Methylation of Amines and Ketones with Methanol Catalyzed by an Iridium Complex Bearing a 2-Hydroxypyridylmethylene Fragment

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Crystallographic Details

1: A total of 12642 reflections ($-12 \leq h \leq 12$, $-8 \leq k \leq 8$, $-18 \leq l \leq 18$) were collected at T = 173(2) K in the range of 2.794 to 24.972^{o} of which 2183 were unique (R_{int} = 0.0380); Mo_K radiation (λ = 0.71073 Å). The structure was solved by the direct methods. All non-hydrogen atoms were refined anisotropically, and hydrogen atoms were placed in calculated idealized positions. The residual peak and hole electron densities were 0.626 and -1.190 eA^{-3}, respectively. The least squares refinement converged normally with residuals of $R(F)$ = 0.0163, $wR(F^2)$ = 0.0382 and a GOF = 1.180 ($I>2\sigma(I)$). $C_{22}H_{27}Cl_4IrN_2O_2$, Mw = 685.45, space group Pmc2(1), Orthorhombic, a = 10.7919(7), b = 7.2894(5), c = 15.3894(10) Å, V = 1210.63(14) $Å^3$, Z = 2, ρ_{calcd} = 1.880 Mg/m^3. CCDC-1859831 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

^{1}H NMR Spectra

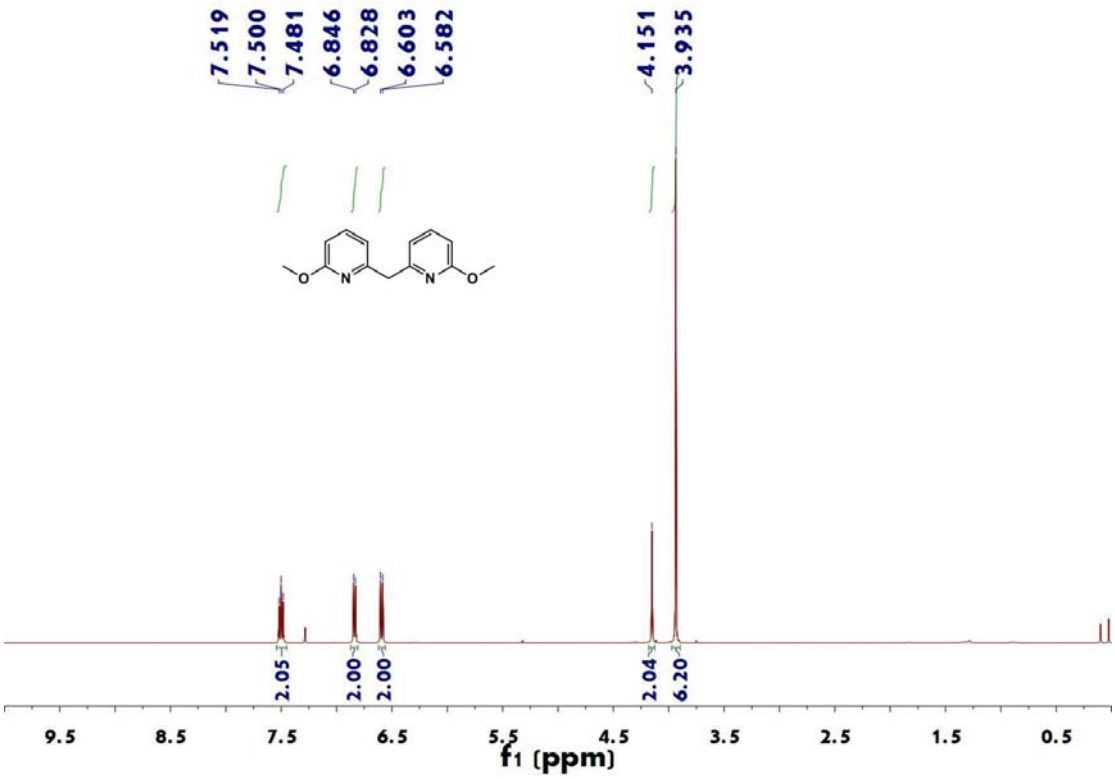

Figure S1. ^{1}H NMR spectrum of 2-methoxy-6-((6-methoxypyridin-2-yl)methyl)pyridine in $CDCl_3$.

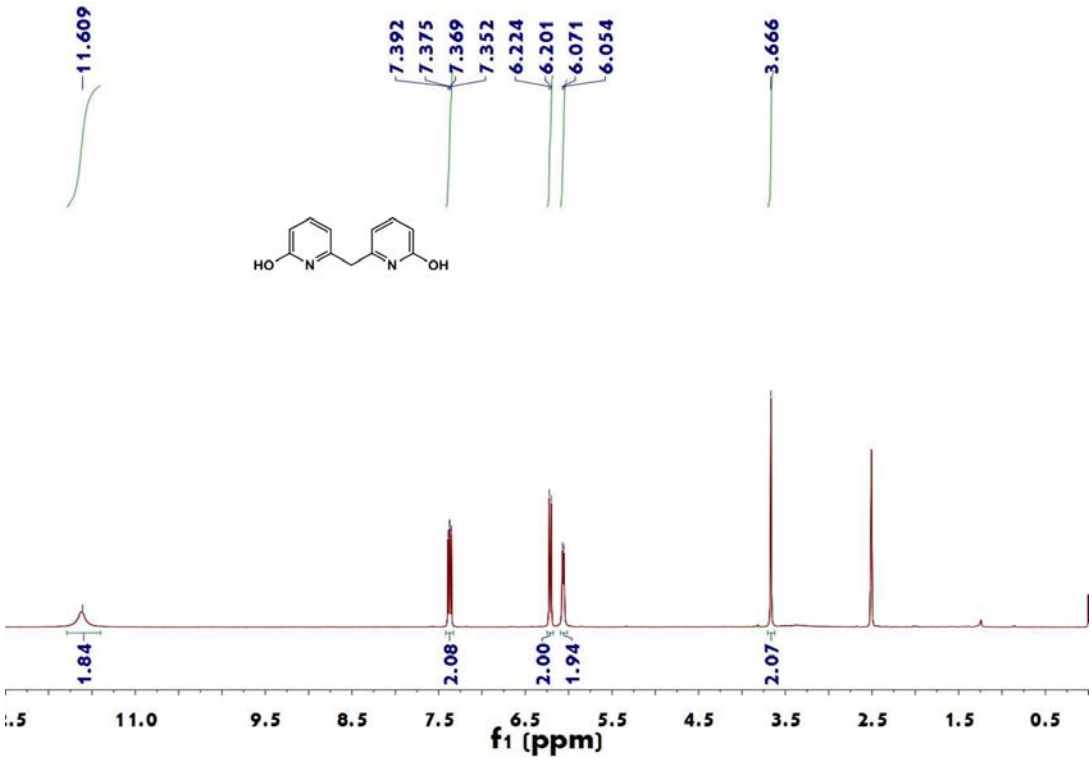

Figure S2. ^{1}H NMR spectrum of complex $\mathbf{L_1}$ in d_6-DMSO.

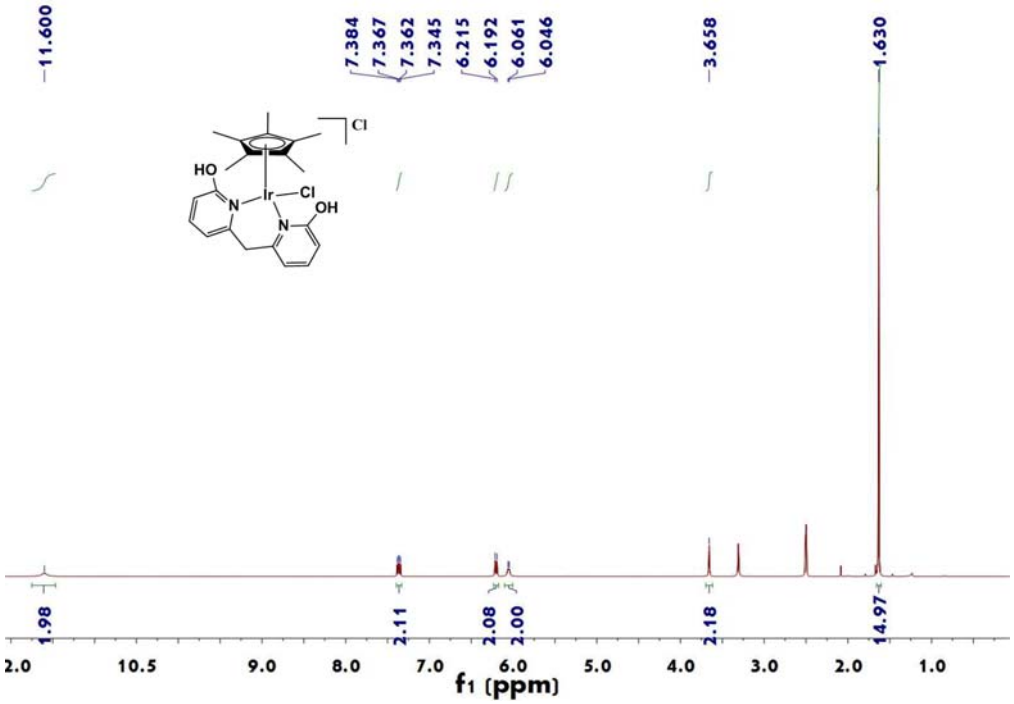

Figure S3. ^{1}H NMR spectrum of complex **1** in d_6-DMSO.

