ACCSCIENCE PUBLISHING

Journal of Energy and Sustainability

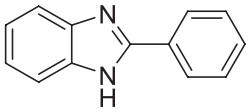
SHORT COMMUNICATION

Ultrasound-assisted one-pot cyclization for the synthesis of 2-substituted benzimidazole derivatives: A rapid access via NaOH/I_2 as an efficient oxidant system

Supplementary Files

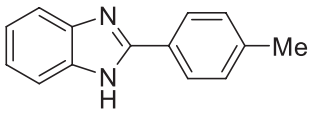
Supplementary File 1. Spectroscopic data of the compounds

2-Phenyl-benzimidazole (Figure 2 and 3a)



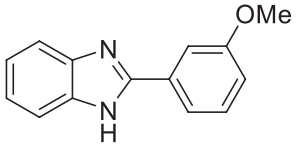
Pale yellow solid; mp: 289–290°C (mp: 288–290°C[1]); IR (KBr)/ν (cm^{-1}): 3446 (NH), 1622 (C=N), 1590, 1445 (C=C, Ar); ^{1}H-NMR (DMSO, 400 MHz; δ ppm): 7.19 (2H, m, Ar), 7.48–7.64 (5H, m, Ar), 8.17 (2H, m, Ar), 12.9 (1H, s, NH); ^{13}C-NMR (DMSO, 100 MHz; δ ppm): 111.1, 118.6, 121.9, 126.2, 128.6, 129.5, 130.0, 134.8, 143.5, 151.0; MS: m/z: 193 ([M-H]$^+$, 100%).

2-(4-Methylphenyl)-benzimidazole (Figure 2 and 3b)



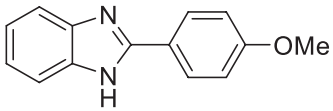
White solid; mp: 260–261°C (mp: 261–263°C[2]); IR (KBr)/ν (cm^{-1}): 3429 (NH), 1620 (C=N), 1587, 1433 (C=C, Ar); ^{1}H-NMR (DMSO, 400 MHz; δ ppm): 2.36 (3, s, Me) 7.18 (2H, m, Ar), 7.34 (2H, d, Ar, J = 8.0 Hz), 7.57 (2H, m, Ar), 8.06 (2H, d, Ar, J = 8.0 Hz), 12.8 (1H, s, NH); ^{13}C-NMR (DMSO, 100 MHz; δ ppm): 21.4, 115.5, 122.4, 125.9, 127.9, 130, 139, 140.0, 151.9.

2-(3-Methoxyphenyl)-benzimidazole (Figure 2 and 3c)



Yellow solid; mp: 200–202°C (mp: 205–206°C[3]); IR (KBr)/ν (cm^{-1}): 3437 (NH), 1596 (C=N), 1541, 1464 (C=C, Ar); ^{1}H-NMR (DMSO, 400 MHz; δ ppm): 3.9 (3H, s, Me), 7.05 (1H, m, Ar), 7.19 (2H, m, Ar), 7.45 (1H, s, Ar), 7.59 (2H, m Ar), 7.74 (2H, m, Ar), 12.9 (1H, s, NH); ^{13}C-NMR (DMSO, 100 MHz; δ ppm): 56.0, 114.0, 115.2, 116.5, 117.8, 124.5, 130.5, 132.6, 139, 152.2, 159.1.

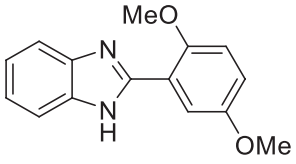
2-(4-Methoxyphenyl)-benzimidazole (Figure 2 and 3d)



Yellow solid; mp: 229–230°C, (mp: 229– 231°C[4]); IR (KBr)/ν (cm^{-1}): 3344 (NH), 1608 (C=N), 1506, 1461 (C=C, Ar); ^{1}H-NMR (DMSO, 400 MHz; δ ppm): 3.89 (3H, s, Me), 7.2 (2H, d, Ar, J = 8.0 Hz), 7.52 (2H, m, Ar), 7.79 (2H, m, Ar),

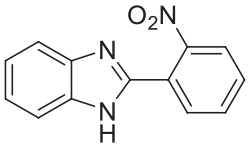
8.38 (2H, d, Ar, J = 8.0 Hz), 15.3 (1H, s, NH); ^{13}C-NMR (DMSO, 100 MHz; δ ppm): 56.2, 114.1, 115.3, 115.5, 126.0, 130.6, 132, 148.9, 163.6.

2-(2,5-Dimethoxyphenyl)-benzimidazole (Figure 2 and 3e)



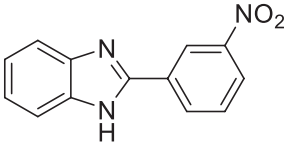
Yellow solid; mp: 197–198°C, (mp: 197–198°C[5]); IR (KBr)/v (cm^{-1}): 3408 (NH), 1622 (C=N), 1511, 1458 (C=C, Ar); ^{1}H-NMR (DMSO, 400 MHz; δ ppm): 4.02 (3H, s, Me), 3.85 (3H, s, Me) 7.33 (2H, d, Ar, J = 6.0 Hz), 7.55 (2H, m, Ar), 7.89 (2H, m, Ar), 8.02 (1H, s, Ar), 15 (1H, s, NH); ^{13}C-NMR (DMSO, 100 MHz; δ ppm): 56.7, 57.0, 11.2, 114.4, 114.6, 114.6, 122.2, 126.3, 131.7, 146.1, 152.6, 153.8.

2-(2-Nitrophenyl)-benzimidazole (Figure 2 and 3f)



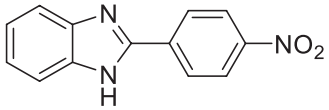
Pale yellow solid; mp: 212–214°C (mp: 209–210°C[6]); IR (KBr)/v (cm^{-1}): 3428 (NH), 1622 (C=N), 1530, 1458 (C=C, Ar); ^{1}H-NMR (DMSO, 400 MHz; δ ppm): 7.54–7.57 (2H, m, Ar), 7.85–7.87 (2H, m, Ar), 7.99 (1H, t, Ar), 8.05 (1H, m, Ar), 8.12 (1H, d, Ar, J = 7.6 Hz), 8.37 (1H, d, Ar, J = 8.0 Hz), 13 (1H, s, NH); ^{13}C-NMR (DMSO, 100 MHz; δ ppm): 114.5, 121.7, 127.9, 128.7, 130.5, 137.2, 140.9, 152.8; MS: m/z: 239 (M$^+$).

2-(3-Nitrophenyl)-benzimidazole (Figure 2 and 3g)



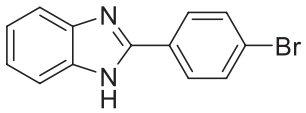
Yellow solid; mp: 309–310°C, (mp: 310–311°C[1]); IR (KBr)/v (cm^{-1}): 3308 (NH), 1630 (C=N), 1531, 1482 (C=C, Ar); ^{1}H-NMR (DMSO, 400MHz; δ ppm): 7.47 (2H, m, Ar), 7.79 (2H, m, Ar), 7.92–7.95 (1H, m, Ar), 8.1–8.9 (2H, m, Ar), 9.1 (1H, s, Ar), 10.1 (1H, s, NH); ^{13}C-NMR (DMSO, 100 MHz; δ ppm): 113.3, 121.2, 122.7, 125.2, 130.3, 132.1, 133.6, 139, 147.5, 152.7; MS: m/z: 238 ([M-H]$^+$, 100%).

2-(4-Nitrophenyl)-benzimidazole (Figure 2 and 3h)



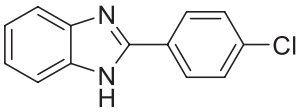
Pale yellow solid; mp: 307–309°C (mp: 312–314°C[6]); IR (KBr)/v (cm^{-1}): 3421 (NH), 1606 (C = N), 1524, 1451 (C = C, Ar); ^{1}H-NMR (DMSO, 400 MHz; δ ppm): 7.51–7.54 (2H, m, Ar), 7.83 (2H, m, Ar), 8.47 (2H, d, Ar, J = 8.4 Hz), 8.48–8.64 (2H, d, Ar, J = 8.4 Hz), 8.4 (1H, s, NH); ^{13}C-NMR (DMSO, 100 MHz; δ ppm): 114.5, 121.7, 127.9, 128.7, 130.5, 137.2, 140.9, 152.8; MS: m/z: 239 (M$^+$).

2-(4-Bromophenyl)-benzimidazole (Figure 2 and 3i)



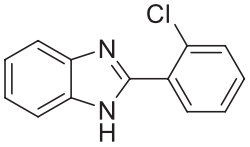
Pale yellow solid; mp: 292–293°C (mp: 250–252°C[3]); IR (KBr)/v (cm^{-1}): 3449 (NH), 1628 (C=N), 1598, 1456 (C=C, Ar); ^{1}H-NMR (DMSO, 400 MHz; δ ppm): 7.53 (2H, m, Ar), 7.83 (2H, m, Ar), 7.95 (2H, d, Ar, J = 4.8 Hz), 8.28 (2H, d, Ar, J = 4.8 Hz); ^{13}C-NMR (DMSO, 100 MHz; δ ppm): 102.5, 121.5, 141.6, 127, 128.1, 130.4, 138.2, 150.2.

