

Article

Efficient and Eco-Friendly Perspectives for C-H Arylation of Benzothiazole Utilizing Pd Nanoparticle-Decorated Chitosan

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Supporting Information

S1. Characterization of the catalyst

S1a. Powder XRD

S1b. DR UV-*vis*

S1c. H₂-TPR of Pd@Chitosan sample

S2. The physical and spectroscopic data of the synthesized compounds

S3. NMR Charts of the synthesized compounds

Characterization of the catalyst

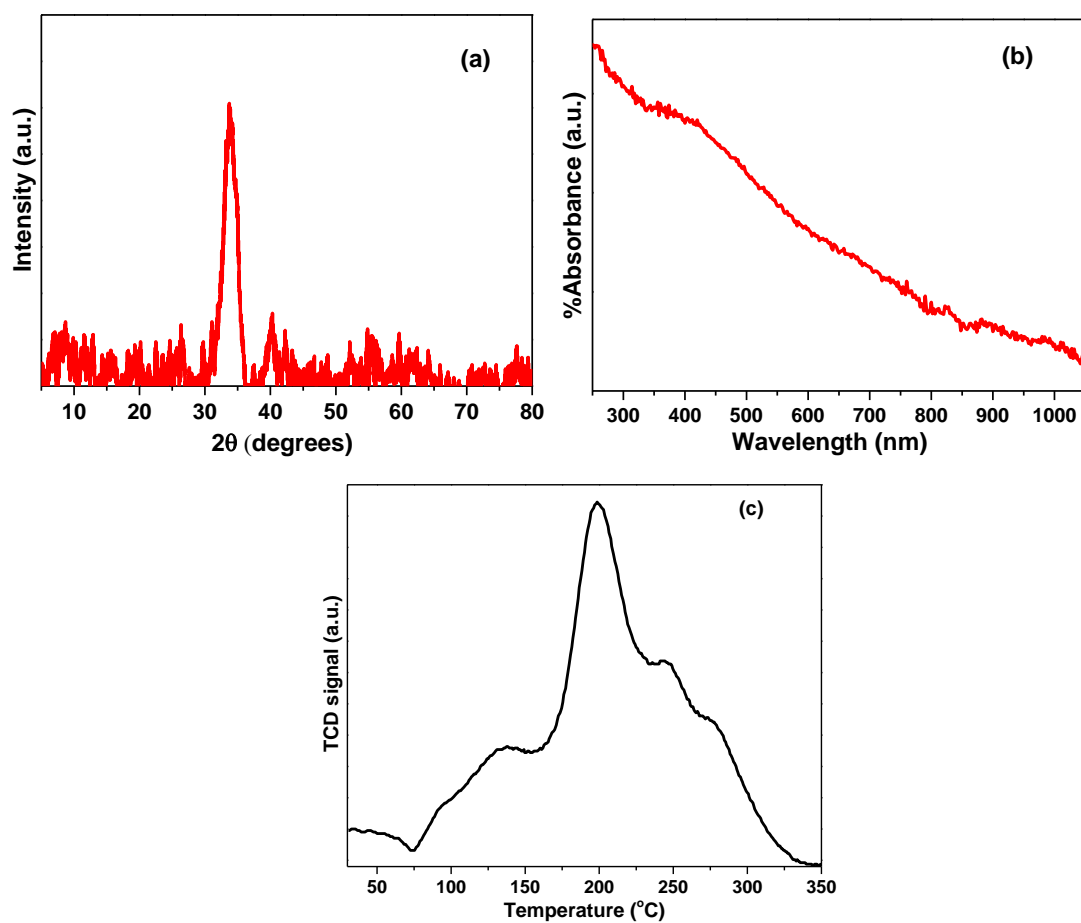


Figure.S1. (a) Powder XRD (b) DR UV-*vis* (c) H_2 -TPR of 5%Pd@Chitosan sample

S2: The physical and spectroscopic data of the synthesized compounds

1-(4-(Benzthiazol-2-yl)phenyl)ethan-1-one (3a)

¹H NMR (400 MHz, DMSO-*d*₆) δ 2.54 (s, 3H, CH₃), 7.16 (d, 2H, *J* = 7.6 Hz, ArH), 7.44-7.60 (m, 4H), 7.77 (d, 2H, *J* = 7.6 Hz, ArH). ¹³C NMR (100 MHz, DMSO-*d*₆) δ 26.8, 129.9, 128.7, 128.8, 129.4, 129.9, 133.9, 135.4, 137.8, 144.0, 149.0, 160.9, 193.0.