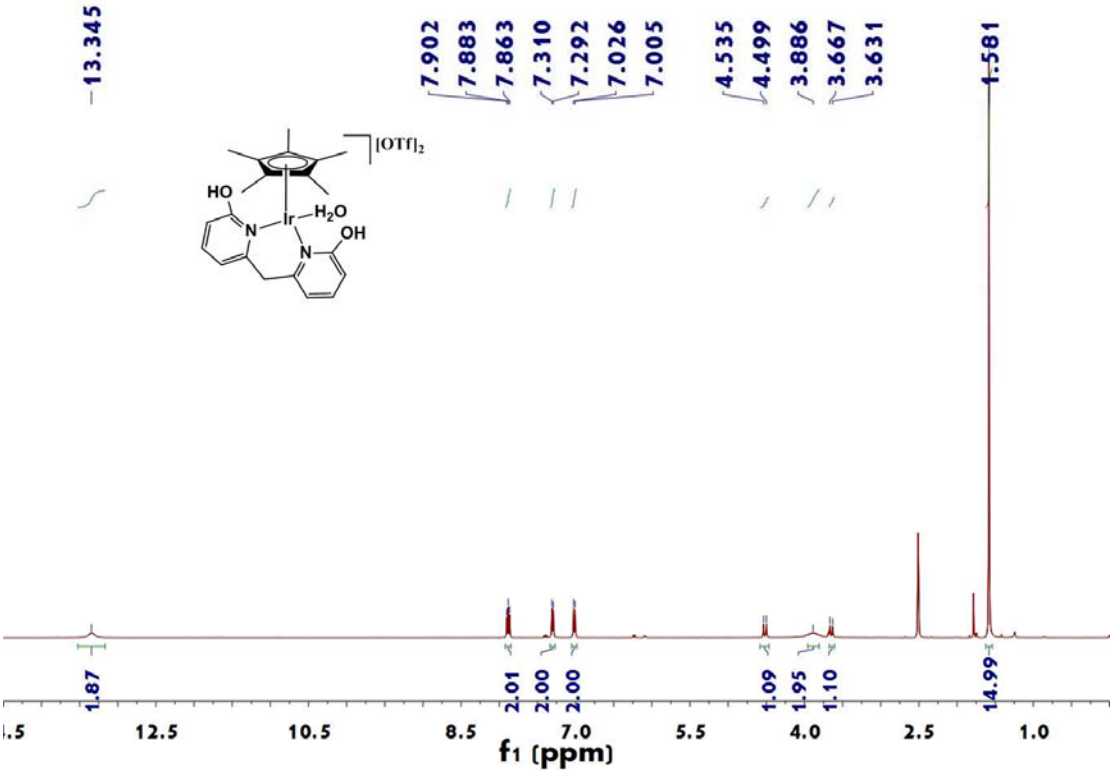

Figure S4. ^{1}H NMR spectrum of complex **2** in d_6-DMSO.

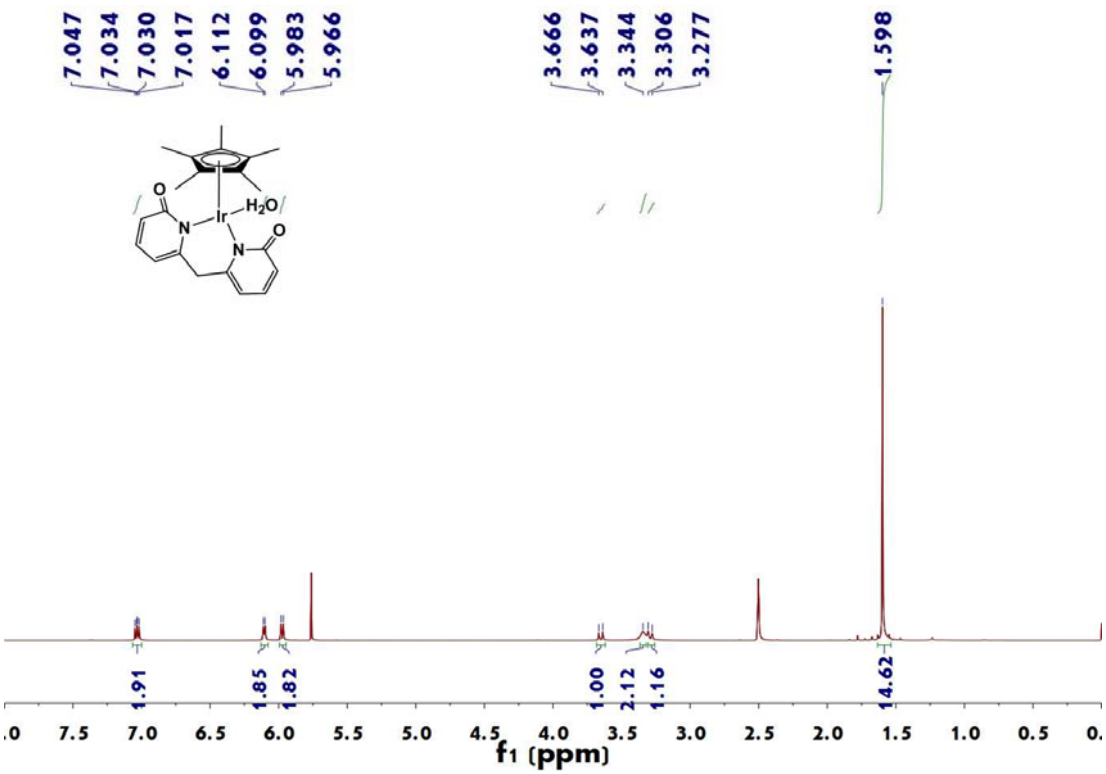

Figure S5. ^{1}H NMR spectrum of complex **3** in d_6-DMSO.

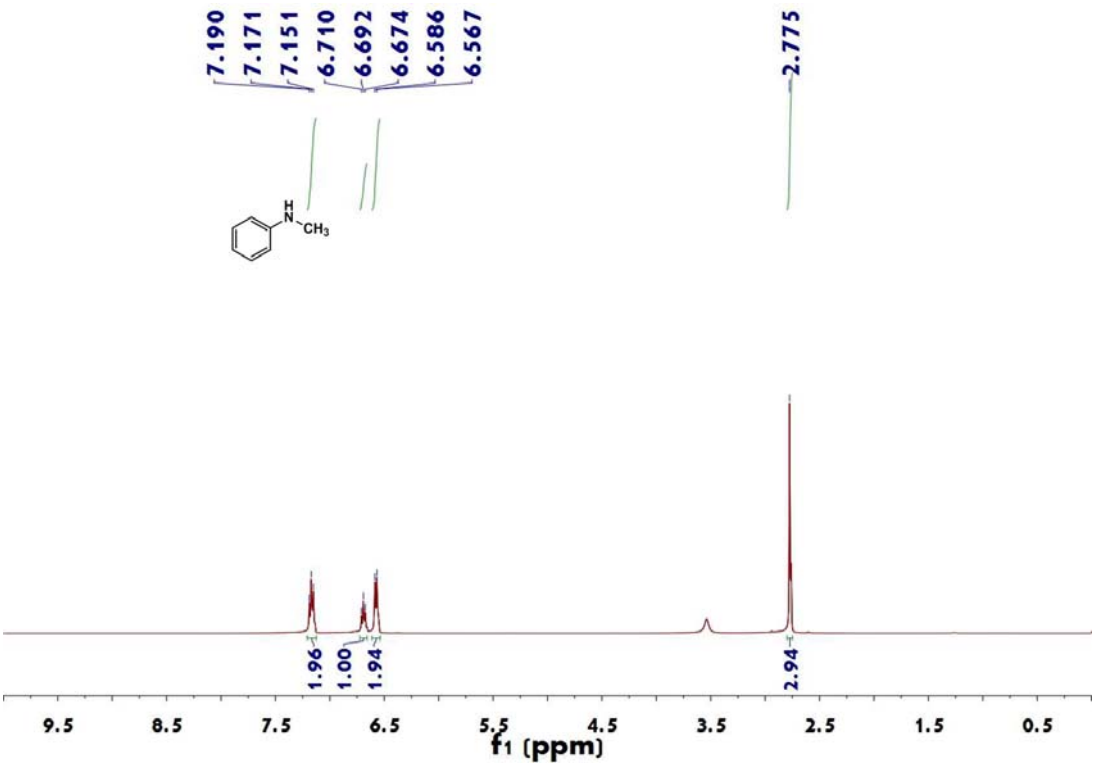

Figure S6. ^{1}H NMR spectrum of *N*-methylbenzenamine in $CDCl_3$.