2-(4-Chlorophenyl)-benzimidazole (Figure 2 and 3j)



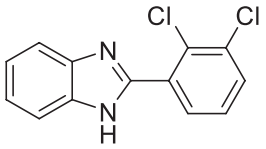
Yellow solid; mp: 290–291°C, (mp: 291–293°C[7]); IR (KBr)/v (cm^{-1}): 3442 (NH), 1598 (C=N), 1580, 1429 (C=C, Ar); ^{1}H-NMR (DMSO, 400 MHz; δ ppm): 7.2 (2H, m, Ar), 7.50–7.89 (6H, m, Ar), 13 (1H, s, NH); ^{13}C-NMR (DMSO, 100 MHz; δ ppm): 113.5, 123.7, 127.6, 128.3, 129.4, 133.4, 138.9, 151.8; MS: m/z: 229.0 (M+H)$^+$.

2-(2-Chlorophenyl)-benzimidazole (Figure 2 and 3k)



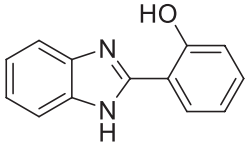
Yellow solid; mp: 231–233°C (mp: 232–234°C[2]); IR (KBr)/v (cm^{-1}): 3444 (NH), 1591 (C=N), 1575, 1440 (C=C, Ar); ^{1}H-NMR (DMSO, 400 MHz; δ ppm): 7.2 (2H, m, Ar), 7.50–7.89 (6H, m, Ar), 12.9 (1H, s, NH); ^{13}C-NMR (DMSO, 100 MHz; δ ppm): 116.0, 122.7, 127.9, 130.4, 130.8, 131.6, 132.2, 132.5, 139.5, 149.6; MS: m/z: 228.5 (M$^+$).

2-(2,3-Dichlorophenyl)-benzimidazole (Figure 2 and 3l)



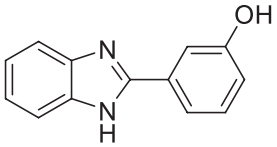
Yellow solid; mp: 224–226°C, (mp: 224–226°C[5]); IR (KBr)/ν (cm^{-1}): 3095 (NH), 1624 (C=N), 1540, 1433 (C=C, Ar); ^{1}H-NMR (DMSO, 400 MHz; δ ppm): 7.2 (2H, m, Ar), 7.53 (1H, m, Ar), 7.64 (2H, m, Ar), 7.82 (2H, m, Ar), 12.8 (1H, s, NH); ^{13}C-NMR (DMSO, 100 MHz; δ ppm): 115.9, 122.9, 128.9, 130.5, 131.3, 132.1, 132.9, 133.2, 139.3, 149.1.

2-(2-Hydroxyphenyl)-benzimidazole (Figure 2 and 3m)



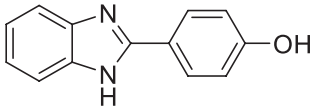
White solid; mp: 238–240°C (mp: 238–240°C[7]); IR (KBr)/ν (cm^{-1}): 3325 (OH), 3055 (NH), 1593 (C=N), 1530, 1491 (C=C, Ar); ^{1}H-NMR (DMSO, 400 MHz; δ ppm): 6.99–7.05 (2H, m, Ar), 7.27 (2H, m, Ar), 7.37 (1H, t, Ar), 7.66 (2H, m, Ar), 8.06 (1H, m, Ar), 13.19 (1H, s, OH), 13.19 (1H, s, NH); ^{13}C-NMR (DMSO, 100 MHz; δ ppm): 112.1, 113.1, 117.7, 119.6, 123.3, 126.7, 132.2, 141.2, 152.2, 158.6; MS: m/z: 210 (M).

2-(3-Hydroxyphenyl)-benzimidazole (Figure 2 and 3n)



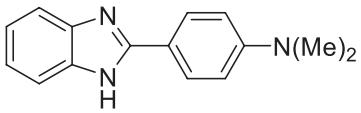
Yellow solid; mp: 245–247°C, (mp: 245–247°C[5]); IR (KBr)/ν (cm^{-1}): 3434 (OH), 3243 (NH), 1588 (C=N), 1541, 1445 (C=C, Ar); ^{1}H-NMR (DMSO, 400 MHz; δ ppm): 6.9 (1H, d, Ar, J = 5.6 Hz), 7.18 (2H, m, Ar), 7.33 (1H, t, Ar), 7.59 (4H, d, Ar, J = 6.6 Hz), 12.9 (1H, s, NH); ^{13}C-NMR (DMSO, 100 MHz; δ ppm): 113.9, 115.6, 117.6, 117.8, 122.6, 130.5, 131.9, 139.9, 151.9, 158.3.

2-(4-Hydroxyphenyl)-benzimidazole (Figure 2 and 3o)



White solid; mp: 254–255°C (mp: 254–255°C[4]); IR (KBr)/ν (cm^{-1}): 3383 (OH), 3202 (NH), 1668 (C=N), 1600, 1457 (C=C, Ar); ^{1}H-NMR (DMSO, 400 MHz; δ ppm): 6.91–7.50 (4H, m, Ar), 7.73–8.21 (4H, m, Ar), 9.7 (1H, s, OH), 15.2 (1H, s, NH); ^{13}C-NMR (DMSO, 100 MHz; δ ppm): 113.0, 114.0, 116.9, 126.0, 131.6, 132, 149.9, 160.7.

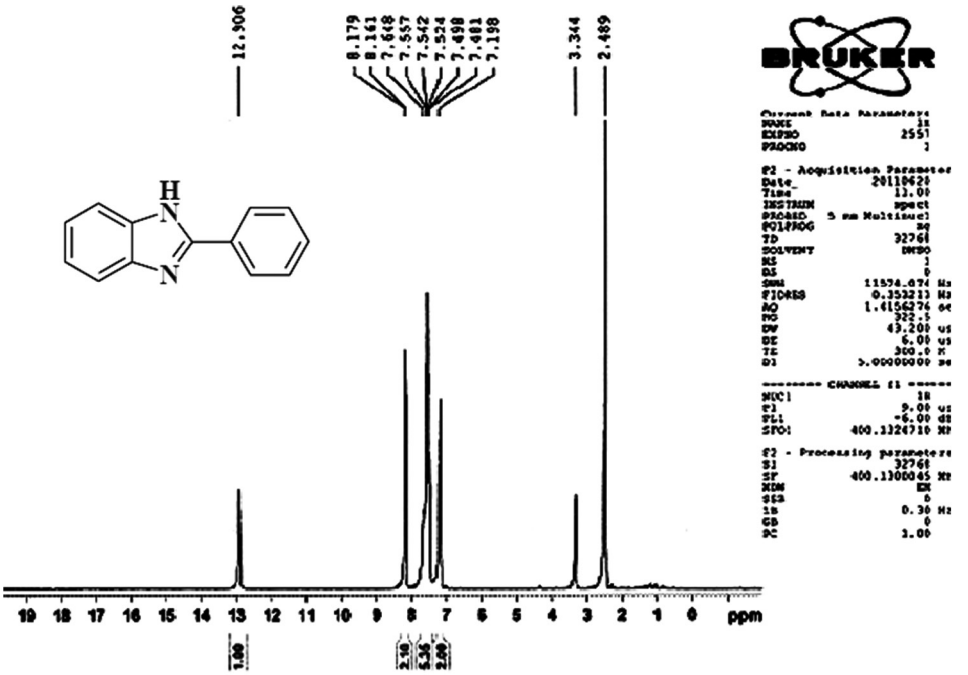
2-(N,N-dimethylphenyl)-benzimidazole (Figure 2 and 3p)



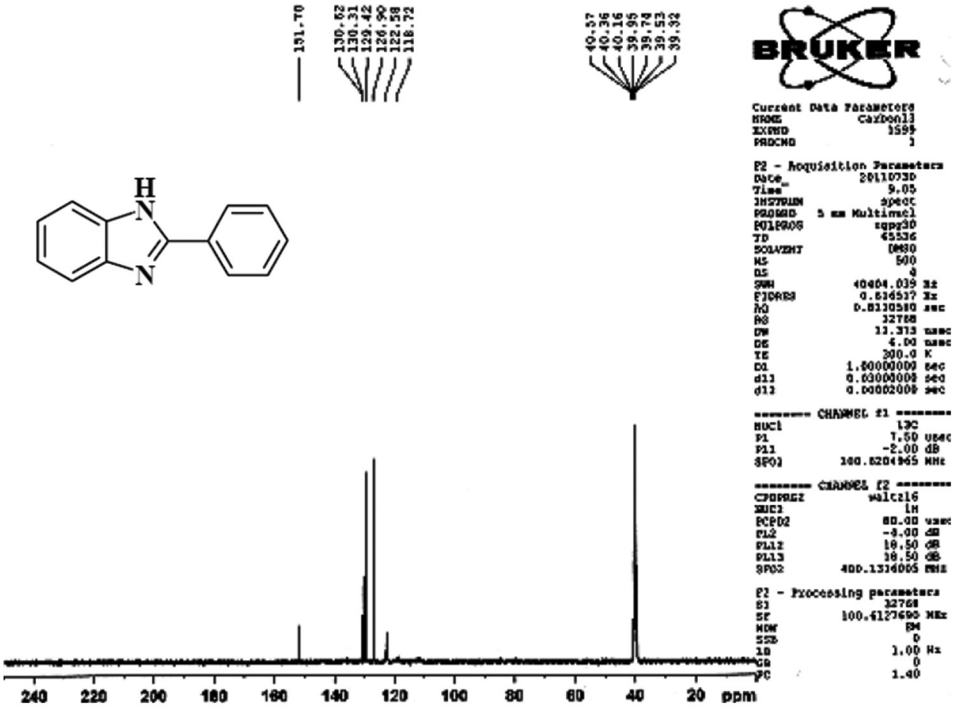
Yellow solid; mp: 293–295°C (mp: 293–295°C[4]); IR (KBr)/ν (cm^{-1}): 3391 (NH), 1605 (C=N), 1518, 1459 (C=C, Ar); ^{1}H-NMR (DMSO, 400 MHz; δ ppm): 6.84 (2H, d, Ar, J = 7.8 Hz), 7.43 (2H, m, Ar), 7.70 (2H, m, Ar), 8.21 (2H, d, Ar, J = 7.8 Hz), 15,2 (1H, s, NH); ^{13}C-NMR (DMSO, 100 MHz; δ ppm): 39.5, 107.8, 111.8, 113.2, 125.1, 129.1, 131.5, 149.8, 153.2.

