

Supporting Information

g-C₃N₄/γ-Fe₂O₃/TiO₂/Pd: A new magnetically separable photocatalyst for visible-light-driven fluoride-free Miyaura and Suzuki–Miyaura cross-coupling reactions at room temperature

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XRD analysis:

XRD analysis was carried out to understand the structural features of the fabricated photocatalyst (Figure S1). Figure S1-a presents the XRD pattern of $\gamma\text{-Fe}_2\text{O}_3$, which revealed a series of characteristic peaks appeared at 30.3° , 35.8° , 43.4° , 53.9° , 57.4° and 63° (2θ) associated with (2 2 0), (3 1 1), (4 0 0), (4 2 2), (5 1 1) and (4 4 0) planes of the cubic structure of maghemite (JCPDS card No. 04-0755).^[1] As it can be perceived from the XRD pattern of $\text{g-C}_3\text{N}_4/\gamma\text{-Fe}_2\text{O}_3/\text{TiO}_2$ (Figure S1-b), the characteristic peaks concerning $\gamma\text{-Fe}_2\text{O}_3$ can be easily observed. Also, two indicative diffraction peaks observed at 27.4° and 13.1° (2θ) can be attributed to the (0 0 2) and (1 0 0) crystallographic faces of $\text{g-C}_3\text{N}_4$ in the final photocatalyst.^[2,3] The appearance of the characteristic peaks at around 25.3° , 37.8° , 48.2° , 54.2° , 55.3° , 62.5° , and 70.4° (2θ) relating to the (1 0 1), (0 0 4), (2 0 0), (1 0 5), (2 1 1), (2 0 4) and (2 2 0) plane diffractions, confirms the anatase phase formation of TiO_2 (JCPDS card No. 21-1272)^[4] in the $\text{g-C}_3\text{N}_4/\gamma\text{-Fe}_2\text{O}_3/\text{TiO}_2$.

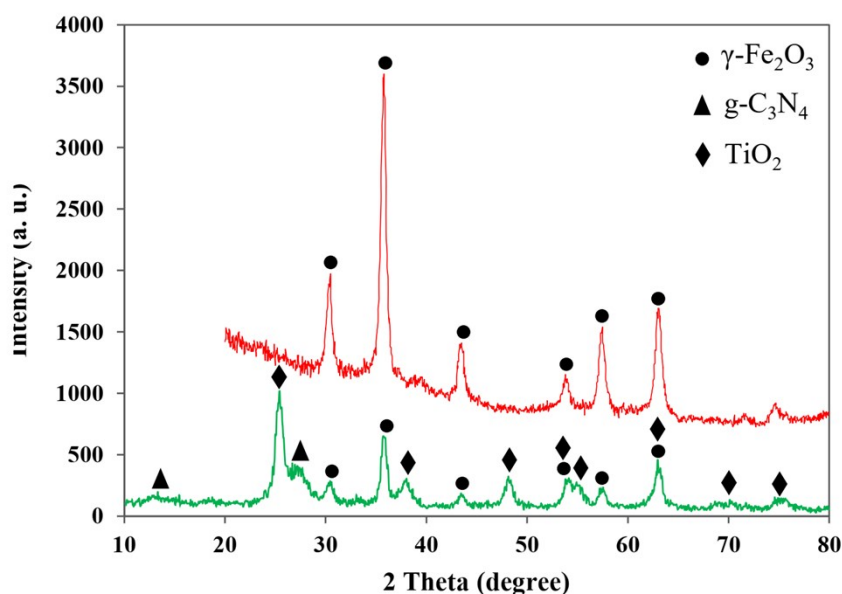


Figure S1. XRD patterns of (a) $\gamma\text{-Fe}_2\text{O}_3$ and (b) $\text{g-C}_3\text{N}_4/\gamma\text{-Fe}_2\text{O}_3/\text{TiO}_2$.

Elemental mapping analysis:

Moreover, to explore the elemental composition uniformity of $g\text{-C}_3\text{N}_4/\gamma\text{-Fe}_2\text{O}_3/\text{TiO}_2$, elemental mapping analysis was conducted (Figure S2). As it is evident, the simultaneous existence of C, O, N, Fe and Ti elements with homogeneous distribution was well proved.