2-Phenylbenzthiazole (3b)

¹H NMR (400 MHz, CDCl₃) δ 7.35 (t, 1H, ArH), 7.46 (m, 4H, ArH), 7.88-8.10 (m, 4H, ArH). ¹³C NMR (100 MHz, CDCl₃) δ 121.6, 123.2, 125.2, 126.3, 127.5, 128.9, 130.9, 133.6, 135.1, 154.1, 168.0.

2-(4-Methoxyphenyl)benzothiazole (3c)

¹H NMR (400 MHz, CDCl₃) δ 3.88 (s, 3H, OCH₃), 6.97-7.0 (m, 2H, ArH), 7.35 (d, 2H, *J* = 7.4 Hz, ArH), 7.36-7.48 (m, 2H, ArH), (8.02-8.04 (m, 2H, ArH), ¹³C NMR (100 MHz, CDCl₃) δ 55.5, 114.4, 121.5, 122.8, 124.8, 126.2, 126.4, 129.2, 134.8, 154.2, 161.9, 167.9.

2-(4-Methylphenyl)benzothiazole (3d)

m.p. 85-87 °C, ¹H NMR (CDCl₃, 400 MHz) δ 2.42 (s, 3H, CH₃), 6.85 (d, 2H, *J* = 8 Hz, ArH), 6.90-6.93 (m, 2H, ArH), 7.04 (d, 2H, *J* = 8 Hz, ArH), 7.14-7.41 (m, 2H, ArH). ¹³C NMR (100 MHz, CDCl₃) 21.1, 120.9, 124.0, 126.4, 128.7, 129.2, 129.4, 130.7, 136.7, 137.7, 151.2, 167.8. MS (*m/z*): 225 (M⁺). Anal. Calcd for C₁₄H₁₁NS: C, 74.63; H, 4.92; N, 6.22; S, 14.23. Found: C, 74.76; H, 4.87; N, 6.20; S, 14.17 %.

Ethyl 4-(benzothiazol-2-yl)benzoate (3e)

¹H NMR (400 MHz, DMSO-*d*₆) δ 0.96 (t, 3H, *J* = 7.00 Hz, CH₃ ester), 4.02 (q, 2H, *J* = 7.00 Hz, CH₂ ester), 7.36 (d, 2H, *J* = 7.2 Hz, ArH), 7.46-7.88 (m, 4H, ArH), 8.04 (d, 2H, *J* = 7.2 Hz, ArH). ¹³C NMR (100 MHz, DMSO-*d*₆) δ 13.9, 61.3, 119.9, 121.7,

122.8, 124.6, 127.5, 129.3, 130.9, 135.5, 144.9, 145.6, 154.9, 165.3, 169.4. MS (m/z): 283 (M⁺). Anal. Calcd for C₁₆H₁₃NO₂S: C, 67.82; H, 4.62; N, 4.94; S, 11.31. Found: C, 67.98; H, 4.59; N, 4.89; S, 11.27 %.

