3, 28.5, 30.6, 32.2, 51.4, 54.6, 65.5, 120.5, 127.3, 128.2, 151.6, 205.6; Anal. calcd for $C_{25}H_{30}BrNO_4$: C, 61.48; H, 6.19; N, 2.87; found: C, 61.37; H, 6.11; N, 2.75%.

15-(4-Nitrophenyl)-3,3,11,11-tetramethyl-15-azadispiro [5.1.5.3] hexadecane-1,5,9,13-tetrone (**4c**): Yellow solid; yield 84%; m.p. 224–226 °C; IR (ν_{max}/cm^{-1}) KBr: 2955, 2932, 2872, 1728, 1703, 1593, 1495, 1325, 1225, 664, 502; ^{1}H NMR (400 MHz, DMSO-d_6) δ 0.99 (s, 6H, CH_3), 1.01 (s, 6H, CH_3), 2.62 (s, 2H, CH_2), 2.76 (d, *J* = 12.0 Hz, 4H, $COCH_2$), 2.89 (d, *J* = 12.0 Hz, 4H, $COCH_2$), 3.86 (s, 4H, NCH_2), 7.24 (d, *J* = 8.6 Hz, 2H, ArH), 8.06 (d, *J* = 8.6 Hz, 2H, ArH); ^{13}C NMR (100 MHz, DMSO-d_6) δ 27.9, 28.1, 31.2, 32.4, 49.3, 50.7, 64.8, 117.5, 125.8, 135.0, 153.8, 206.7; Anal. calcd for $C_{25}H_{30}N_2O_6$: C, 66.06; H, 6.65; N, 6.16; found: C, 66.14; H, 6.75; N, 6.23%.

15-(3-Nitrophenyl)-3,3,11,11-tetramethyl-15 azadispiro [5.1.5.3] hexadecane-1,5,9,13-tetrone (**4d**): Yellow solid; yield 86%; m.p. 187–189 ºC; IR (ν_{max}/cm^{-1}) KBr: 2955, 2929, 1723, 1703, 1534, 1342, 1202, 873, 785, 675; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.01 (s, 6H, CH_3), 1.03 (s, 6H, CH_3), 2.56 (s, 2H, CH_2), 2.68 (d, *J* = 12.0 Hz, 4H, $COCH_2$), 2.87 (d, *J* = 12.0 Hz, 4H, $COCH_2$), 3.59 (s, 4H, NCH_2), 7.42–7.75 (m, 4H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 28.3, 28.5, 30.7, 32.4, 51.3, 53.5, 65.4, 112.8, 115.6, 124.7, 129.6, 149.2, 151.6, 205.7; Anal. calcd for $C_{25}H_{30}N_2O_6$: C, 66.06; H, 6.65; N, 6.16; found: C, 66.12; H, 6.73; N, 6.23%.

15-(2,3-Dichlorophenyl)-3,3,11,11-tetramethyl-15-azadispiro [5.1.5.3]hexadecane-1,5,9,13-tetrone (**4e**): White solid; yield 85%; m.p. 250–252 ºC; IR (ν_{max}/cm^{-1}) KBr: 2964, 2939, 2931, 1725, 1706, 1692, 1584, 1475, 1256, 1223, 998, 768, 669; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.03 (s, 12H, CH_3), 2.51 (s, 2H, CH_2), 2.66 (d, *J* = 14.0 Hz, 4H, $COCH_2$), 2.85 (d, *J* = 14.0 Hz, 4H, $COCH_2$), 3.44 (s, 4H, NCH_2), 7.02 (d, *J* = 8.2 Hz, 1H, ArH), 7.15 (t, *J* = 8.0 Hz, 1H, ArH), 7.38 (d, *J* = 8.8 Hz, 1H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$) δ 28.3, 28.5, 30.7, 32.2, 51.3, 54.4, 65.5, 118.6, 119.8, 124.5, 130.5, 132.8, 150.7, 205.8; Anal. calcd for $C_{25}H_{29}Cl_2NO_4$: C, 62.76; H, 6.11; N, 2.93; found: C, 62.65; H, 6.21; N, 2.85%.

15-(4-Methylphenyl)-3,3,11,11-tetramethyl-15-azadispiro [5.1.5.3] hexadecane-1,5,9,13-tetrone (**4f**): White solid; yield 90%; m.p. 198–200 ºC; IR (ν_{max}/cm^{-1}) KBr: 2953, 2931, 1720, 1706, 1535, 1343, 675; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.03 (12H, s, CH_3), 2.04 (3H, s, CH_3), 2.43 (2H, s, CH_2), 2.68 (4H, d, *J* = 13.8 Hz, $COCH_2$), 2.87 (4H, d, *J* = 13.8 Hz, $COCH_2$), 3.51 (4H, s, NCH_2), 7.26 (2H, d, *J* = 8.0 Hz, ArH), 7.48 (2H, d, *J* = 8.0 Hz, ArH); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.3, 28.5, 28.6, 30.8, 32.3, 51.6, 55.7, 66.2, 119.5, 129.2, 131.8, 149.4, 206.7; Anal. calcd for $C_{26}H_{33}NO_4$: C, 73.73; H, 7.85; N, 3.31; found: C, 73.78; H, 7.93; N, 3.23%.