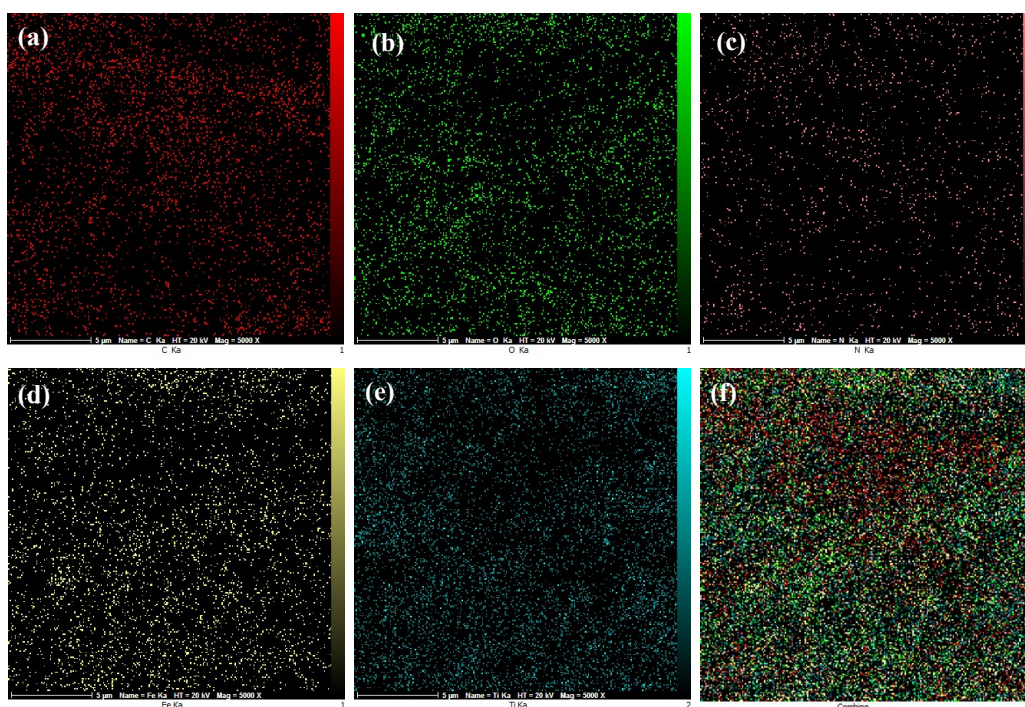
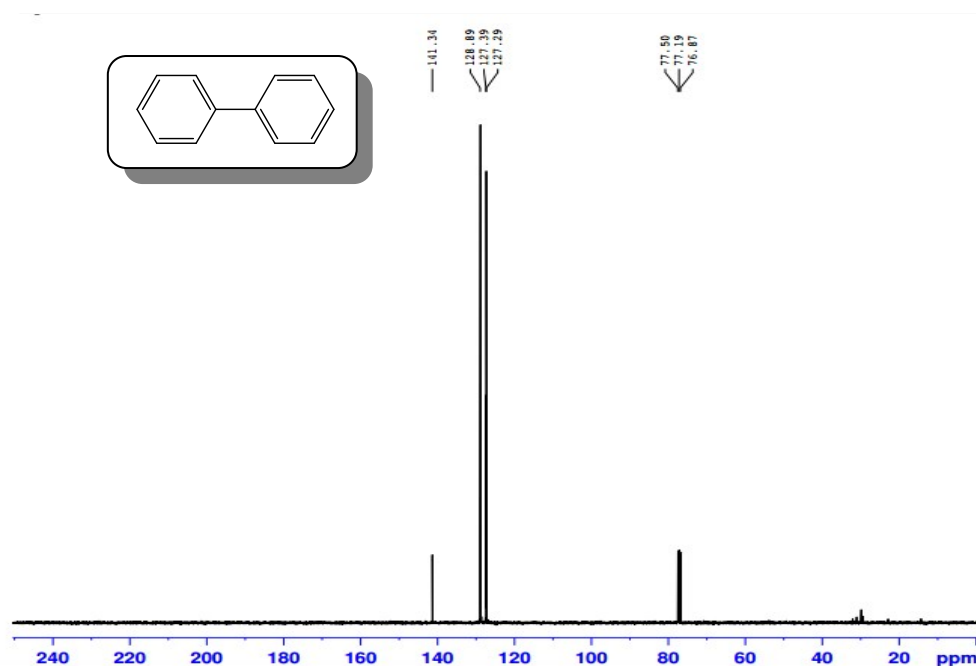