S3. NMR Charts of the synthesized compounds

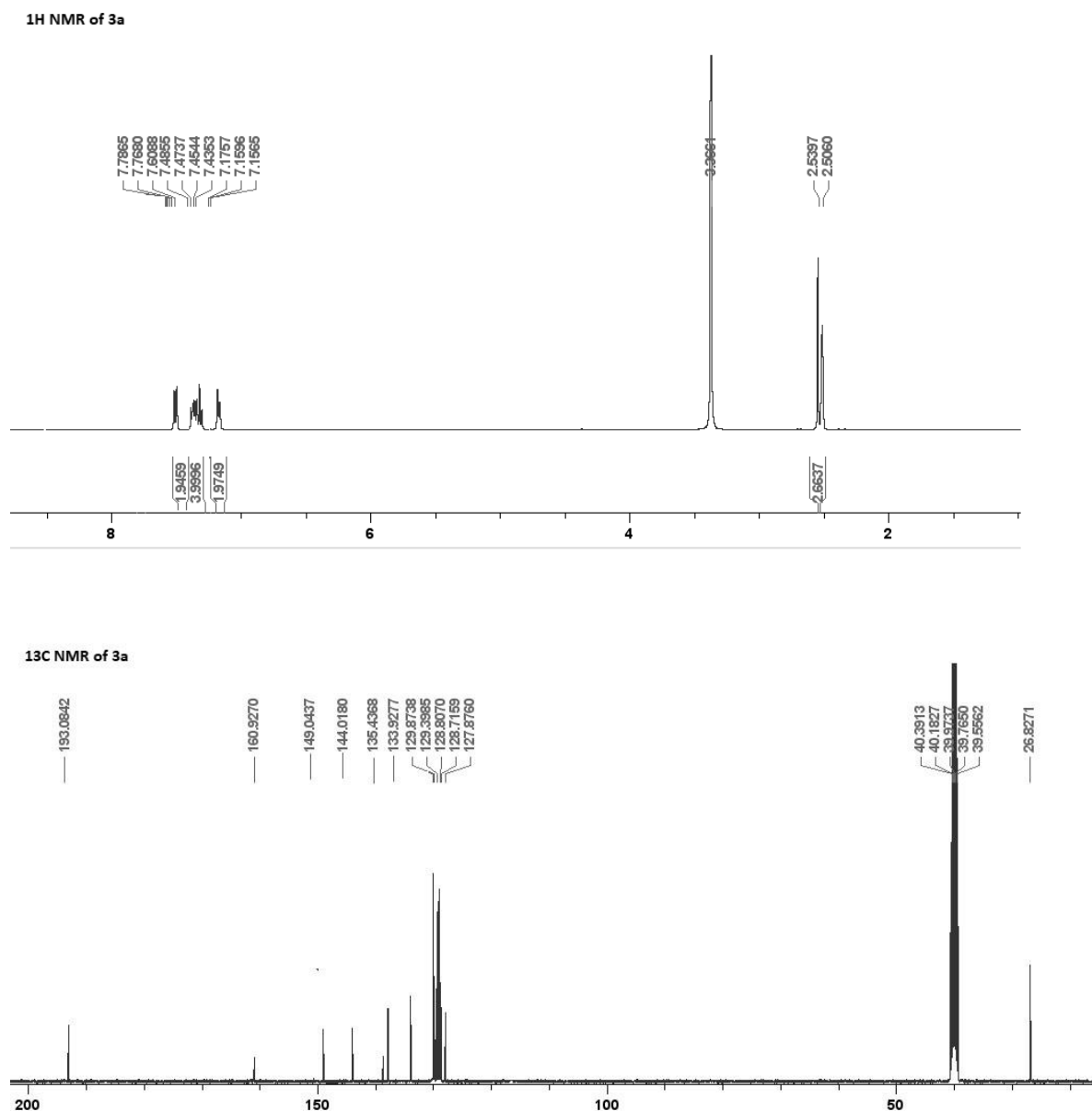
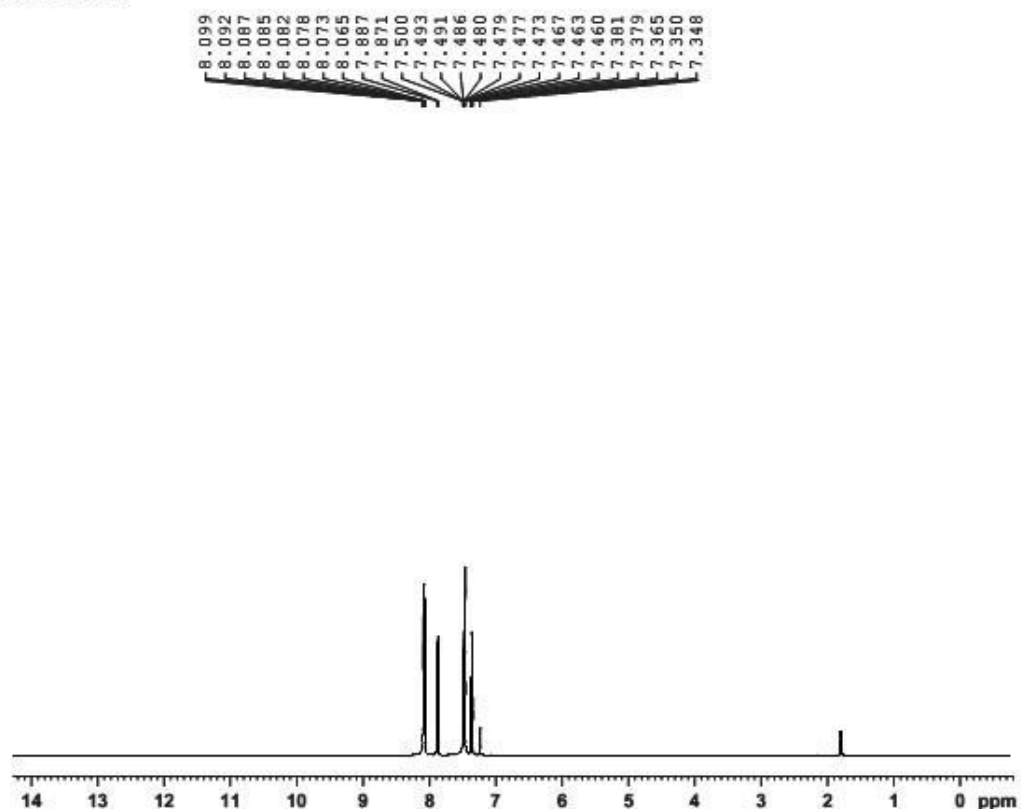