Abbreviations: DMSO: Dimethyl sulfoxide; IR: Infrared; mp: Melting point; MS: Mass spectrometry; NMR: Nuclear magnetic resonance

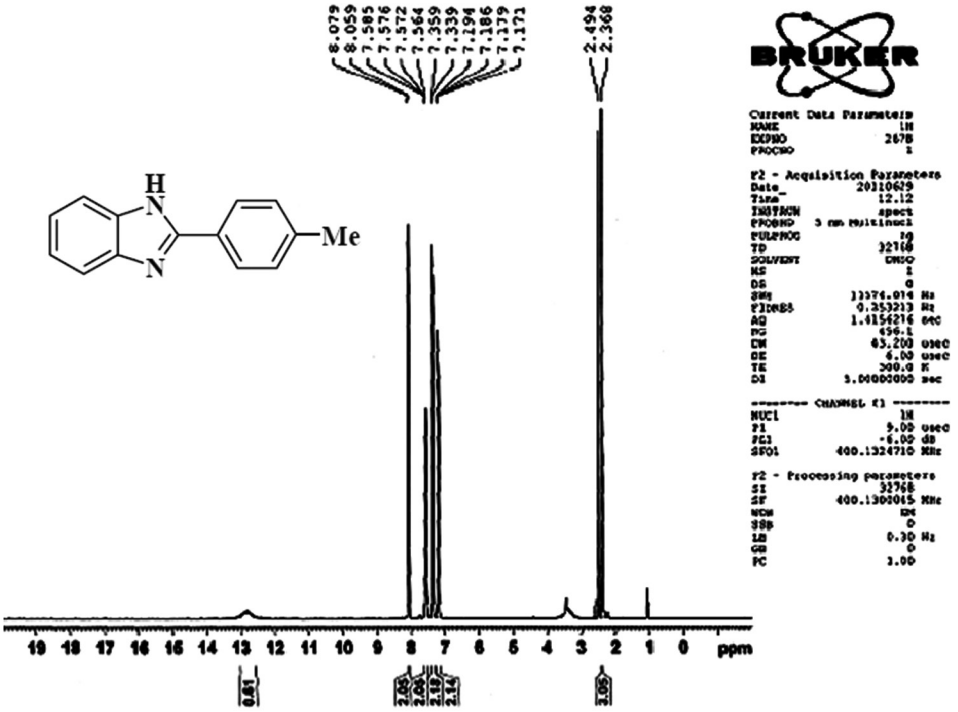
Supplementary File 2. ^{1}H-NMR and ^{13}C-NMR spectra of the products



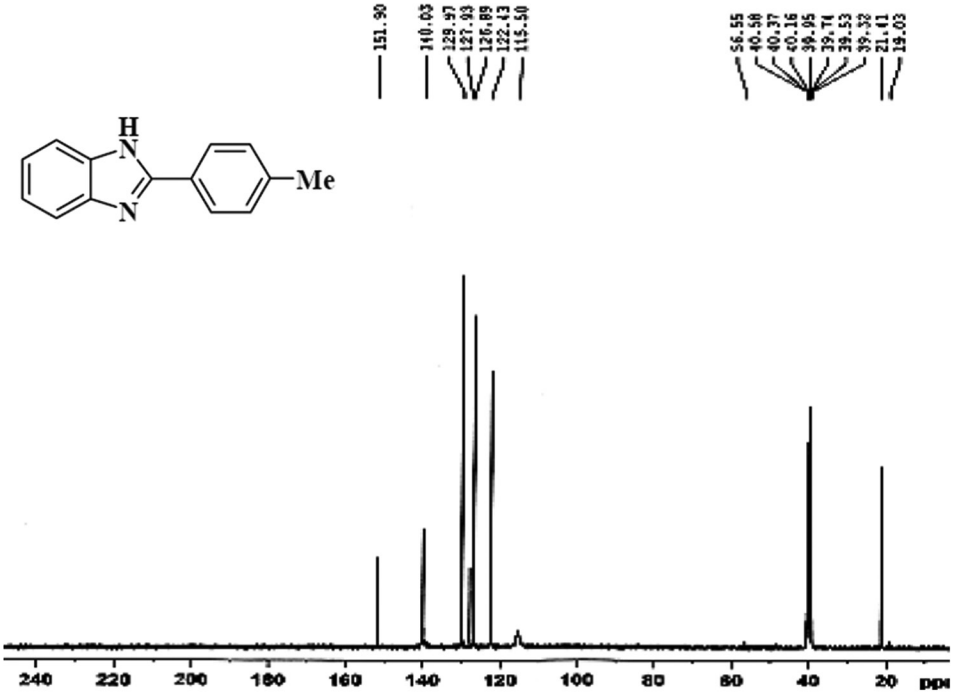
2-Phenyl-benzimidazole (Figure 2 and 3a); ^{1}H-NMR (400 MHz, DMSO-d_6)



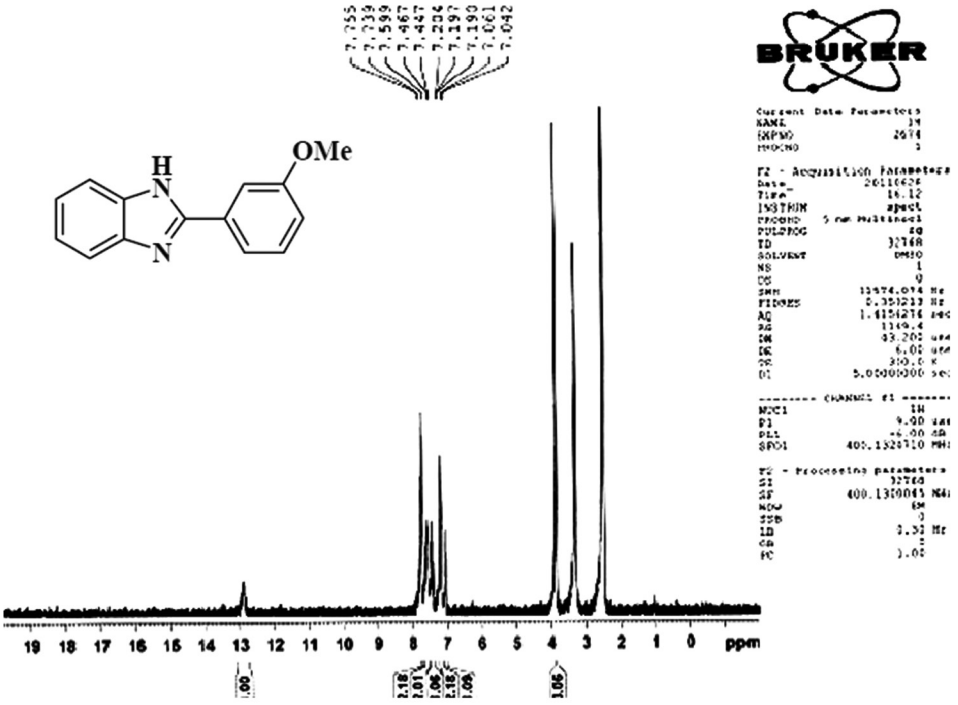
2-Phenyl-benzimidazole (Figure 2 and 3a); ^{13}C-NMR (100 MHz, DMSO-d_6)