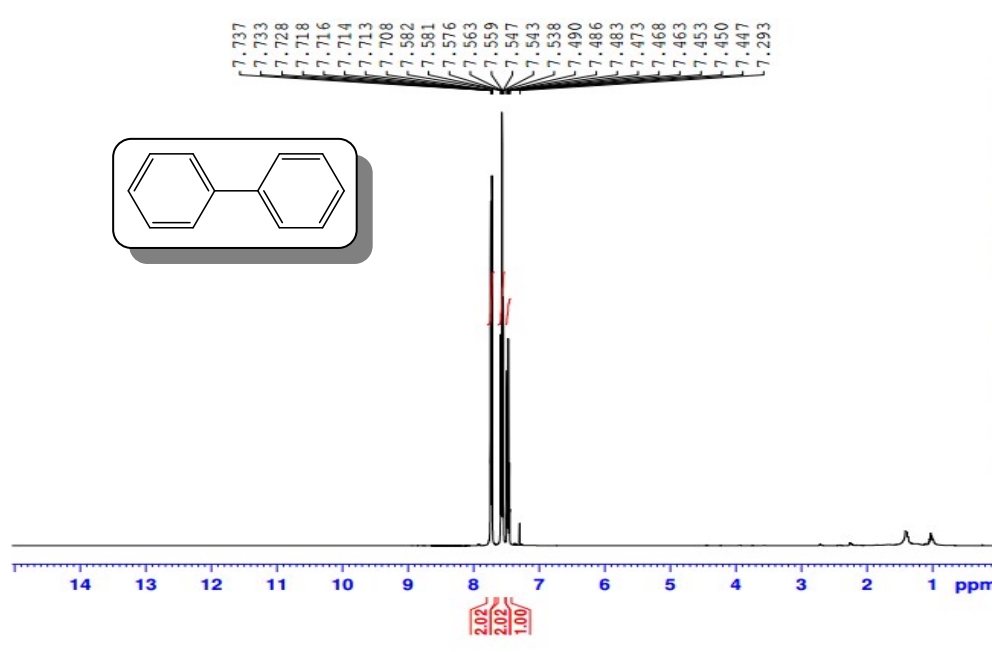
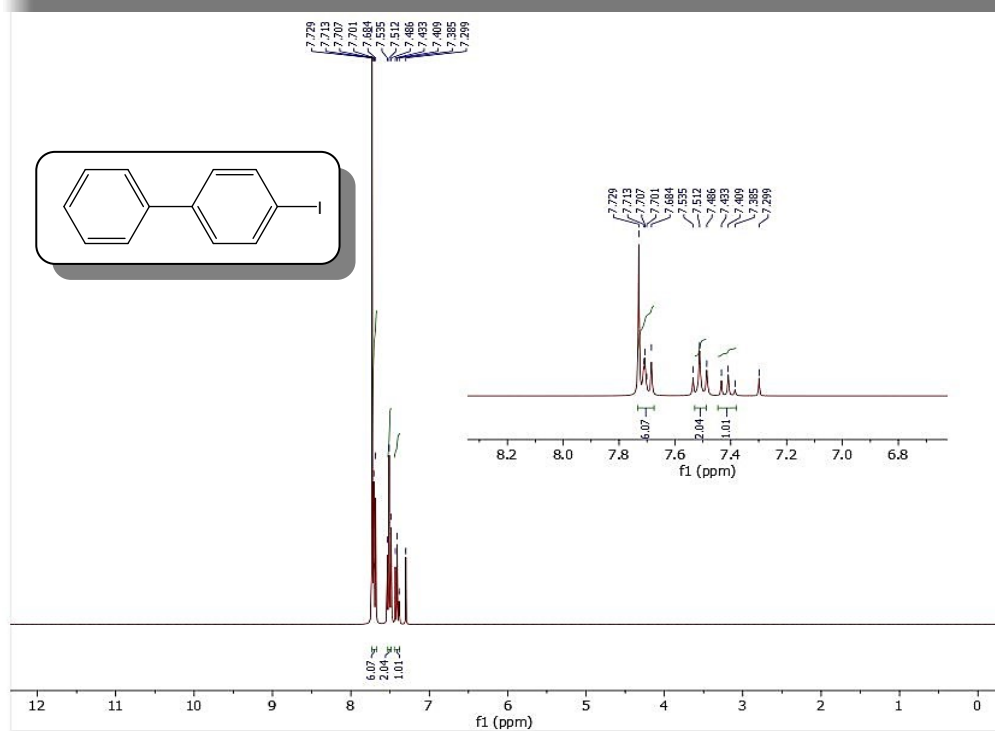