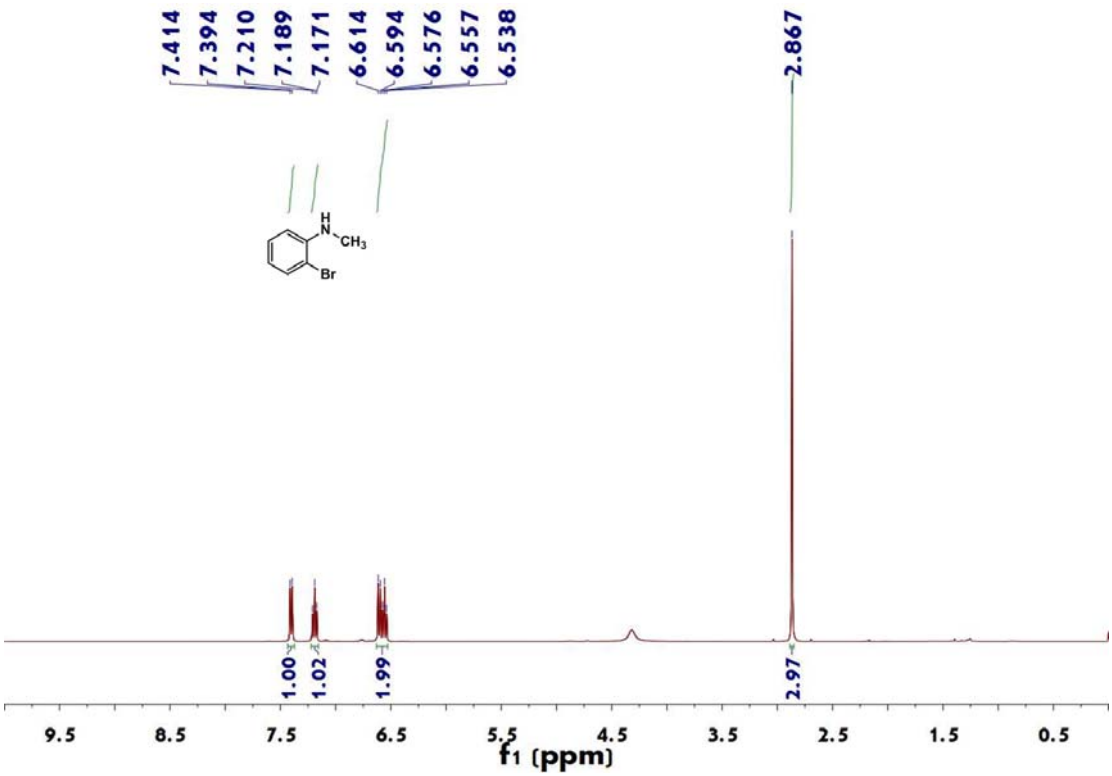

Figure S7. ^{1}H NMR spectrum of 2-bromo-*N*-methylbenzenamine in $CDCl_3$.

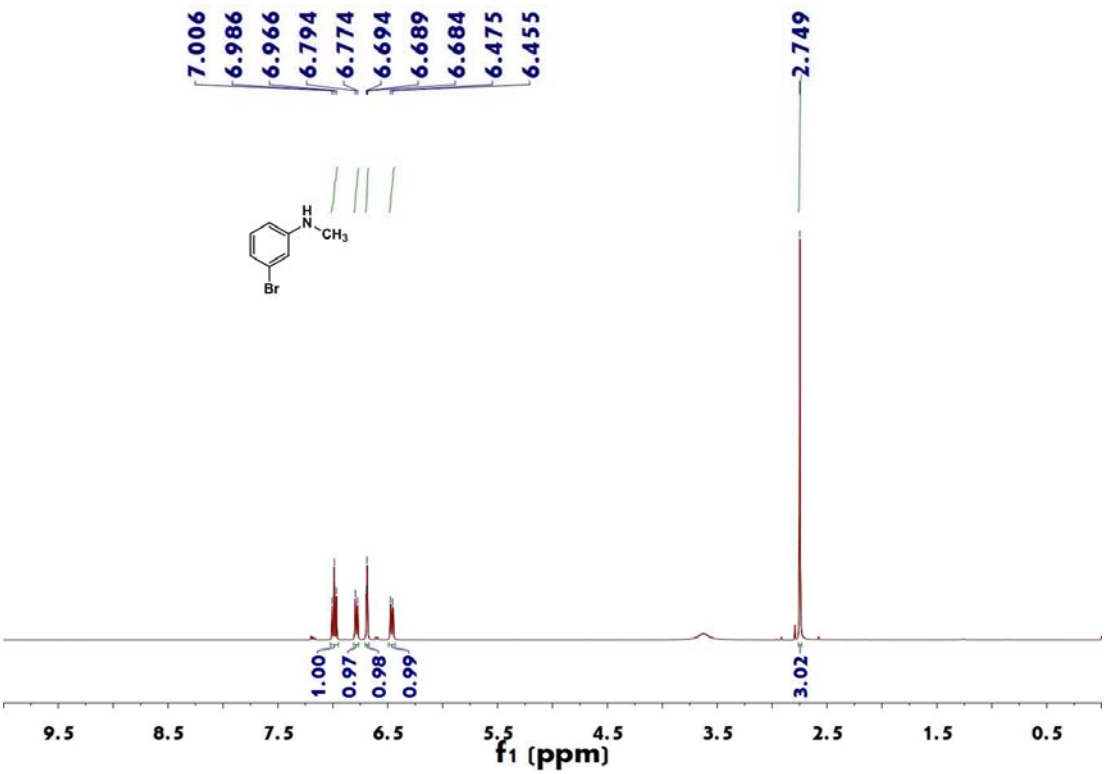

Figure S8. ^{1}H NMR spectrum of 3-bromo-*N*-methylbenzenamine in $CDCl_3$.

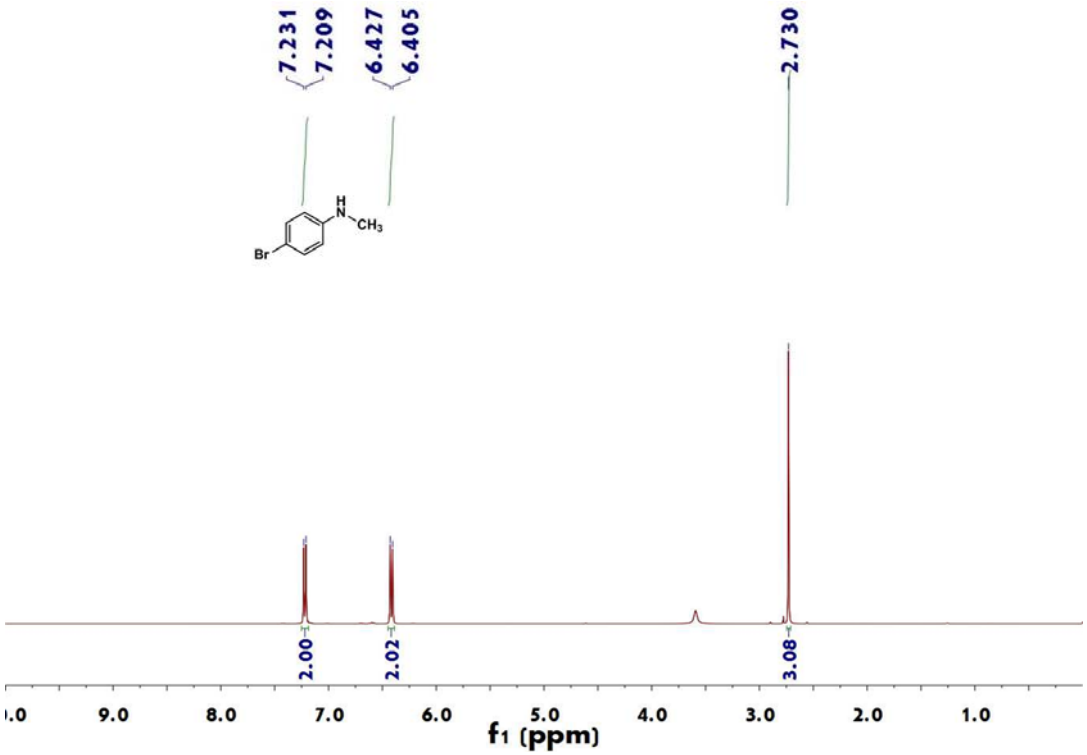

Figure S9. ^{1}H NMR spectrum of 4-bromo-*N*-methylbenzenamine in $CDCl_3$.

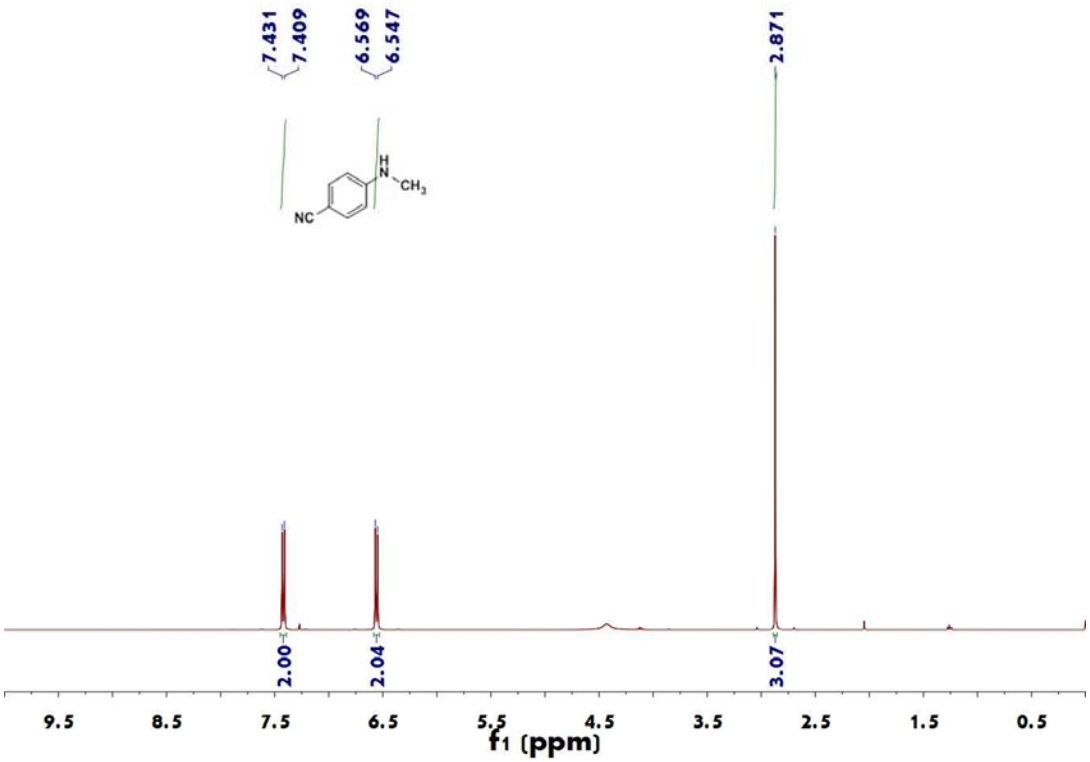

Figure S10. ^{1}H NMR spectrum of 4-(methylamino)benzonitrile in $CDCl_3$.