Figure S2. ¹H NMR and ¹³C NMR of 1-(4-(Benzthiazol-2-yl)phenyl)ethan-1-one (3a)

¹H NMR of 3b



¹³C NMR of 3b

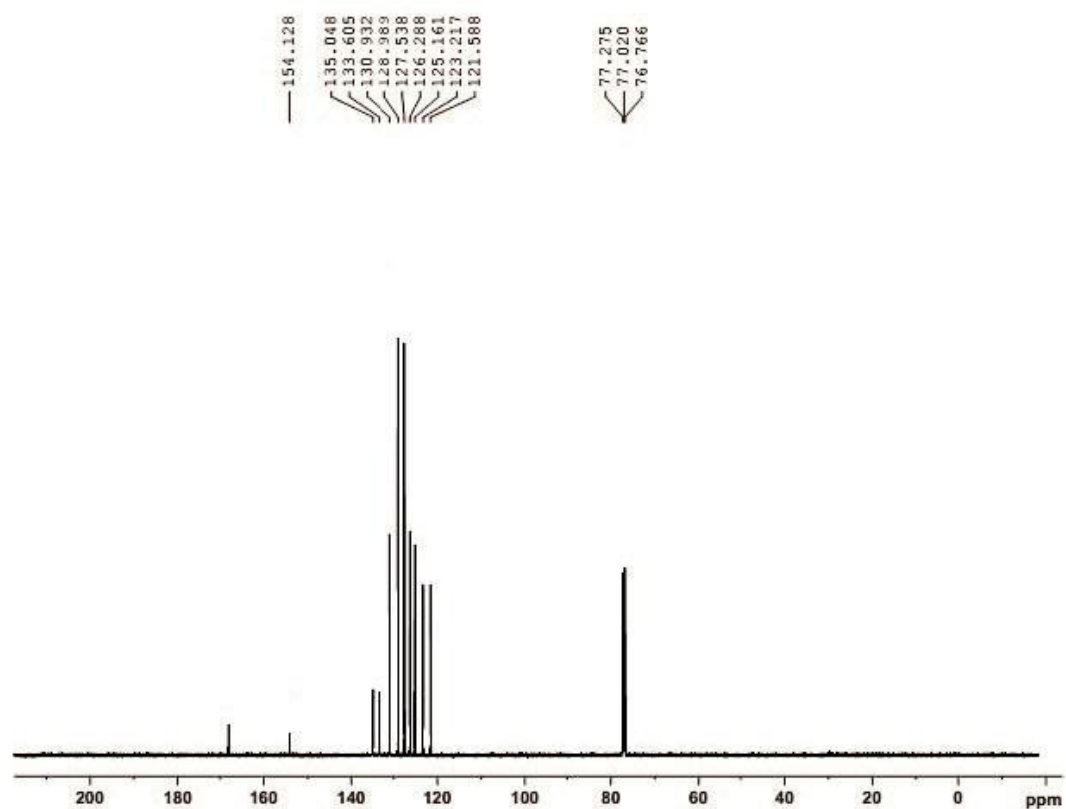
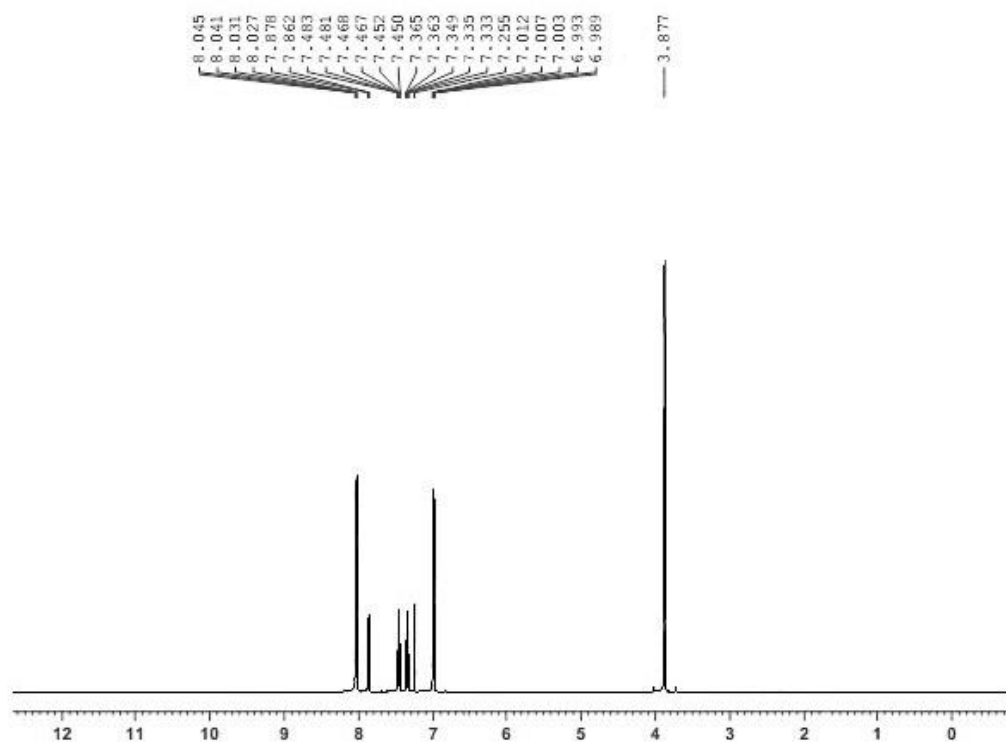


Figure S3. ^1H NMR and ^{13}C NMR of 2-Phenylbenzthiazole (3b)