Figure S2. Elemental mapping images of (a) carbon (red), (b) oxygen (green), (c) nitrogen (pink), (d) iron (yellow), (e) titanium (cyan) and (f) the overlapping of C, O, N, Fe and Ti elements in $g\text{-C}_3\text{N}_4/\gamma\text{-Fe}_2\text{O}_3/\text{TiO}_2$.



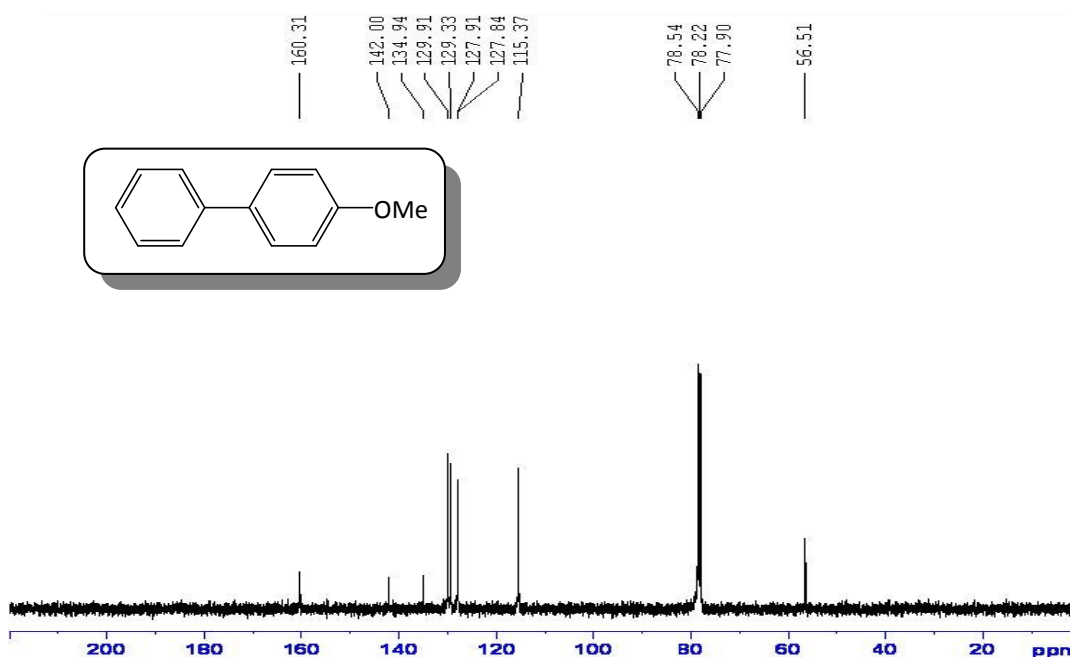
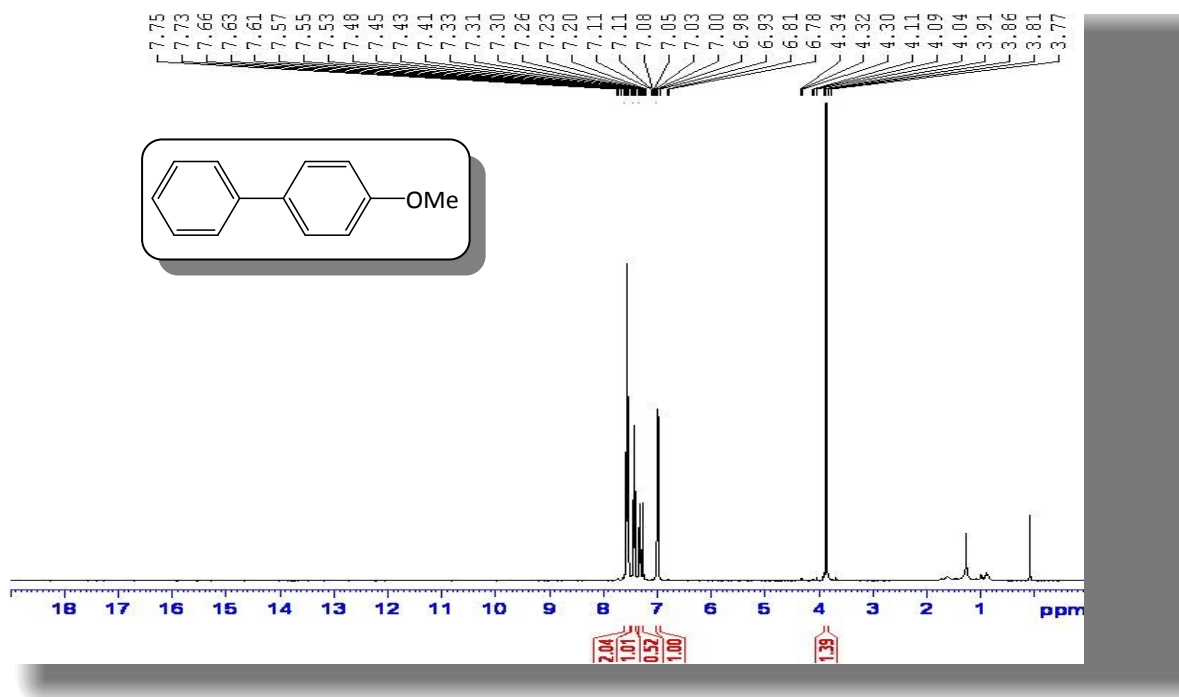
¹H NMR and ¹³C NMR spectra of 1, 1'-Biphenyl