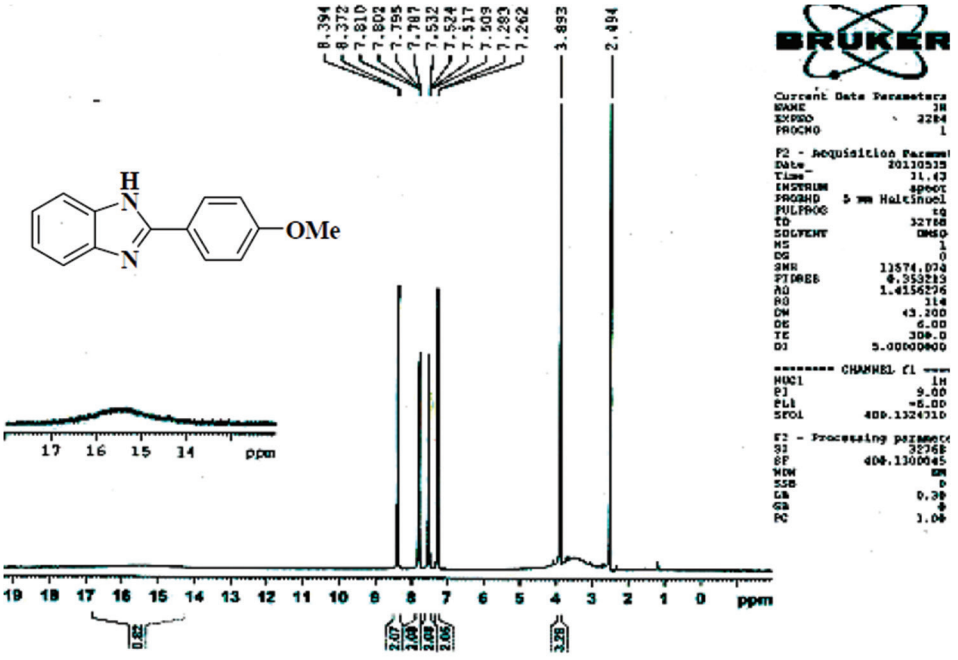
2-(4-Methylphenyl)-benzimidazole (Figure 2 and 3b); ^{1}H-NMR (400 MHz, DMSO-d_6)



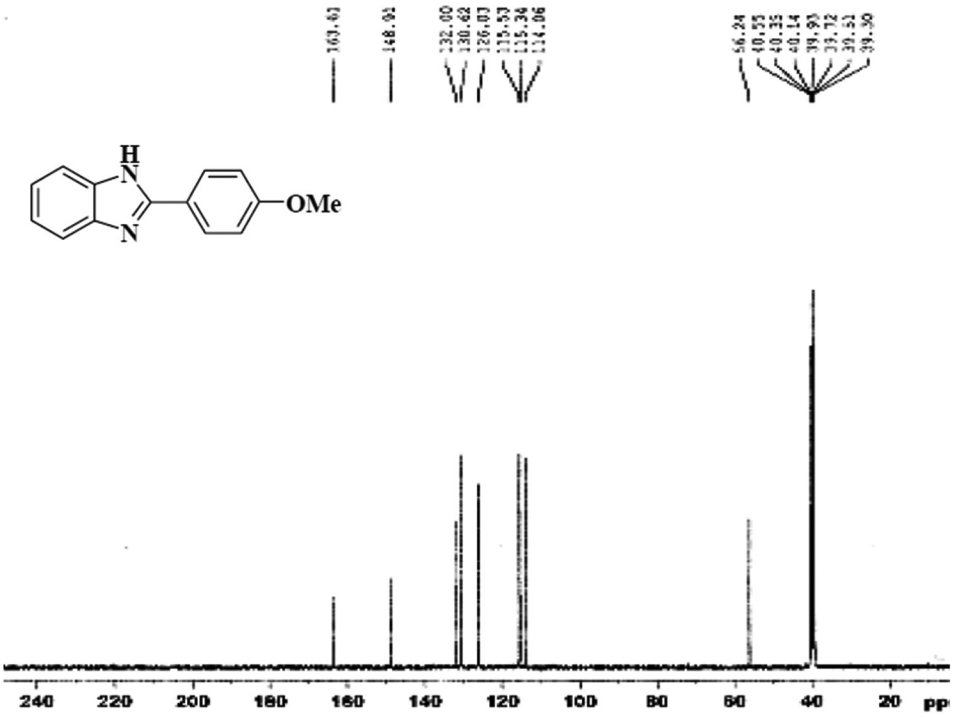
2-(4-Methylphenyl)-benzimidazole (Figure 2 and 3b); ^{13}C-NMR (100 MHz, DMSO-d_6)



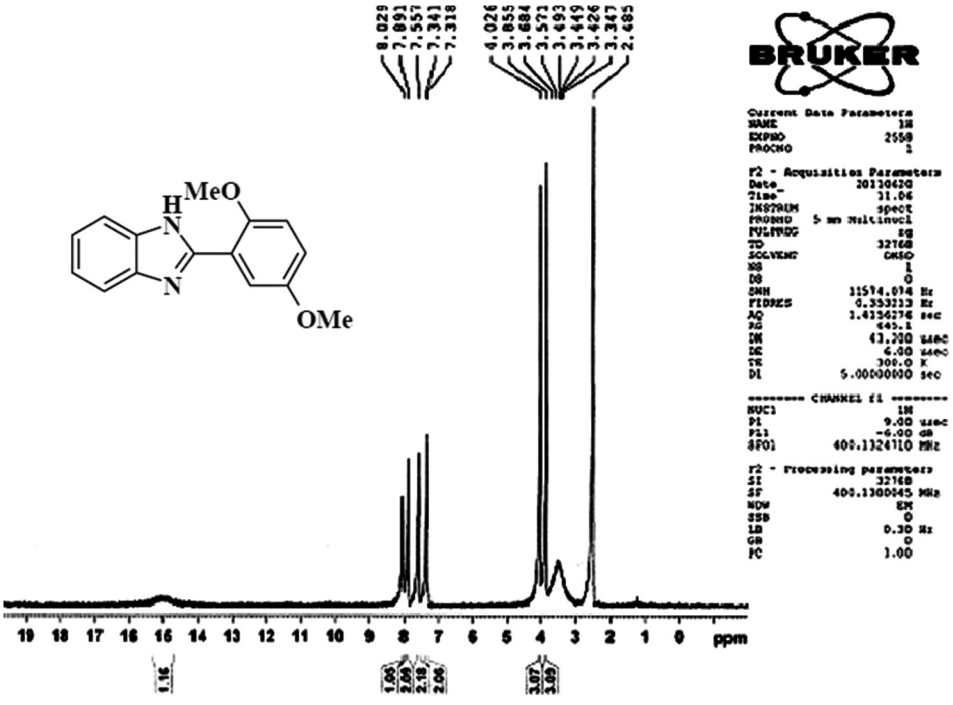
2-(3-Methoxyphenyl)-benzimidazole (Figure 2 and 3c); ^{1}H-NMR (400 MHz, DMSO-d_6)



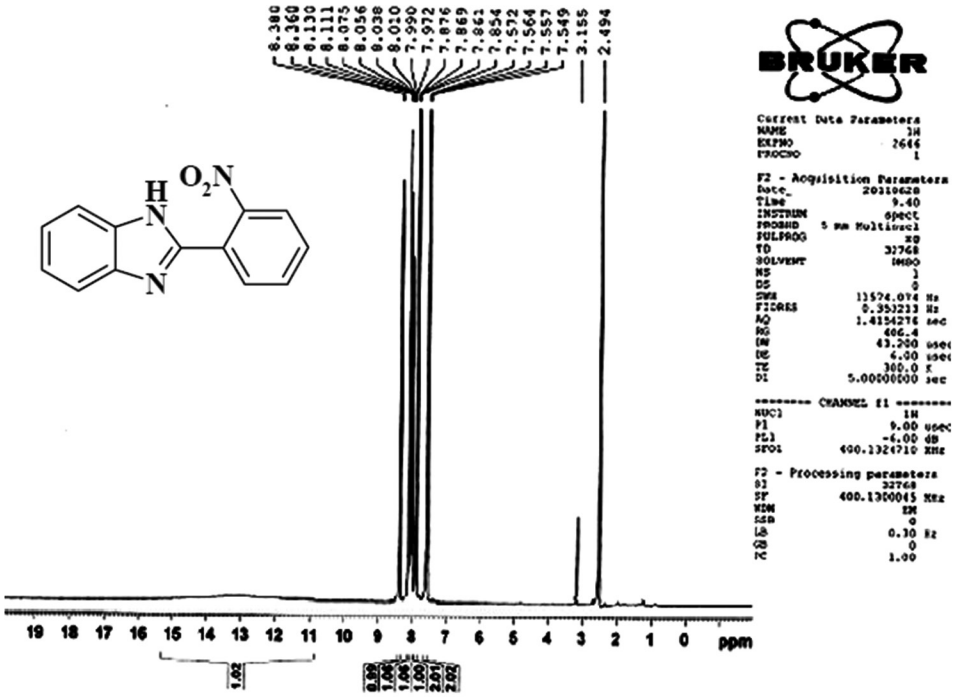
2-(4-Methoxyphenyl)-benzimidazole (Figure 2 and 3d); ^{1}H-NMR (400 MHz, DMSO-d_6)