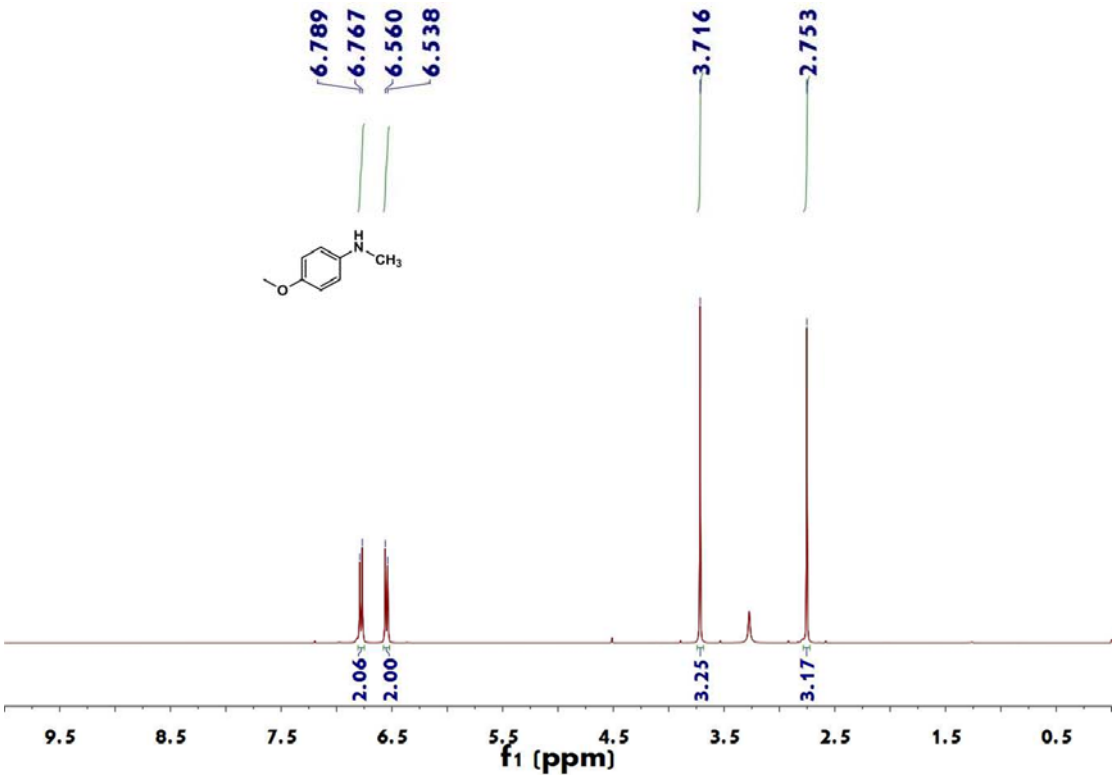

Figure S11. ^{1}H NMR spectrum of 4-methoxyl-*N*-methylbenzenamine in $CDCl_3$.

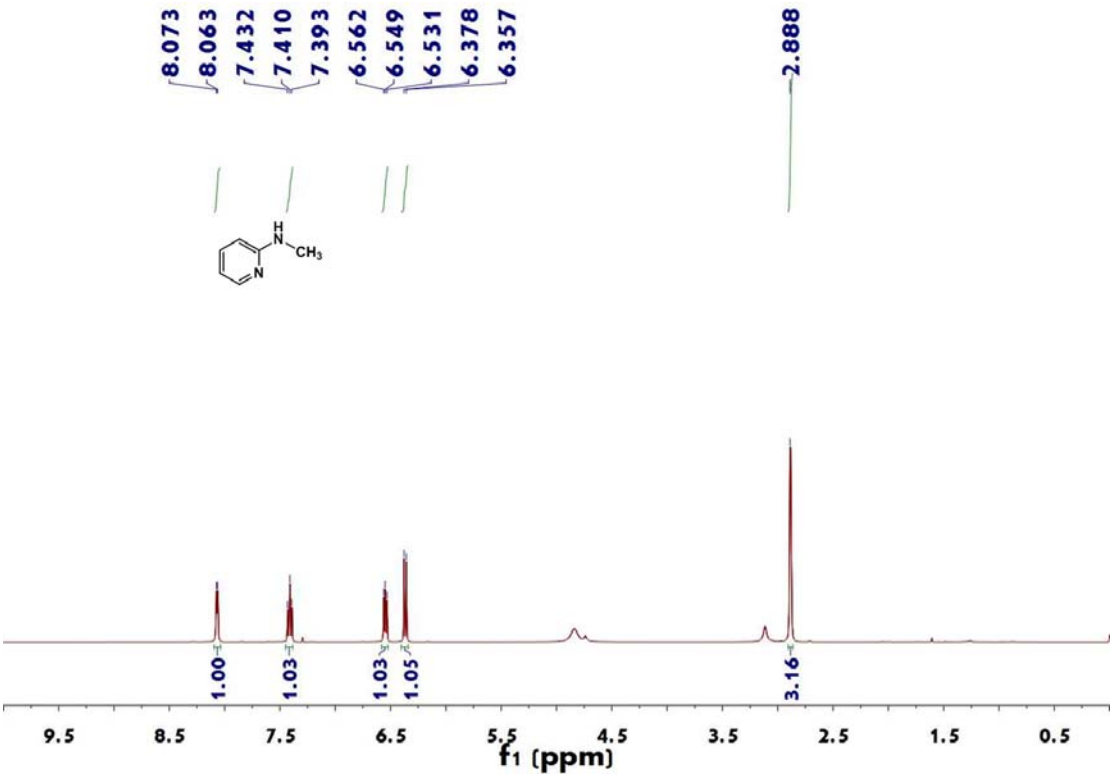

Figure S12. ^{1}H NMR spectrum of *N*-methylpyridin-2-amine in $CDCl_3$.

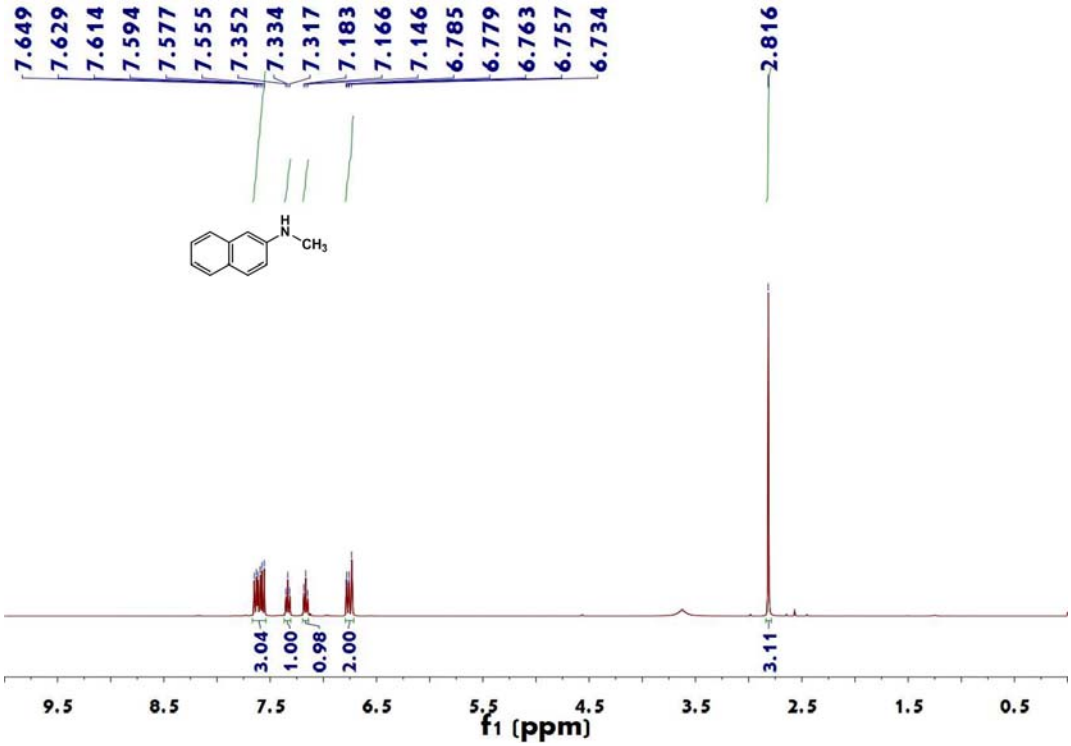

Figure S13. ^{1}H NMR spectrum of *N*-methylnaphthalen-2-amine in $CDCl_3$.