^1H NMR of 3c



¹³C NMR of 3c

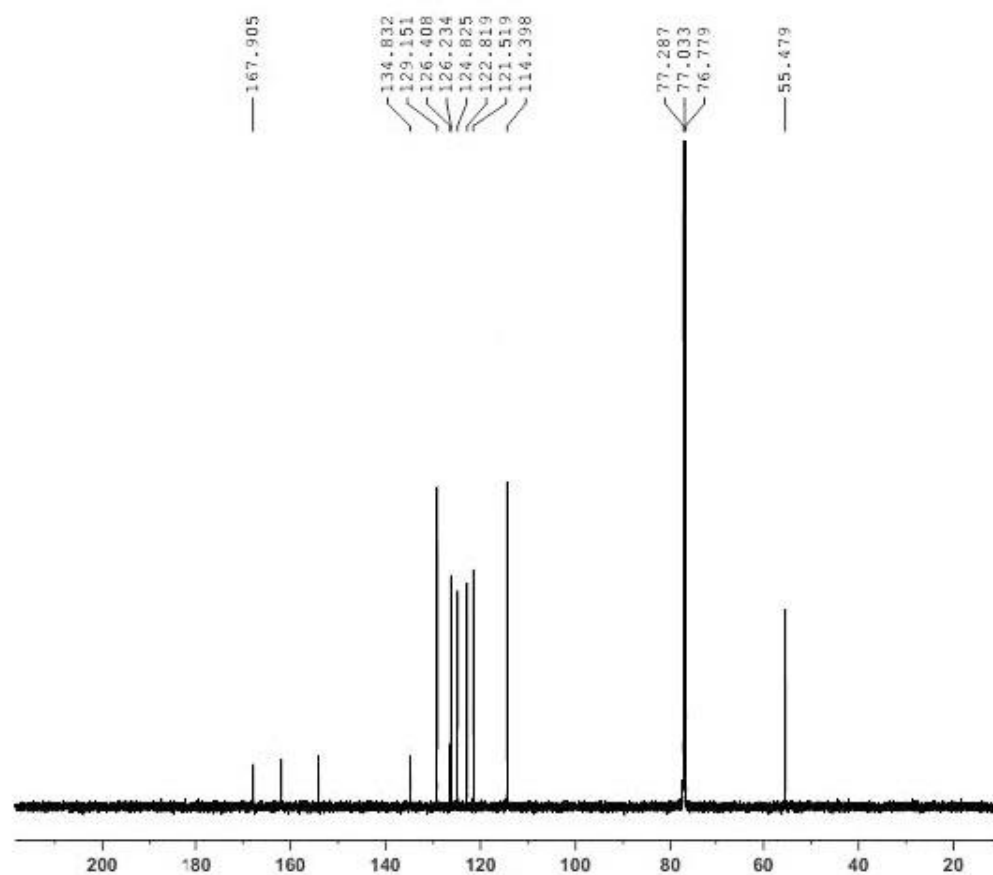
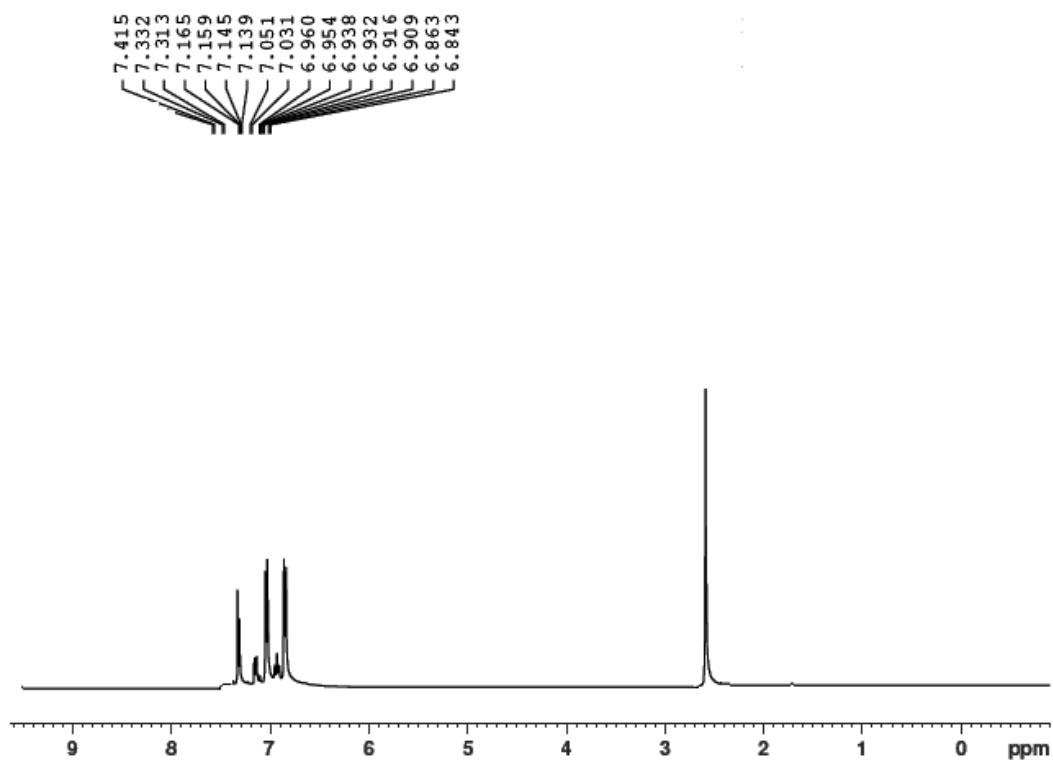