1, 1'-Biphenyl: ¹H NMR (400 MHz, CDCl₃): δ 7.72 (d, 4H, ³J = 6.8 Hz), 7.56 (t, 4H, ³J = 8.0 Hz), 7.46 (t, 2H, ³J = 7.2 Hz) ppm. ¹³C NMR (100 MHz, CDCl₃), δ 141.3, 128.8, 127.3, 127.2 ppm (Table 2 and Table 3: entries 1, 5, 10).



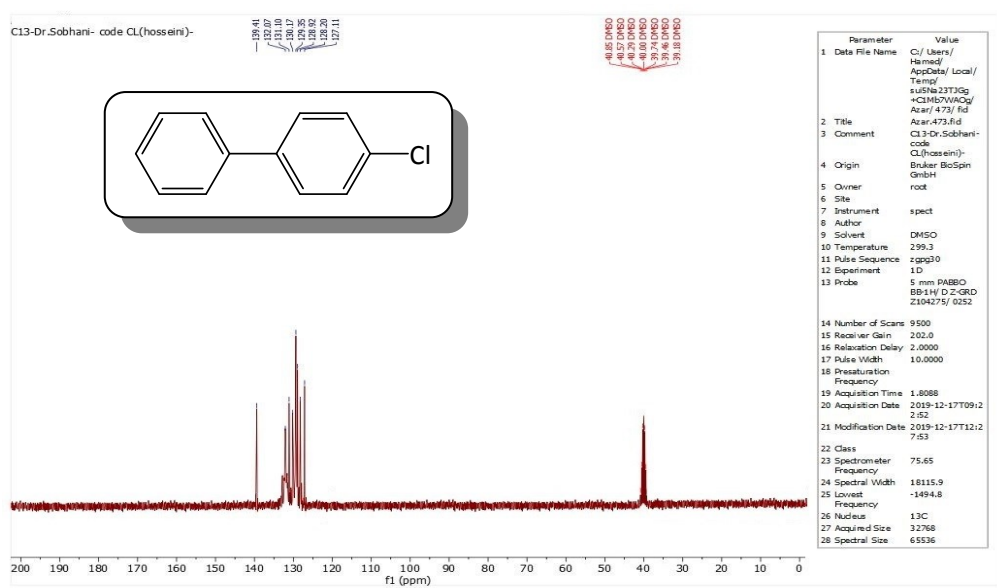
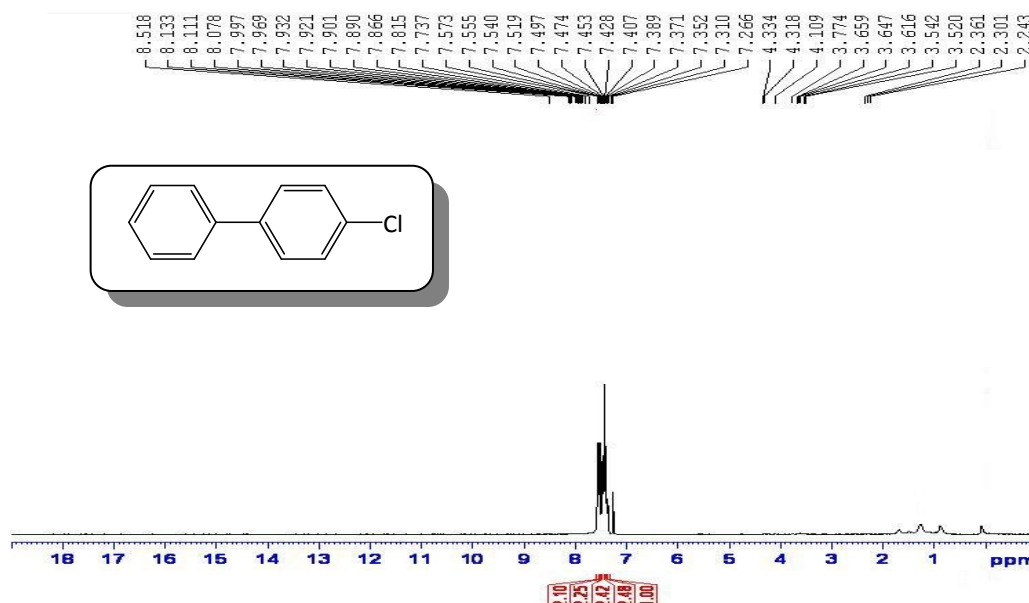
^1H NMR spectrum of 4-iodo-1, 1'-biphenyl