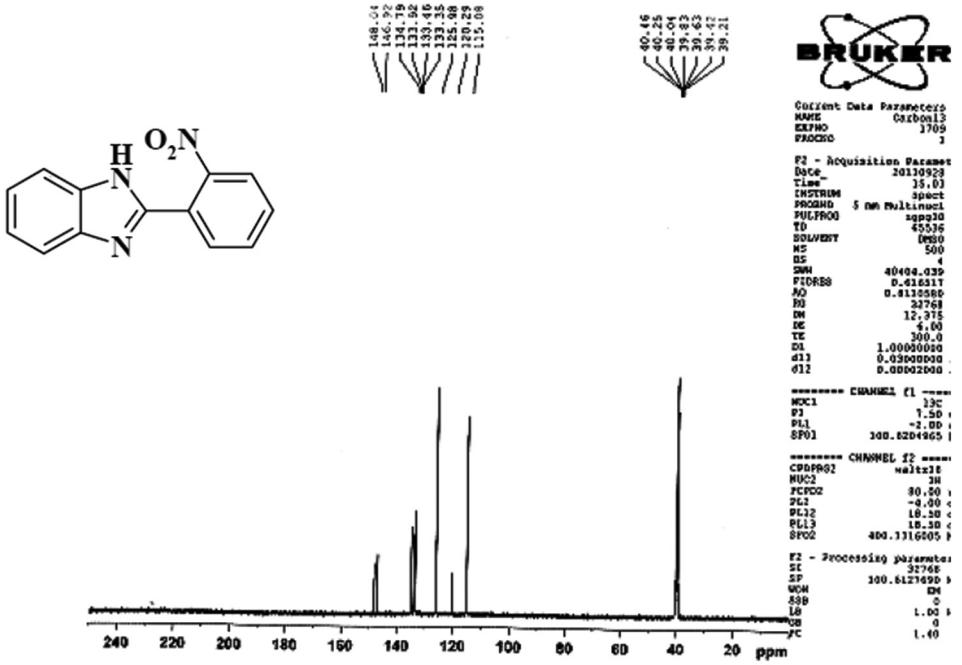
2-(4-Methoxyphenyl)-benzimidazole (Figure 2 and 3d); ^{13}C-NMR (100 MHz, DMSO-d_6)



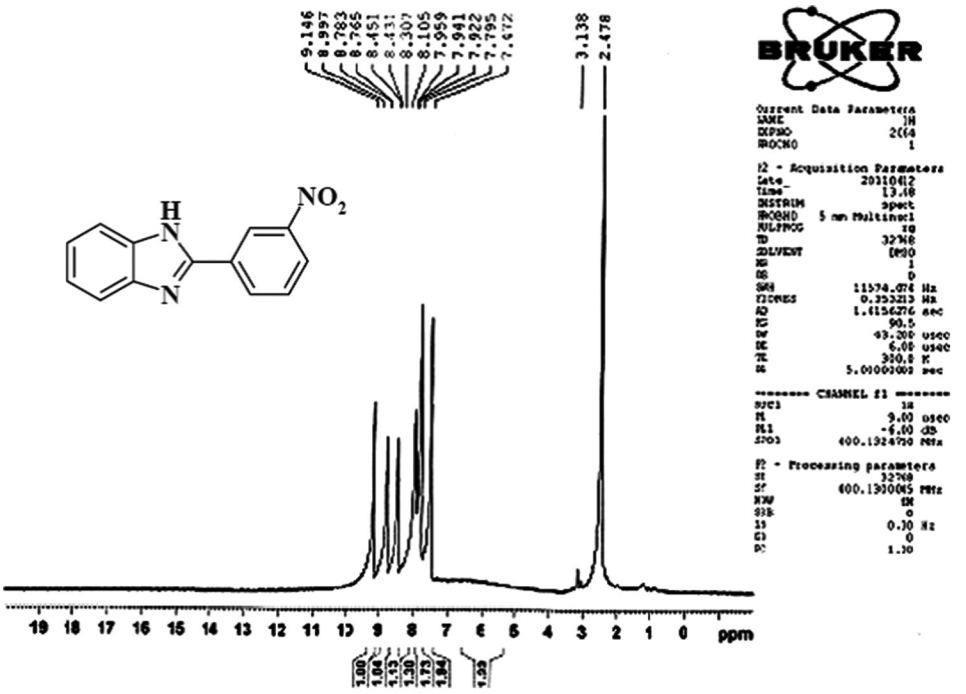
2-(2,5-Dimethoxyphenyl)-benzimidazole (Figure 2 and 3e); ^{1}H-NMR (400 MHz, DMSO-d_6)



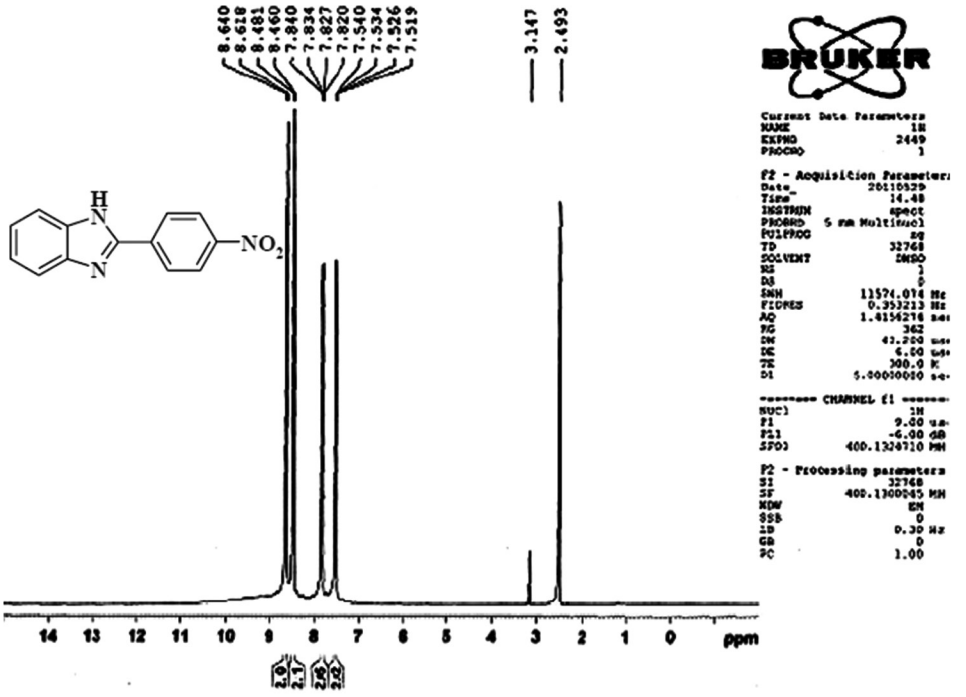
2-(2-Nitrophenyl)-benzimidazole (Figure 2 and 3f); ^{1}H-NMR (400 MHz, DMSO-d_6)



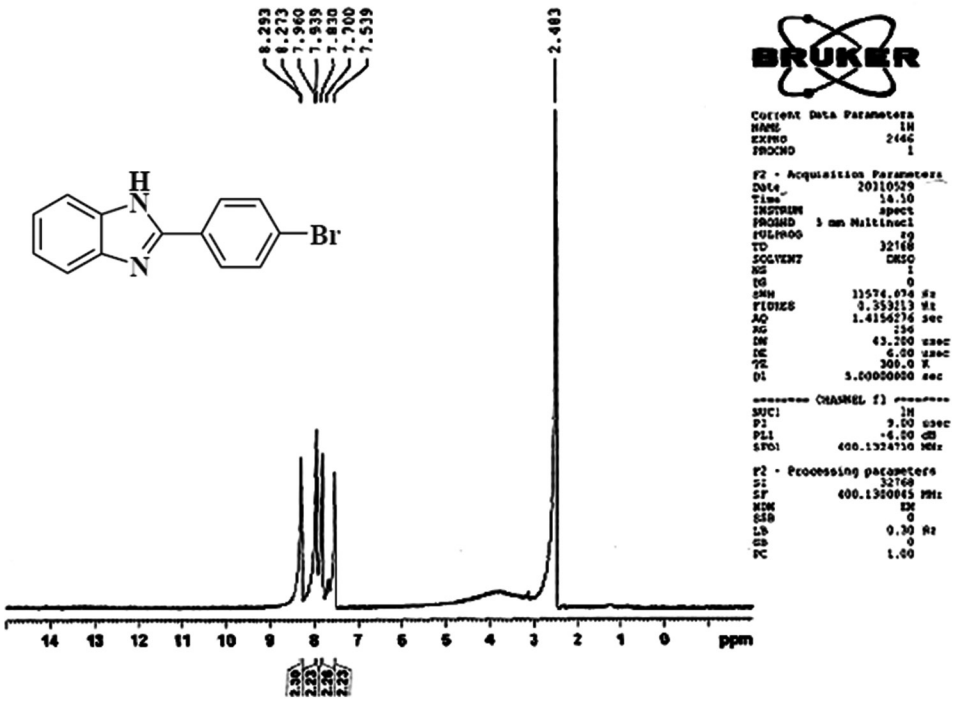
2-(2-Nitrophenyl)-benzimidazole (Figure 2 and 3f); ^{13}C-NMR (100 MHz, DMSO-d_6)