15-(4-Nitrophenyl)-2,4,10,12-tetra-N-*methyl-15-azadispiro [5.1.5.3] hexadecane-1,3,5,9,11,13-hexaone* (**5a**): Yellow solid; yield 80%; m.p. 270–272 ºC; IR (ν_{max}/cm^{-1}) KBr: 3021, 2974, 2936, 1691, 1687, 1582, 1373, 1255, 1220, 996, 763, 662; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.60 (s, 2H, CH_2), 2.75 (s, 12H, NCH_3), 3.44 (s, 4H, NCH_2), 7.05 (d, *J* = 8.2 Hz, 2H, ArH), 8.12 (d, *J* = 8.2 Hz, 2H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$) δ 28.4, 31.1, 38.2, 52.2, 117.4, 124.6, 139.2, 151.7, 157.4, 168.3; Anal. calcd for $C_{21}H_{22}N_6O_8$: C, 51.85; H, 4.56; N, 17.28; found: C, 51.73; H, 4.47; N, 17.14%.

15-(4-Chlorophenyl)-2,4,10,12-tetra-N-*methyl-15-azadispiro [5.1.5.3] hexadecane-1,3,5,9,11,13-hexaone* (**5b**): White solid; yield 82%; m.p. 230–232 ºC; IR (ν_{max}/cm^{-1}) KBr: 3025, 2982, 2935, 1690, 1675, 1585, 1473, 1255, 1220, 996, 763, 662; ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.63 (s, 2H, CH_2), 2.74 (s, 12H, NCH_3), 3.42 (s, 4H, NCH_2), 6.72 (d, *J* = 7.8 Hz, 2H, ArH), 7.12 (d, *J* = 7.8 Hz, 2H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 28.4, 31.1, 38.5, 52.6, 116.2, 124.1, 128.6, 147.6, 151.7, 169.5; Anal. calcd for $C_{21}H_{22}ClN_5O_6$: C, 53.00; H, 4.66; N, 14.72; found: C, 52.92; H, 4.57; N, 14.64%.

15-(2-Nitrophenyl)-2,4,10,12-tetra-N-*methyl-15-azadispiro [5.1.5.3] hexadecane-1,3,5,9,11,13-hexaone* (**5c**): Yellow solid; yield 81%; m.p. 261–263 ºC; IR (ν_{max}/cm^{-1}) KBr: 3009, 2971, 2933, 292, 1693, 1685, 1583, 1379, 1252, 1222, 994, 653; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.59 (s, 2H, CH_2), 2.72 (s, 12H, NCH_3), 3.40 (s, 4H, NCH_2), 6.96 (m, 2H, ArH), 7.35 (t, *J* = 8.2 Hz, 1H, ArH), 8.03 (d, *J* = 8.2 Hz, 1H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$) δ 27.8, 30.6, 37.7, 52.1, 117.2, 120.3, 122.3, 132.4, 136.9, 139.2, 150.8, 168.1; Anal. calcd for $C_{21}H_{22}N_6O_8$: C, 51.85; H, 4.56; N, 17.28; found: C, 51.61; H, 4.38; N, 17.19%.

15-(4-Bromophenyl)-2,4,10,12-tetra-N-*methyl-15-azadispiro [5.1.5.3] hexadecane-1,3,5,9,11,13-hexaone* (**5d**): White solid; yield 83%; m.p. 215–217 ºC; IR (ν_{max}/cm^{-1}) KBr: 3012, 2984, 2935, 2933, 1696, 1679, 1583, 1471, 1253, 1222, 997, 766, 632, 553; ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.65 (s, 2H, CH_2), 2.76 (s, 12H, NCH_3), 3.49 (s, 4H, NCH_2), 6.75 (d, *J* = 7.6 Hz, 2H, ArH), 7.26 (d, *J* = 7.6 Hz, 2H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$): δ 28.8, 30.6, 38.6, 53.1, 112.5, 117.8, 132.4, 148.3, 151.8, 168.8; Anal. calcd for $C_{21}H_{22}BrN_5O_6$: C, 48.47; H, 4.26; N, 13.46; found: C, 48.33; H, 4.18; N, 13.38%.

15-(4-Methylphenyl)-2,4,10,12-tetra-N-*methyl-15-azadispiro [5.1.5.3] hexadecane-1,3,5,9,11,13-hexaone* (**5e**): White solid; yield 85%; m.p. 247–249 ºC; IR (ν_{max}/cm^{-1}) KBr: 3022, 2980, 2935, 1696, 1680, 1583, 1471, 1253, 1222, 997, 766, 632, 553; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.25 (s, CH_3), 2.68 (s, 2H, CH_2), 2.79 (s, 12H, NCH_3), 3.51 (s, 4H, NCH_2), 6.55 (d, *J* = 8.2 Hz, 2H, ArH), 7.01 (d, *J* = 8.2 Hz, 2H, ArH); ^{13}C NMR (100 MHz, $CDCl_3$) δ 23.8, 28.1, 31.2, 39.4, 53.5, 115.2, 127.5, 130.2, 147.8, 151.5, 168.2; Anal. calcd for $C_{22}H_{25}N_5O_6$: C, 58.01; H, 5.53; N, 15.38; found: C, 58.08; H, 5.61; N, 15.41%.

Electronic Supplementary Information

Some characteristics of the catalyst are described in the ESI available through:
stl.publisher.ingentaconnect.com/content/stl/jcr/supp-data/content-jcr1704765_esi

Received 12 May 2017; accepted 4 June 2017
Paper 1704765
https://doi.org/10.3184/174751917X14967701767067
Published online: 2 July 2017

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