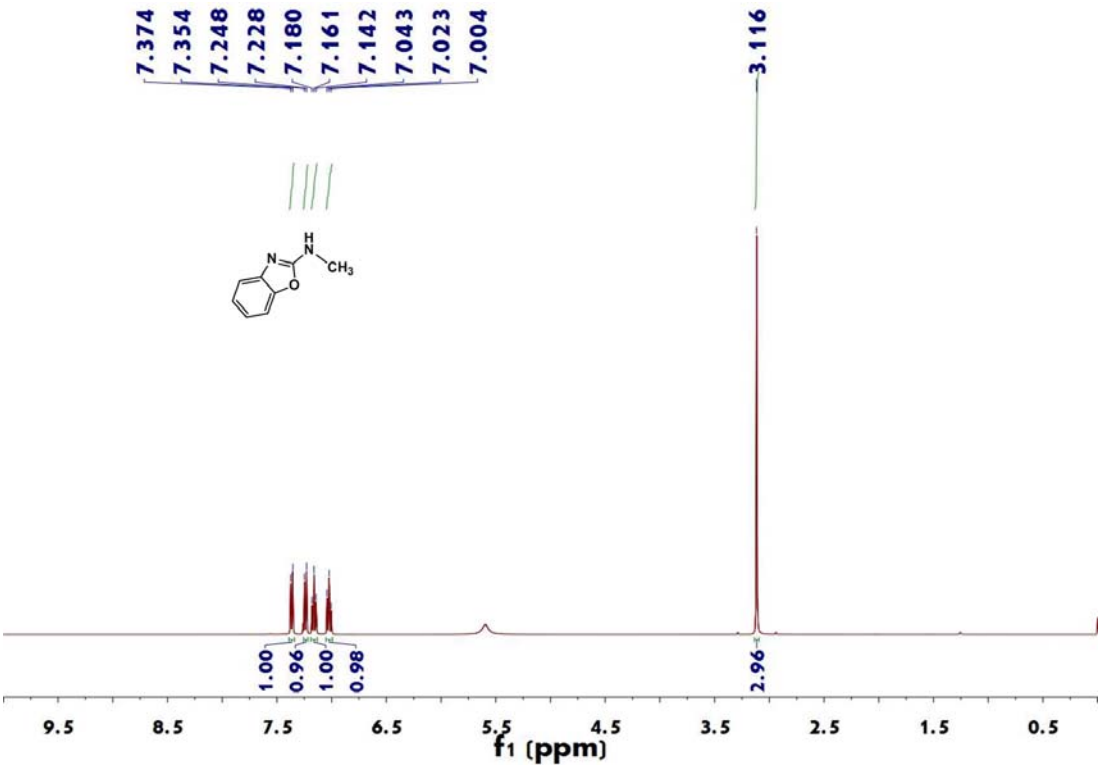

Figure S14. ^{1}H NMR spectrum of *N*-methylbenzo[d]oxazol-2-amine in $CDCl_3$.

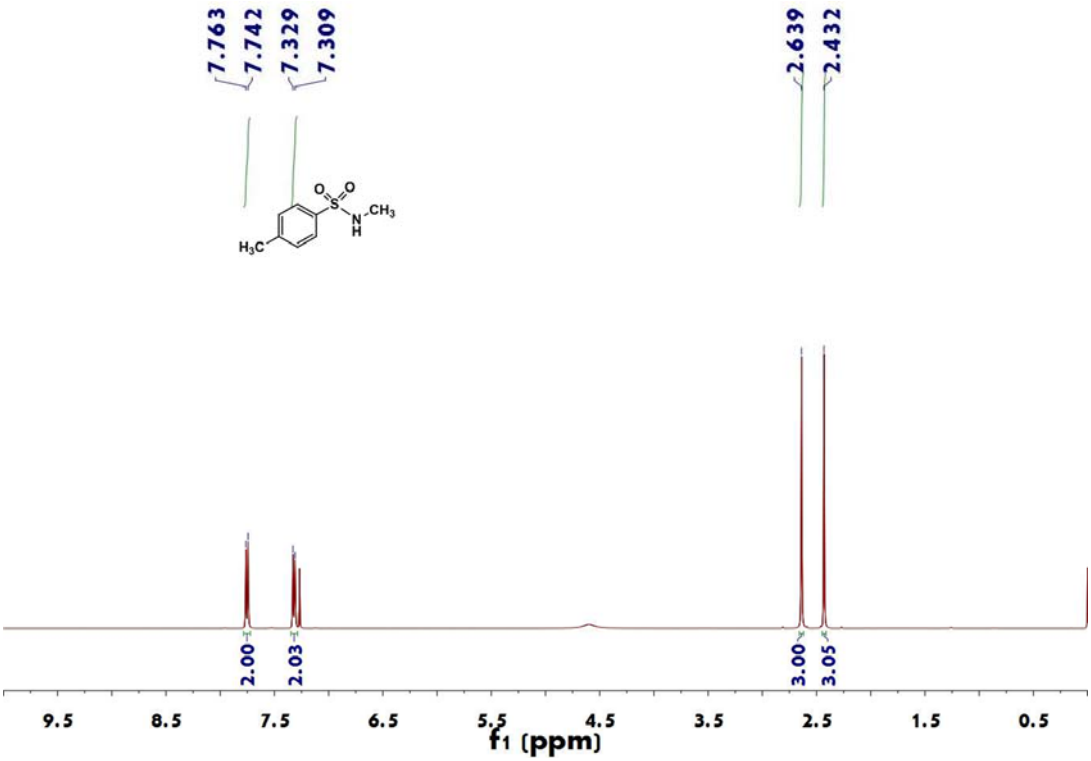

Figure S15. ^{1}H NMR spectrum of *N*,4-dimethylbenzenesulfonamide in $CDCl_3$.

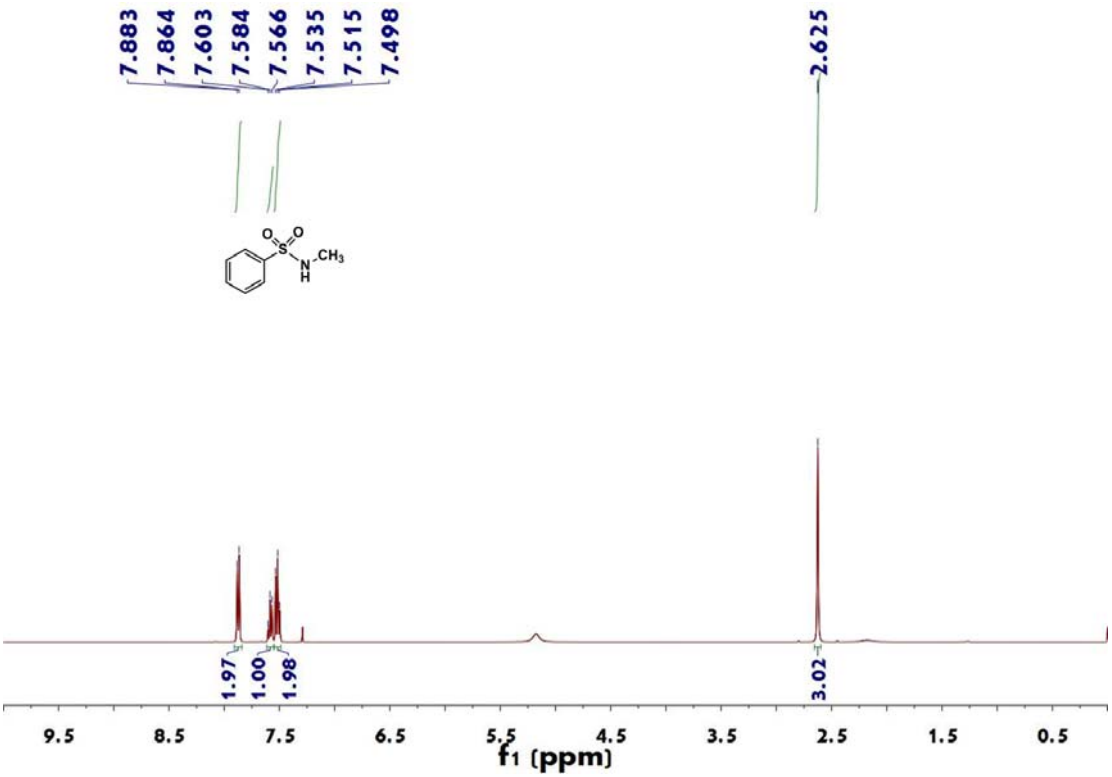

Figure S16. ^{1}H NMR spectrum of *N*-methylbenzenesulfonamide in $CDCl_3$.