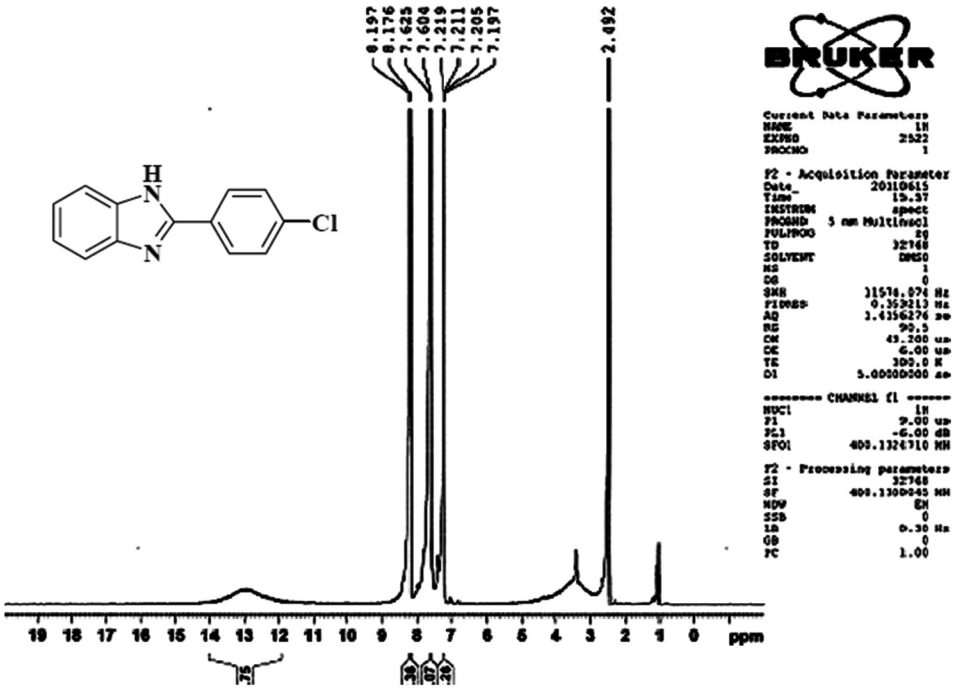
2-(3-Nitrophenyl)-benzimidazole (Figure 2 and 3g); ^{1}H-NMR (400 MHz, DMSO-d_6)



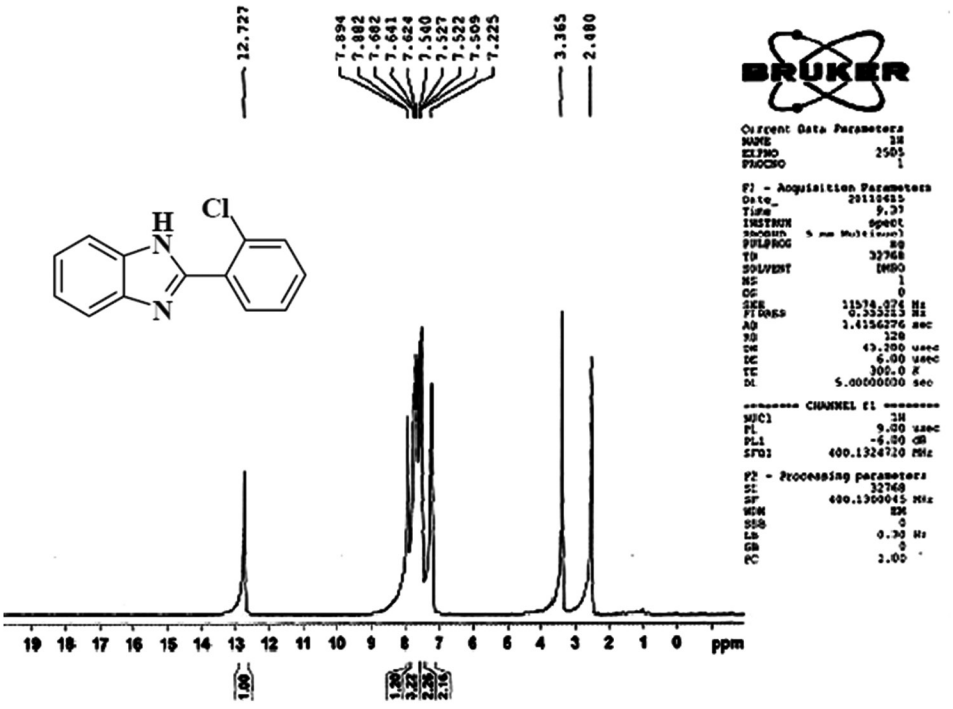
2-(4-Nitrophenyl)-benzimidazole (Figure 2 and 3h); ^{1}H-NMR (400 MHz, DMSO-d_6)



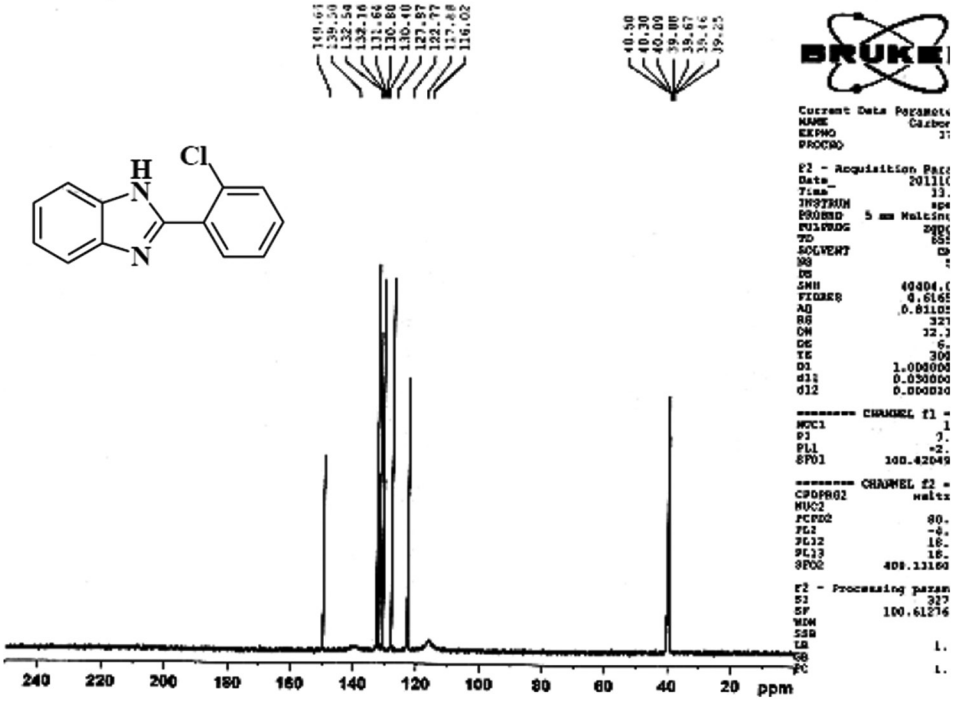
2-(4-Bromophenyl)-benzimidazole (Figure 2 and 3i); ^{1}H-NMR (400 MHz, DMSO-d_6)



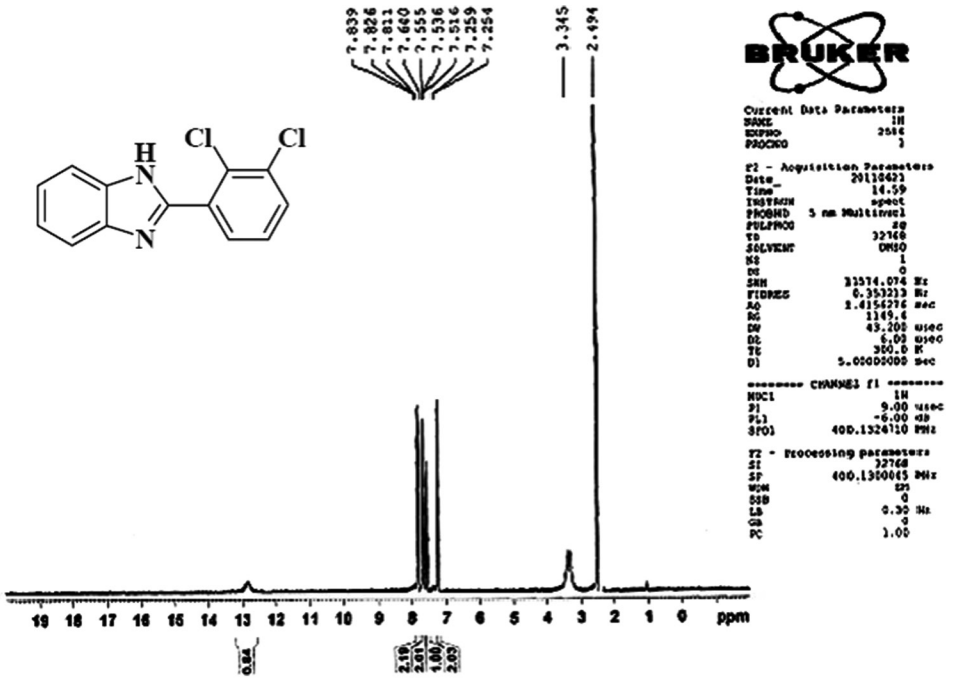
2-(4-Chlorophenyl)-benzimidazole (Figure 2 and 3j); ^{1}H-NMR (400 MHz, DMSO-d_6)