Figure S4. ¹H NMR and ¹³C NMR of 2-(4-Methoxyphenyl)benzothiazole (3c)

¹H NMR of 3d



¹³C NMR of 3d

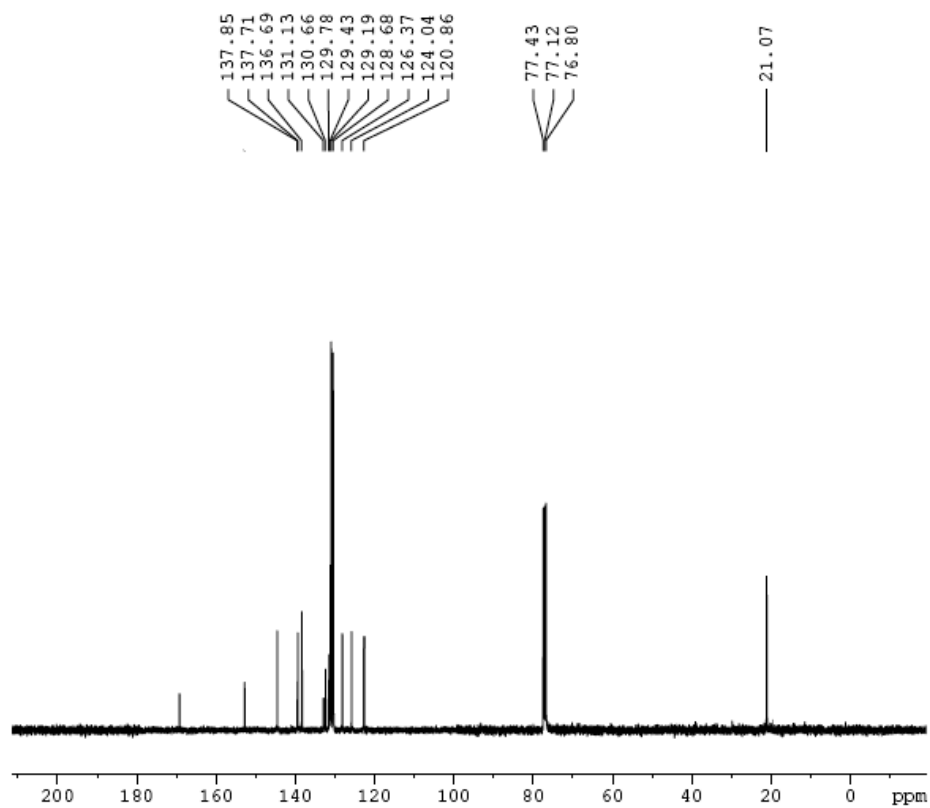


Figure S5. ¹H NMR and ¹³C NMR of 2-(4-Methylphenyl)benzothiazole (3d)