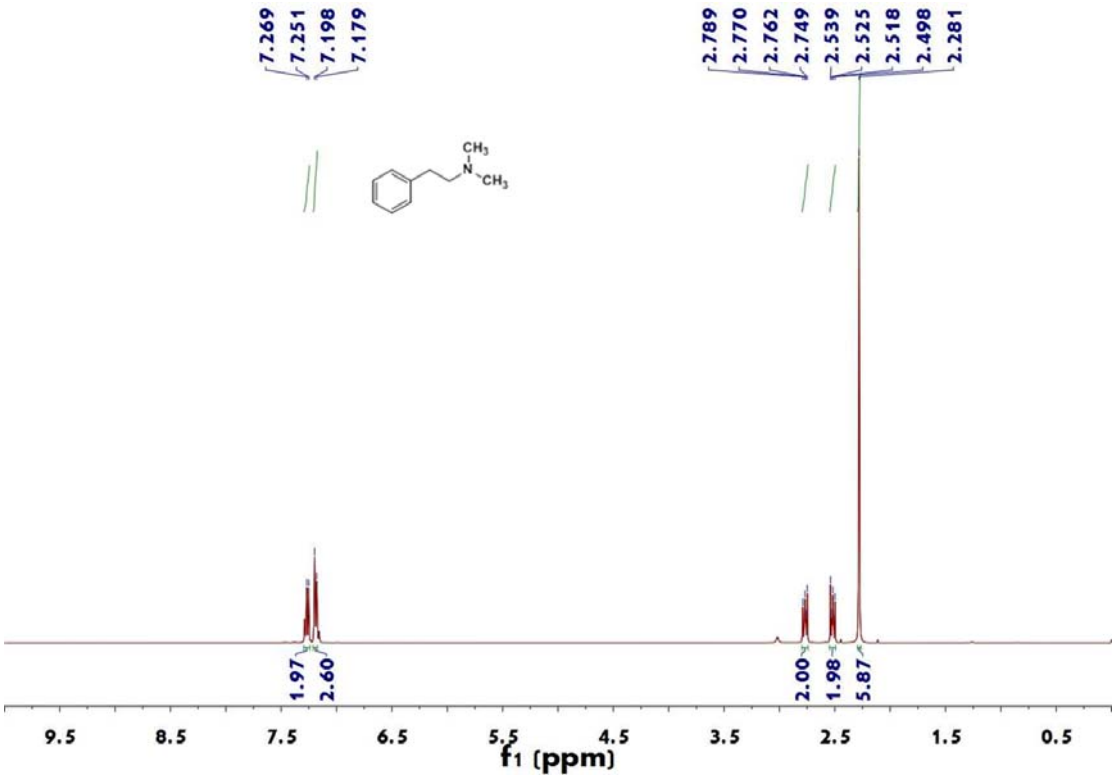

Figure S17. ^{1}H NMR spectrum of *N*,*N*-dimethylbenzeneethanamine in $CDCl_3$.

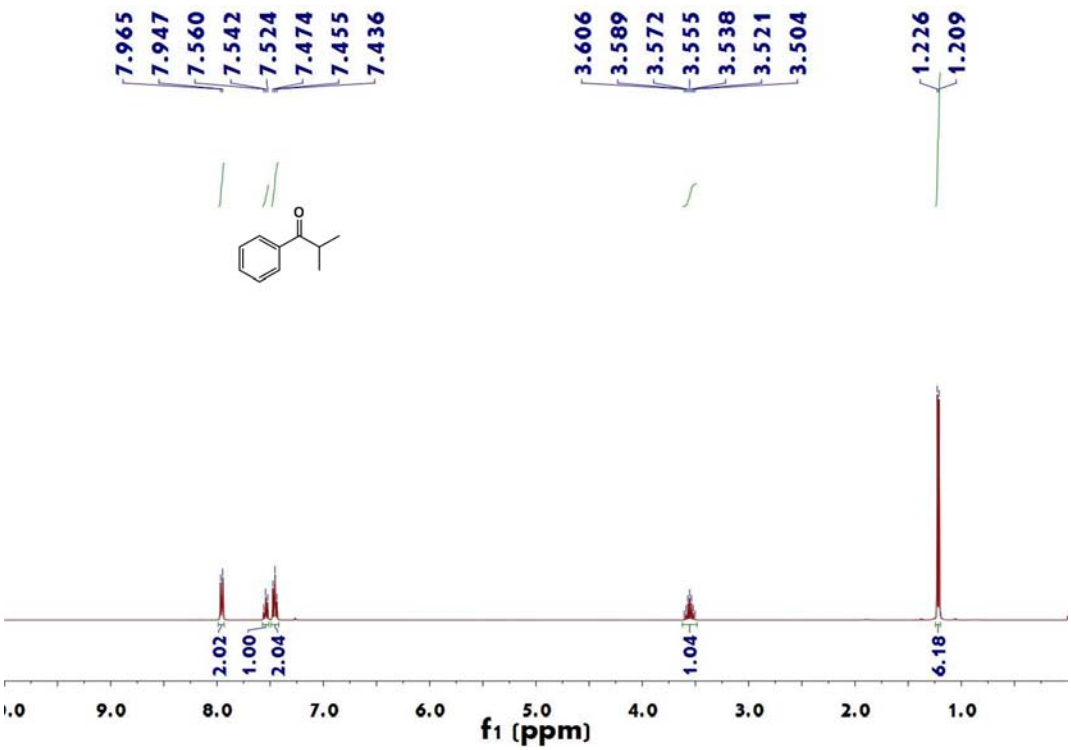

Figure S18. ^{1}H NMR spectrum of 2-methyl-1-phenylpropan-1-one in $CDCl_3$.

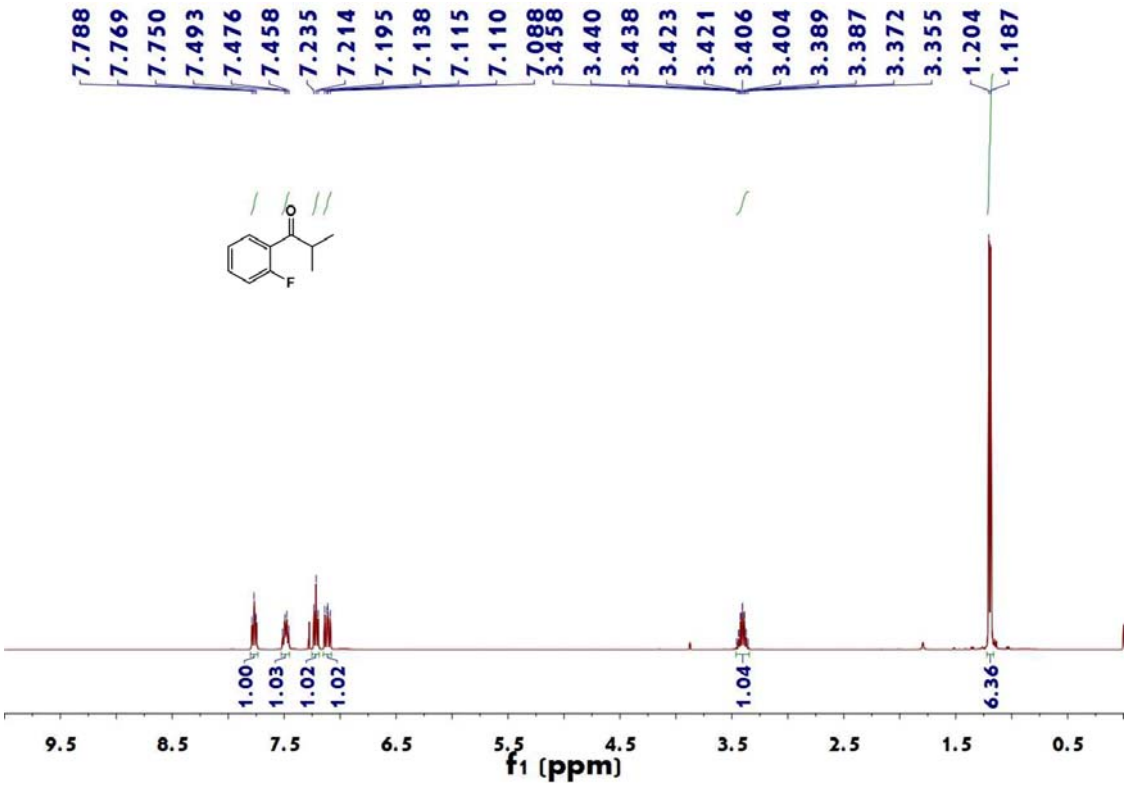

Figure S19. ^{1}H NMR spectrum of 1-(2-fluorophenyl)-2-methylpropan-1-one in $CDCl_3$.