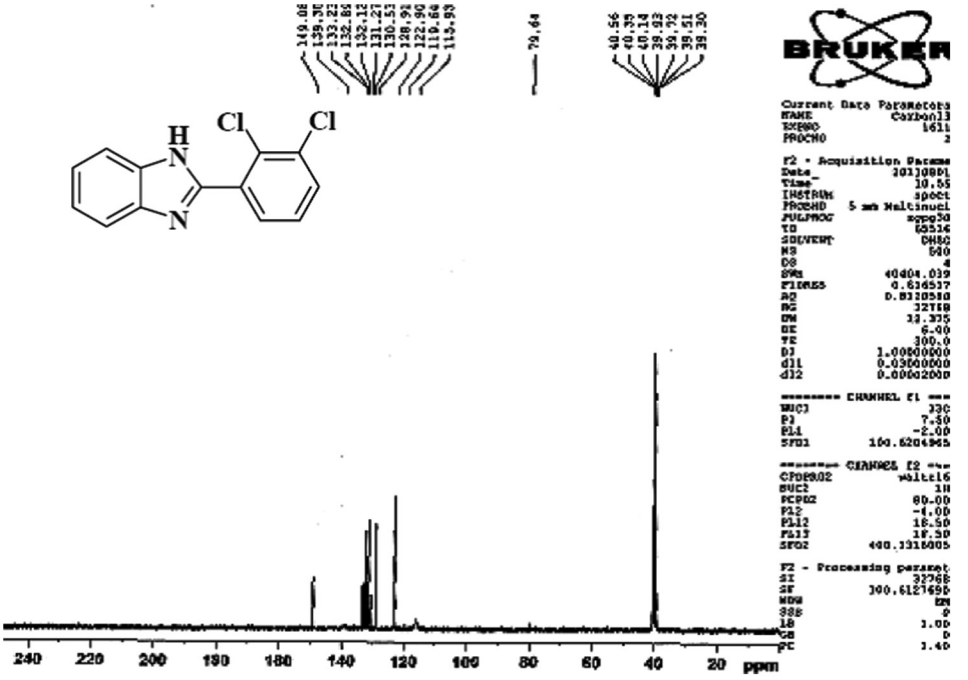
2-(2-Chlorophenyl)-benzimidazole (Figure 2 and 3k); ^{1}H-NMR (400 MHz, DMSO-d_6)



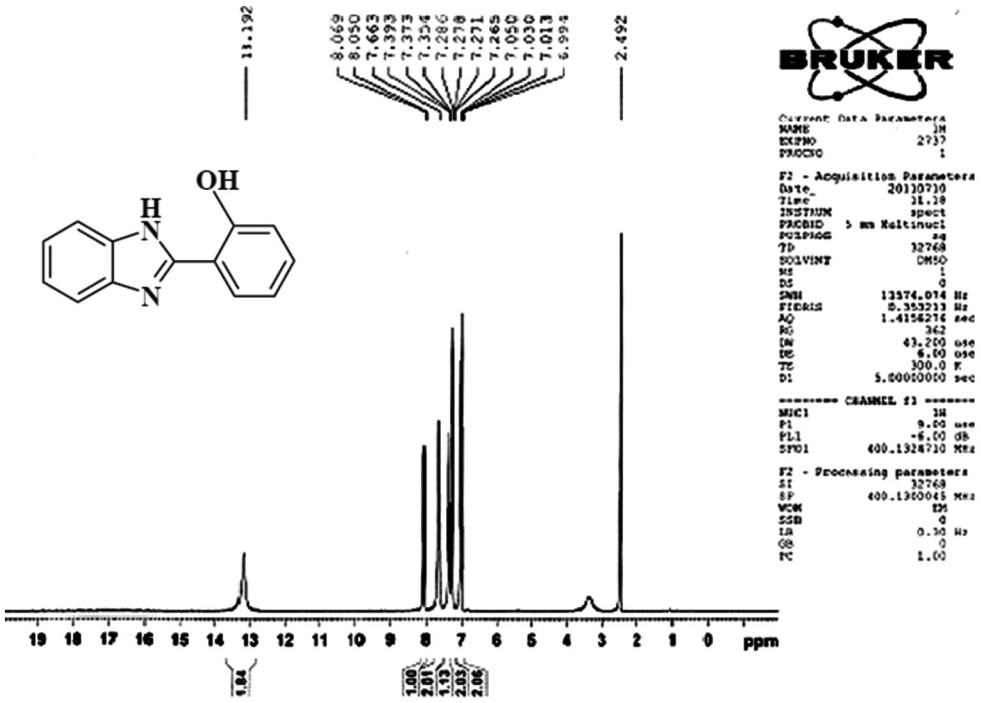
2-(2-Chlorophenyl)-benzimidazole (Figure 2 and 3k); ^{13}C-NMR (100 MHz, DMSO-d_6)



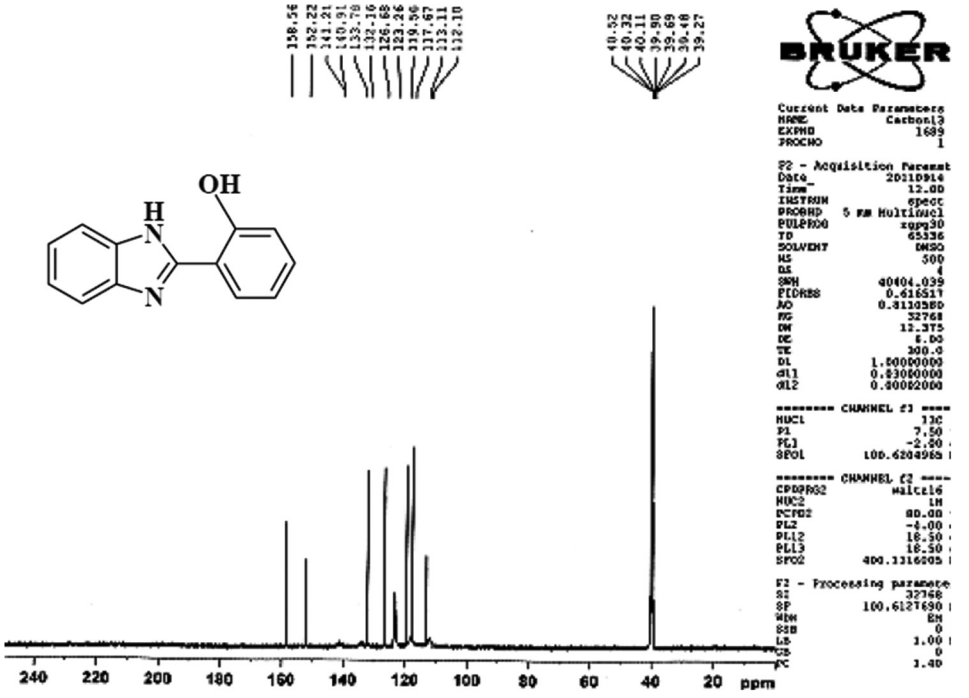
2-(2,3-Dichlorophenyl)-benzimidazole (Figure 2 and 3l); ^{1}H-NMR (400 MHz, DMSO-d_6)



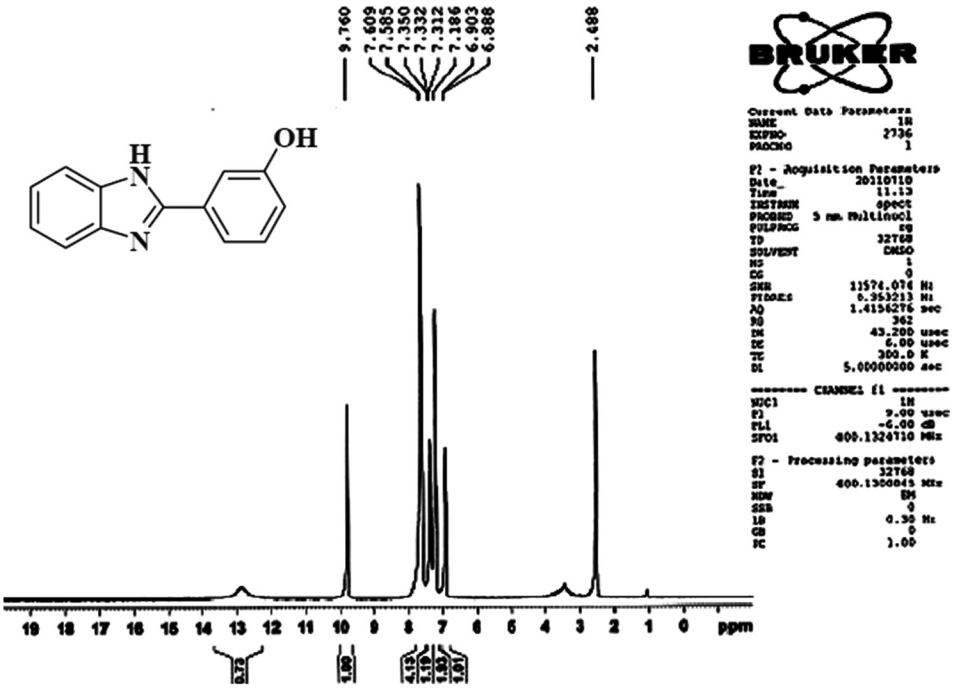
2-(2,3-Dichlorophenyl)-benzimidazole (Figure 2 and 3l); ^{13}C-NMR (100 MHz, DMSO-d_6)