4-iodo-1, 1'-biphenyl: ^1H NMR (300 MHz, CDCl_3): δ 7.67-7.73 (m, 6H), 7.51 (t, 2H, $^3J = 7.8$ Hz), 7.40 (t, 1H, $^3J = 7.2$ Hz) ppm (Table 2 and Table 3: entry 2).



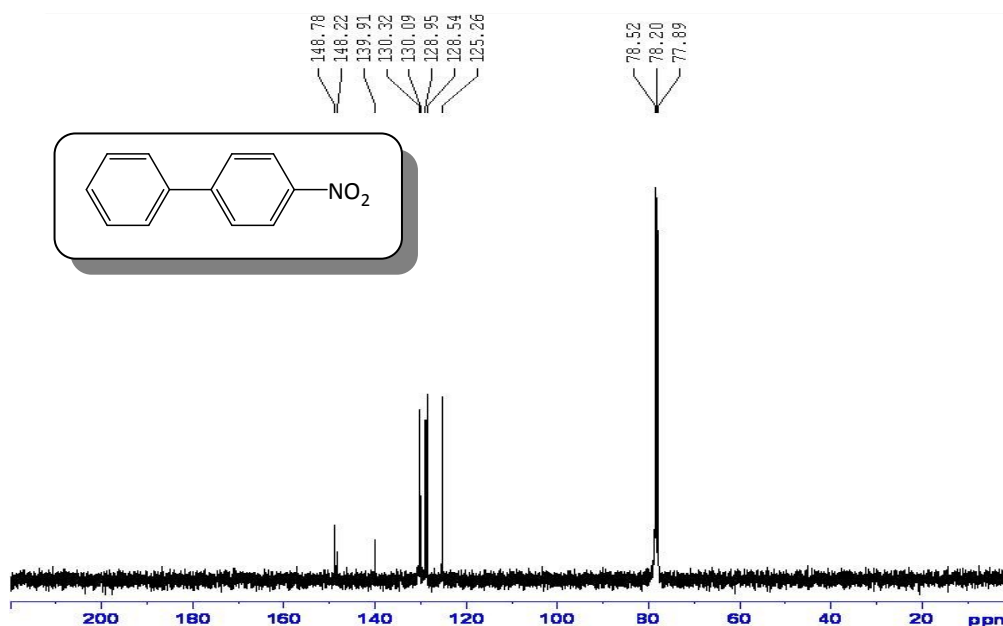
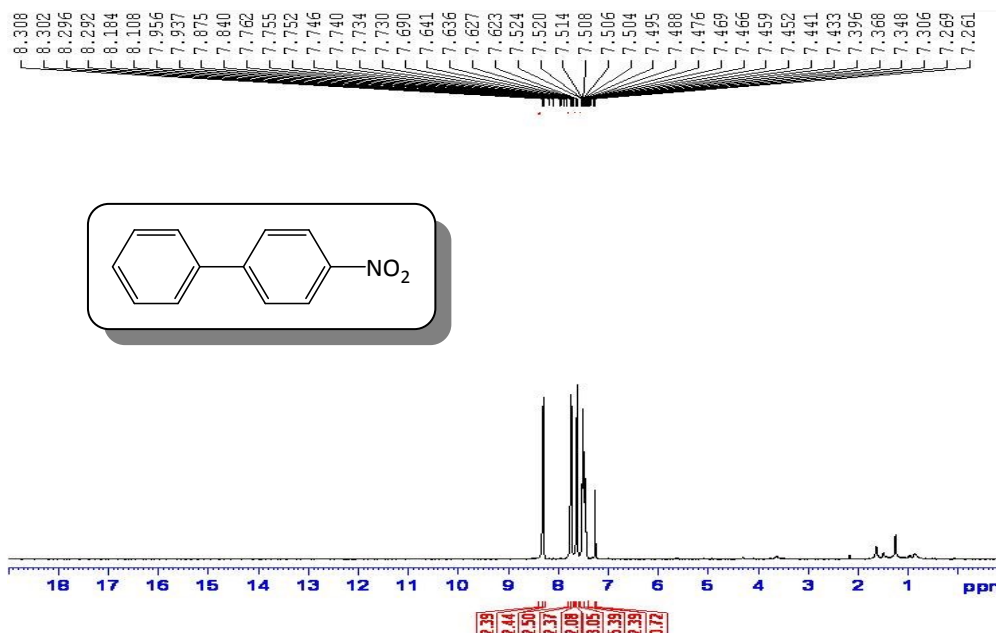
¹H NMR and ¹³C NMR spectra of 4-Methoxy-1, 1'-biphenyl