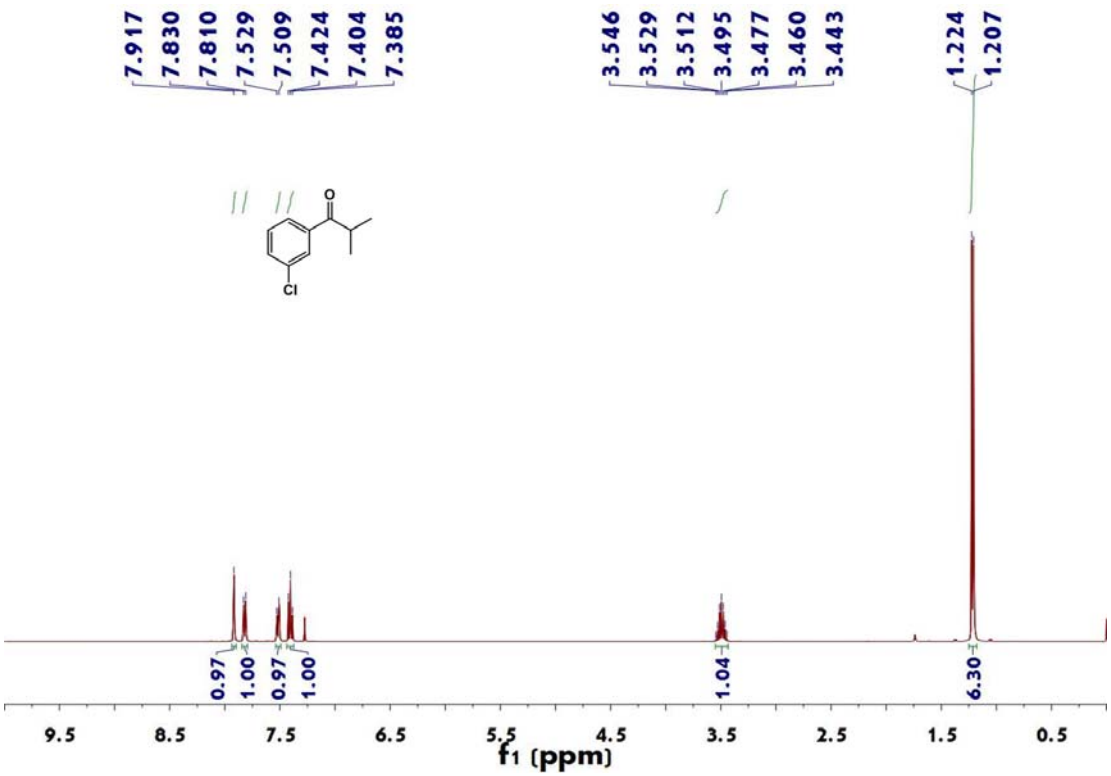

Figure S20. ^{1}H NMR spectrum of 1-(3-chloropheny)-2-methylpropan-1-one in $CDCl_3$.

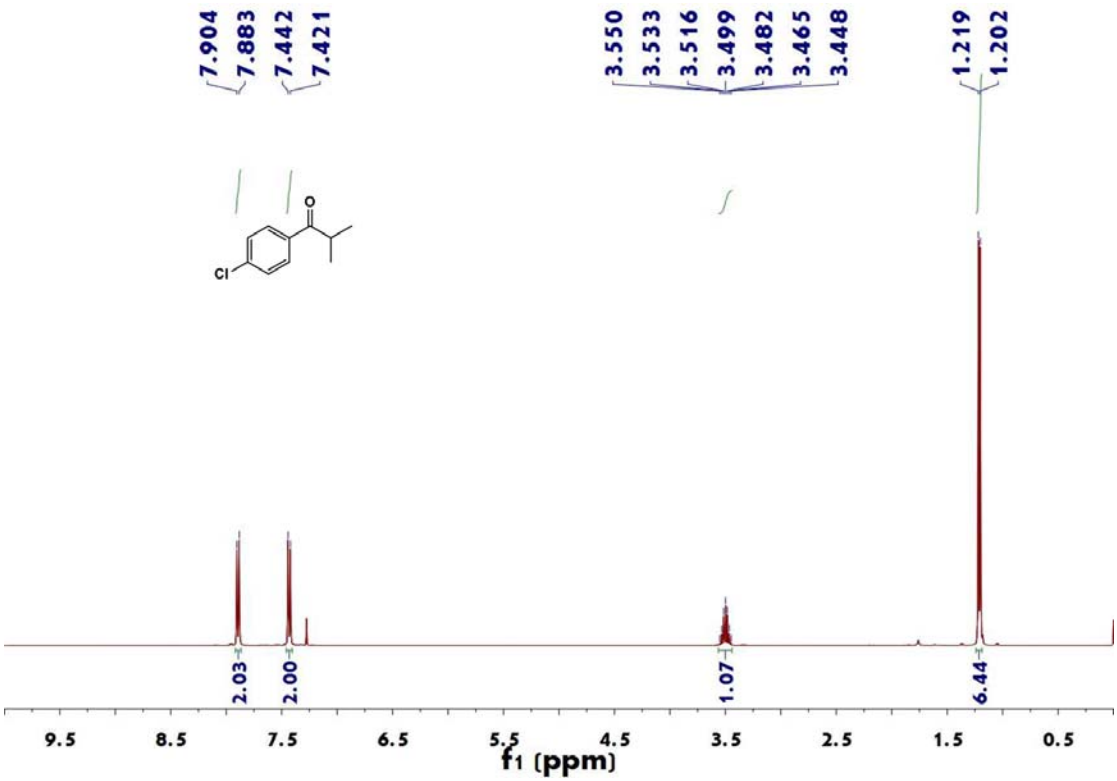

Figure S21. ^{1}H NMR spectrum of 1-(4-chlorophenyl)-2-methylpropan-1-one in $CDCl_3$.

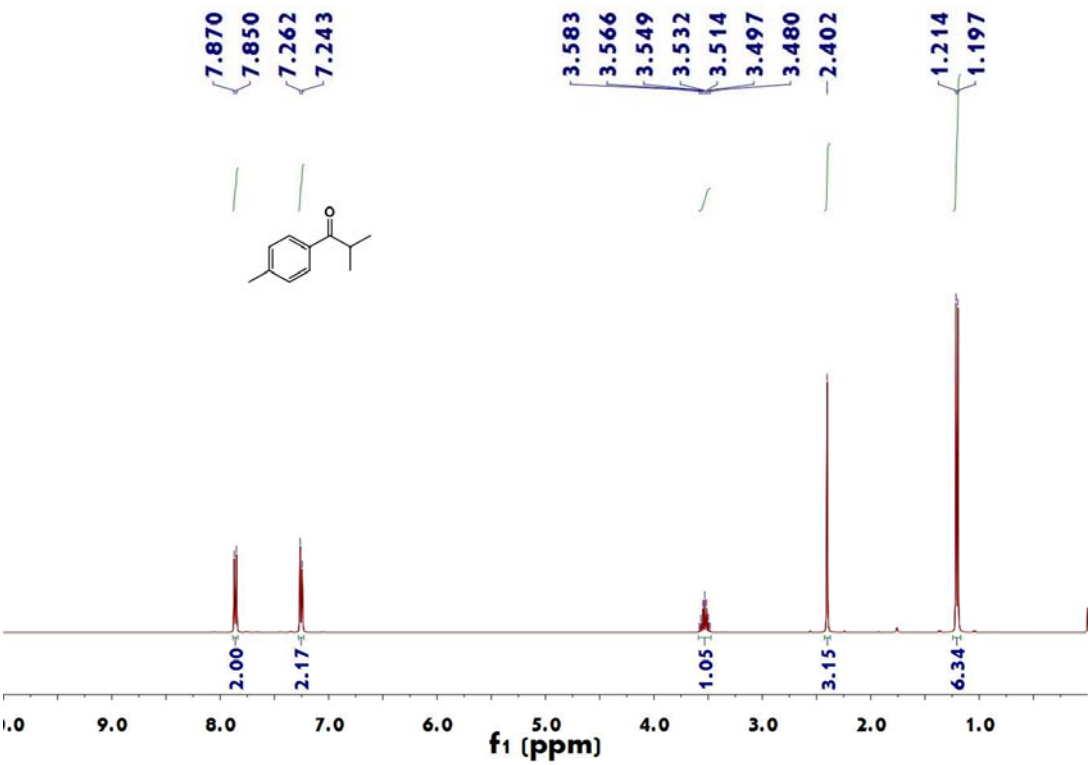

Figure S22. ^{1}H NMR spectrum of 4-methyl-1-(2-methylphenyl)propan-1-one in $CDCl_3$.

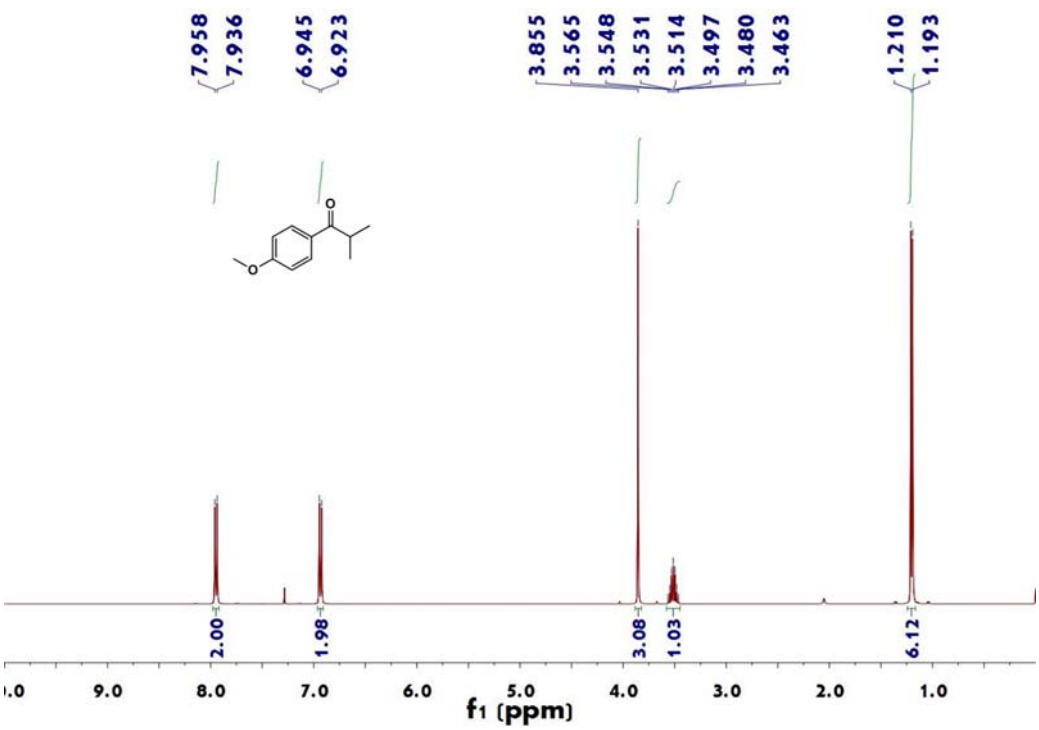

Figure S23. ^{1}H NMR spectrum of 1-(4-methoxyphenyl)-2-methylpropan-1-one in $CDCl_3$.