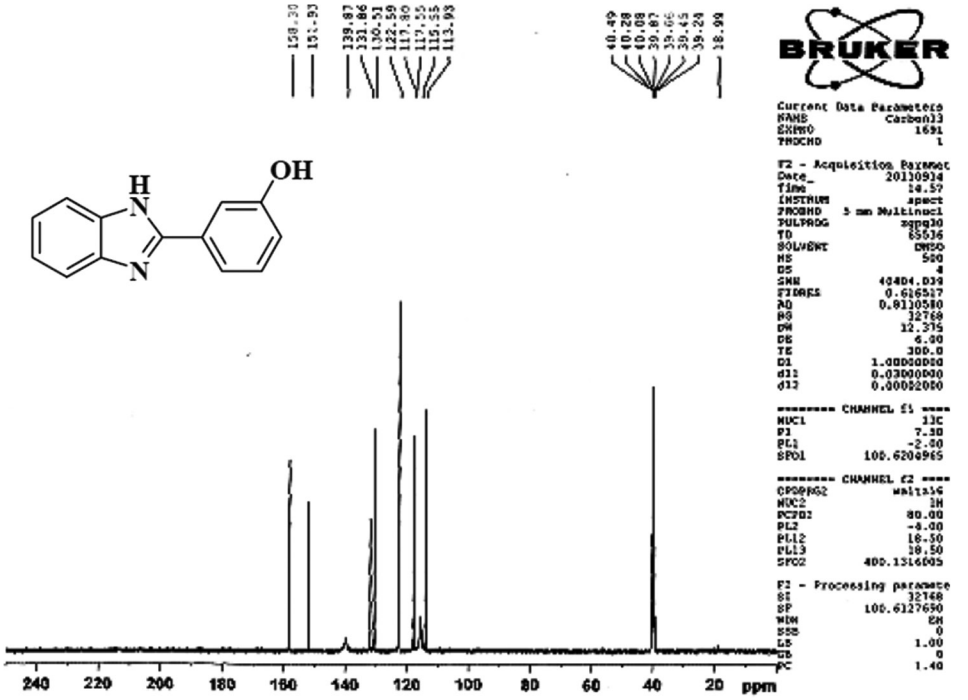
2-(2-Hydroxyphenyl)-benzimidazole (Figure 2 and 3m); ^{1}H-NMR (400 MHz, DMSO-d_6)



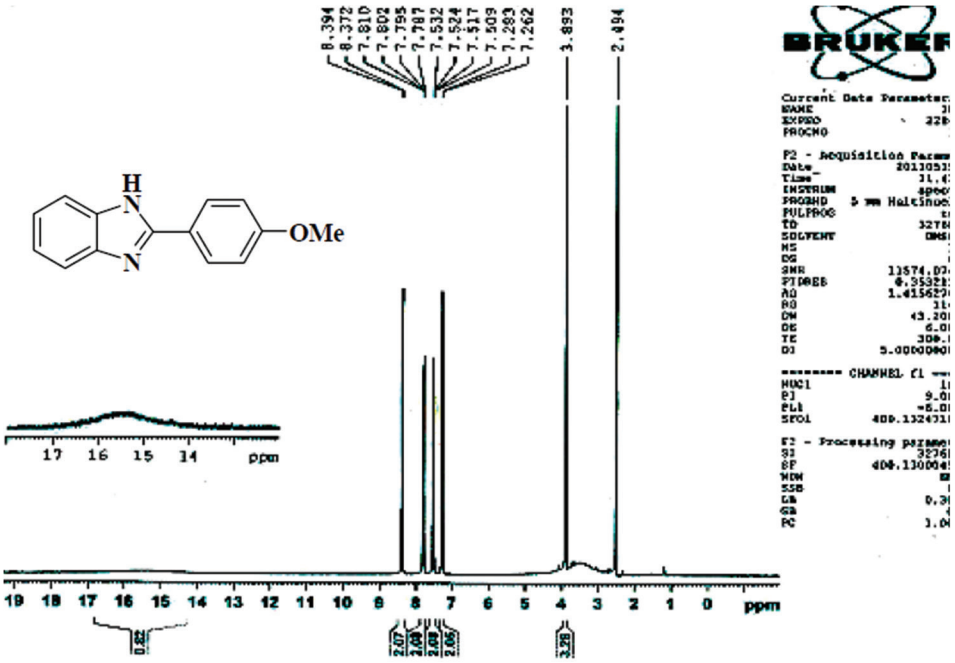
2-(2-Hydroxyphenyl)-benzimidazole (Figure 2 and 3m); ^{13}C-NMR (100 MHz, DMSO-d_6)



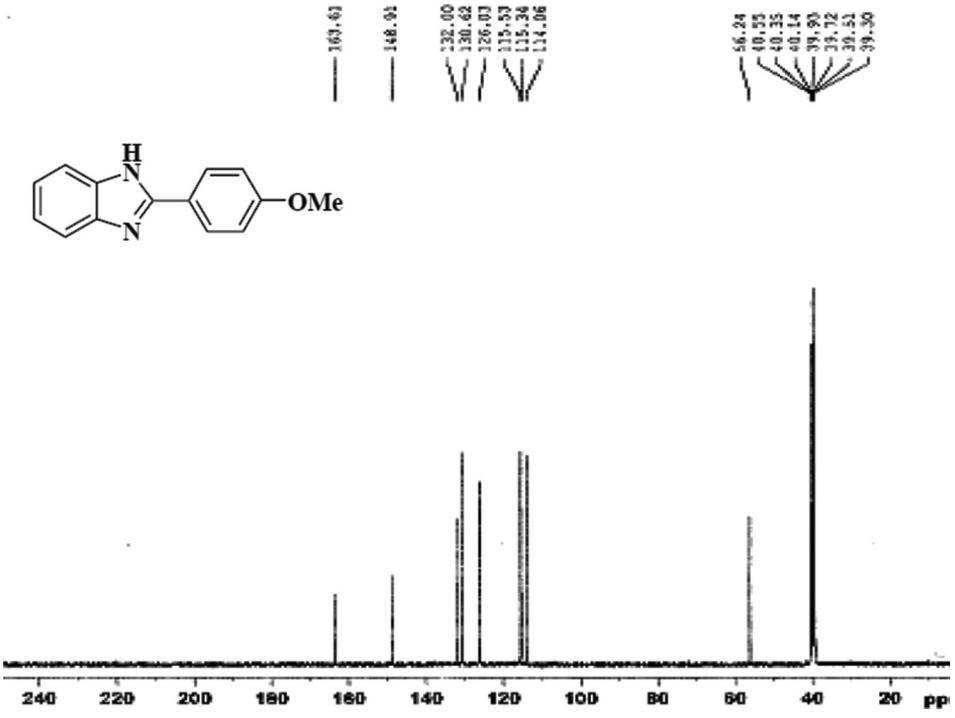
2-(3-Hydroxyphenyl)-benzimidazole (Figure 2 and 3n); ^{1}H-NMR (400 MHz, DMSO-d_6)



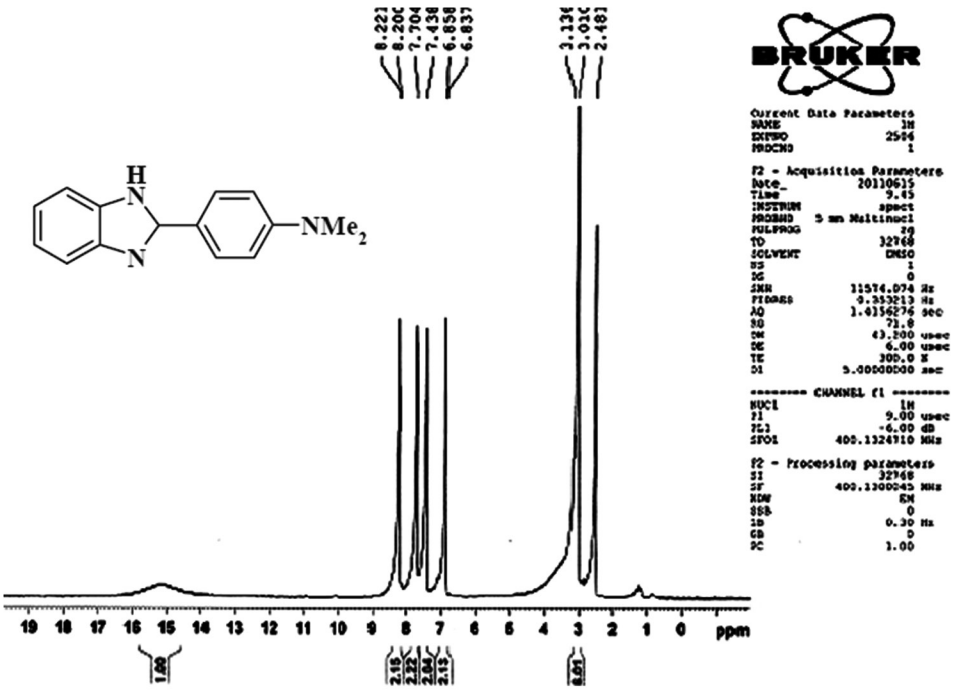
2-(3-Hydroxyphenyl)-benzimidazole (Figure 2 and 3n); ^{13}C-NMR (100 MHz, DMSO-d_6)



2-(4-Hydroxyphenyl)-benzimidazole (Figure 2 and 3o); ^{1}H-NMR (400 MHz, DMSO-d_6)



2-(4-Hydroxyphenyl)-benzimidazole (Figure 2 and 3o); ^{13}C-NMR (100 MHz, DMSO-d_6)



2-(N,N-Dimethylphenyl)-benzimidazole (Figure 2 and 3p); ^{1}H-NMR (400 MHz, DMSO-d_6)

Abbreviations: DMSO: Dimethyl sulfoxide; NMR: Nuclear magnetic resonance

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