4-Methoxy-1, 1'-biphenyl: ¹H NMR (400 MHz, CDCl₃): δ 7.55 (t, 4H, ³J = 8.8 Hz), 7.43 (t, 2H, ³J = 8.0 Hz), 7.31 (t, 1H, ³J = 7.2 Hz), 6.99 (d, 2H, ³J = 8.8 Hz), 3.83 (s, 3H) ppm. ¹³C NMR (100 MHz, CDCl₃), δ 160.3, 142.0, 134.9, 129.9, 129.3, 127.9, 127.8, 115.3, 56.5 ppm (Table 2 and Table 3: entries 3, 7, 13).



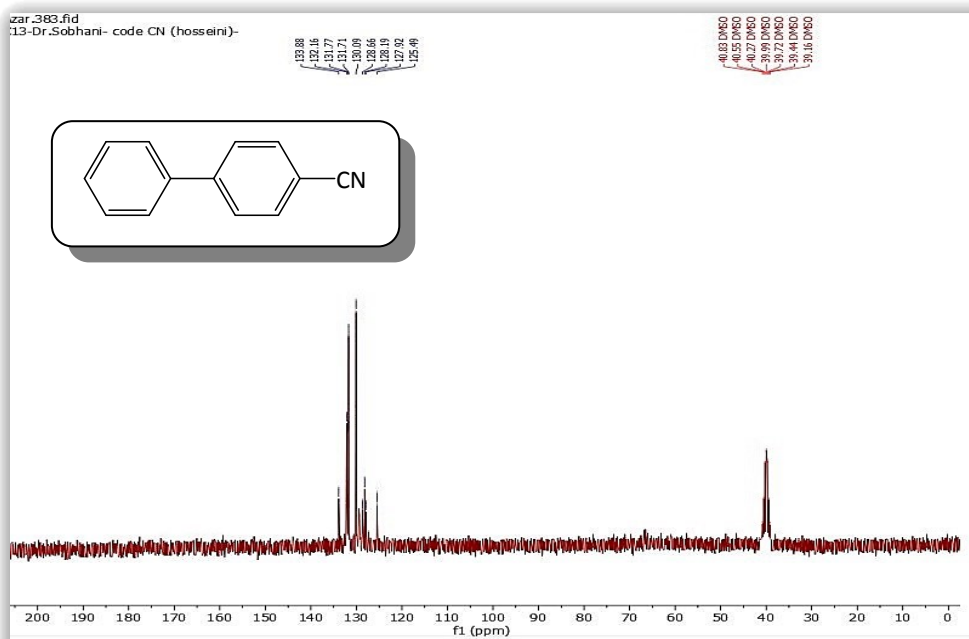
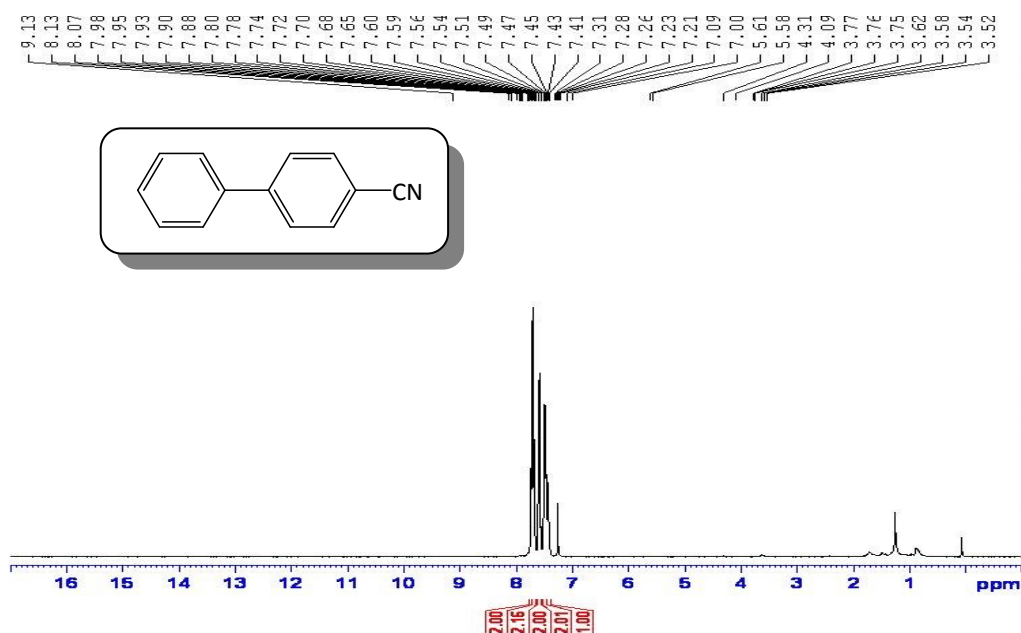
¹H NMR and ¹³C NMR spectra of 4-Chloro-1, 1'-biphenyl

4-Chloro-1, 1'-biphenyl: ¹H NMR (400 MHz, CDCl₃): δ 7.51-7.57 (m, 4H), 7.31-7.49 (m, 5H), ppm. ¹³C NMR (75 MHz, DMSO-*d*₆), δ 139.4, 132.0, 131.1, 130.1, 129.3, 128.9, 128.2, 127.1 ppm (Table 2 and Table 3: entries 4, 8).



¹H NMR and ¹³C NMR spectra of 4-Nitro-1, 1'-biphenyl

4-Nitro-1, 1'-biphenyl: ¹H NMR (400 MHz, CDCl₃): δ 8.29-8.30 (m, 2H), 7.73-7.76 (m, 2H), 7.62-7.64 (m, 2H), 7.43-7.52 (m, 3H) ppm. ¹³C NMR (100 MHz, CDCl₃), δ 148.7, 148.2, 139.9, 130.3, 130.0, 128.9, 128.5, 125.2 ppm (Table 2 and Table 3: entries 6, 11).



¹H NMR and ¹³C NMR spectra of 4-Cyano-1, 1'-biphenyl

4-Cyano-1, 1'-biphenyl: ^1H NMR (400 MHz, CDCl_3): δ 7.68-7.74 (m, 4H), 7.56-7.60 (m, 2H), 7.41-7.51 (m, 3H) ppm. ^{13}C NMR (75 MHz, $\text{DMSO}-d_6$), δ 133.8, 132.1, 131.7, 131.7, 130.0, 128.6, 128.1, 127.9, 125.4 ppm (Table 2 and Table 3: entries 9, 12).

References

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