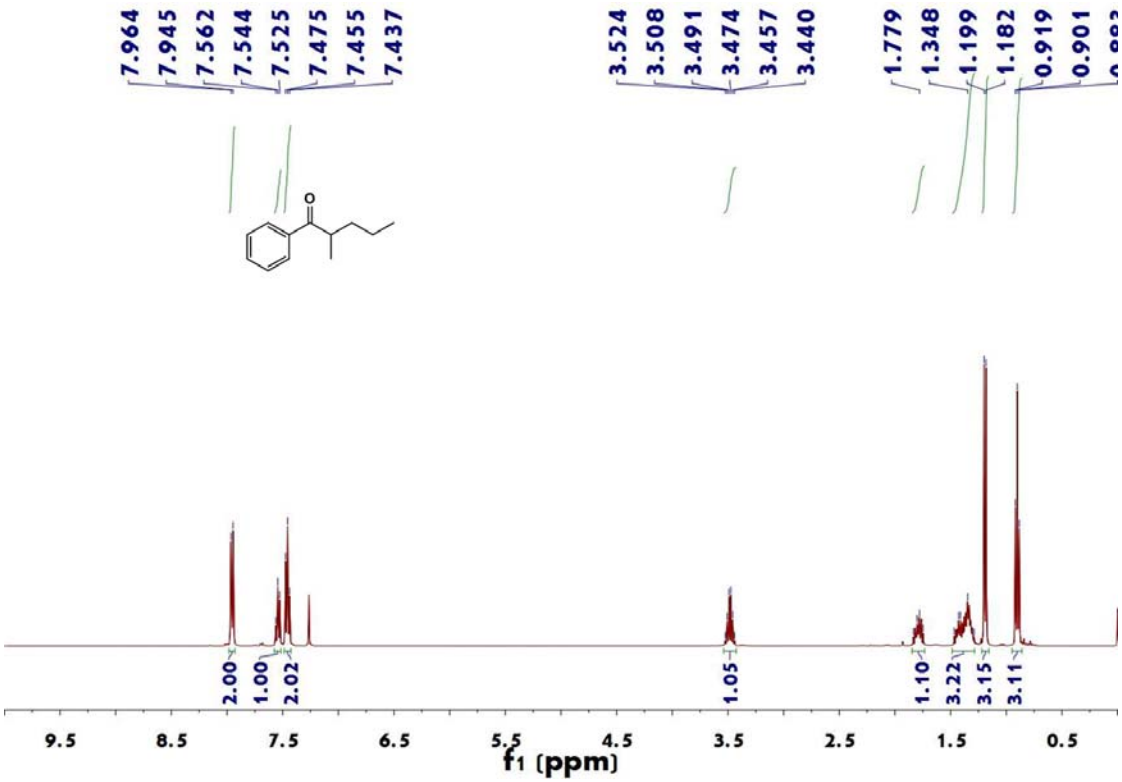

Figure S24. ^{1}H NMR spectrum of 2-methyl-1-phenylpentan-1-one in $CDCl_3$.

^{13}C NMR Spectrum

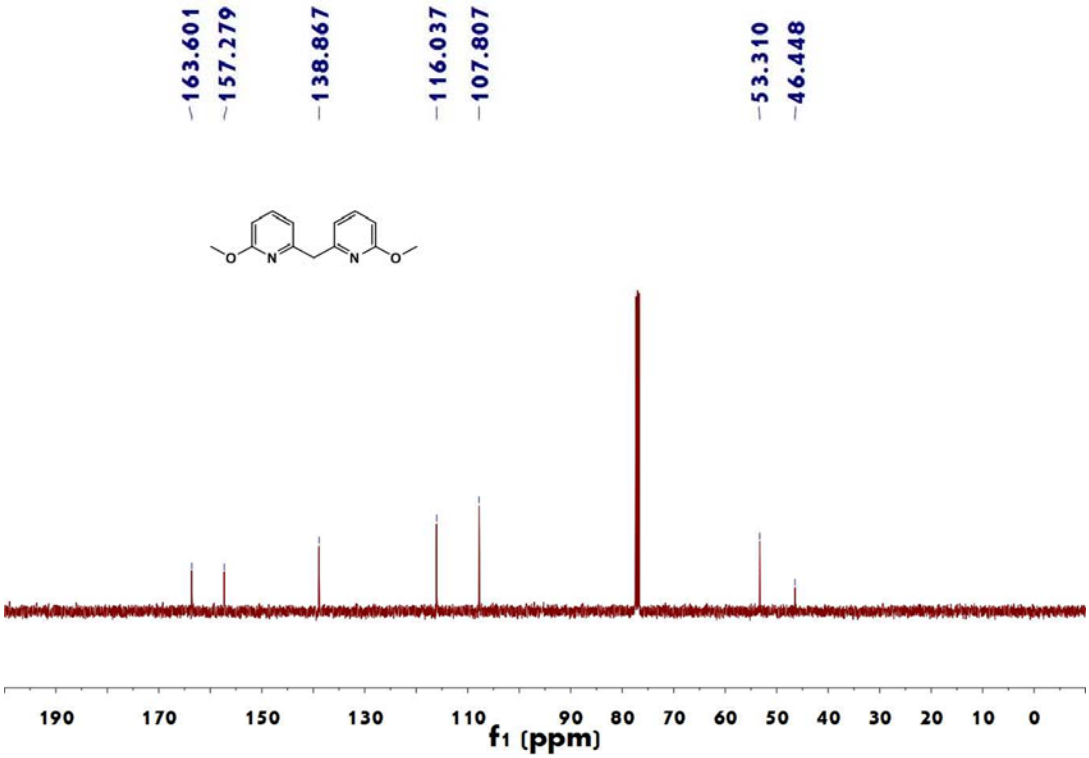

Figure S25. ^{13}C NMR spectrum of 2-methoxy-6-((6-methoxypyridin-2-yl)methyl)pyridine in $CDCl_3$.

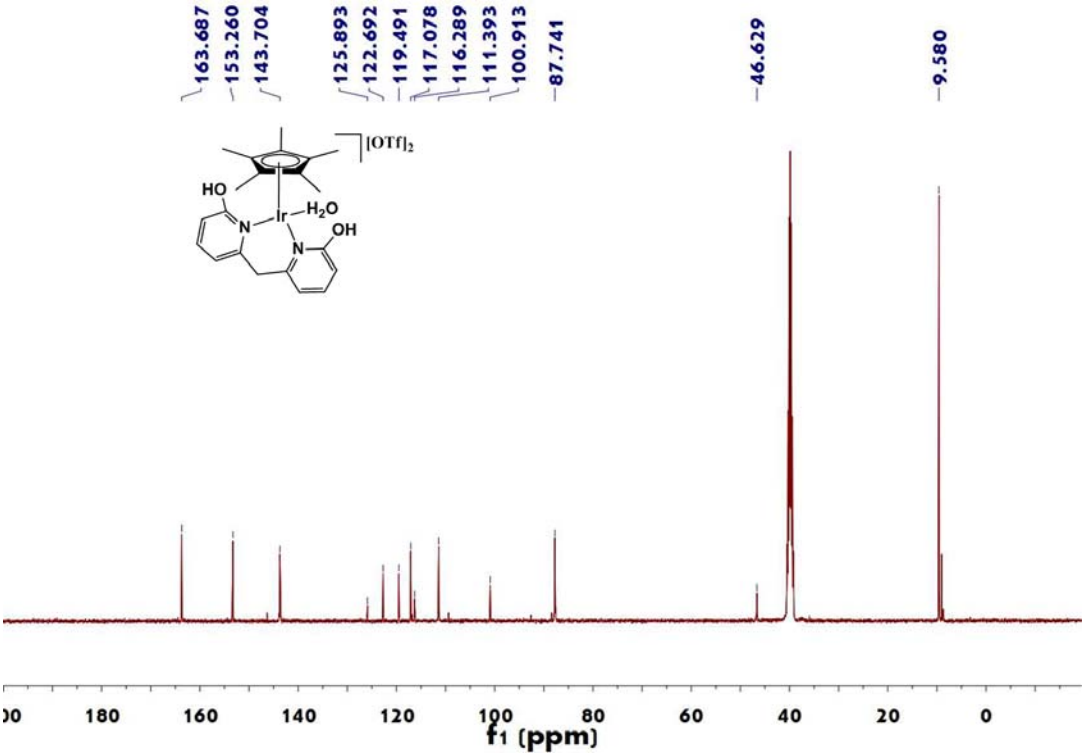

Figure S26. ^{13}C NMR spectrum of complex **2** in d_6-DMSO.

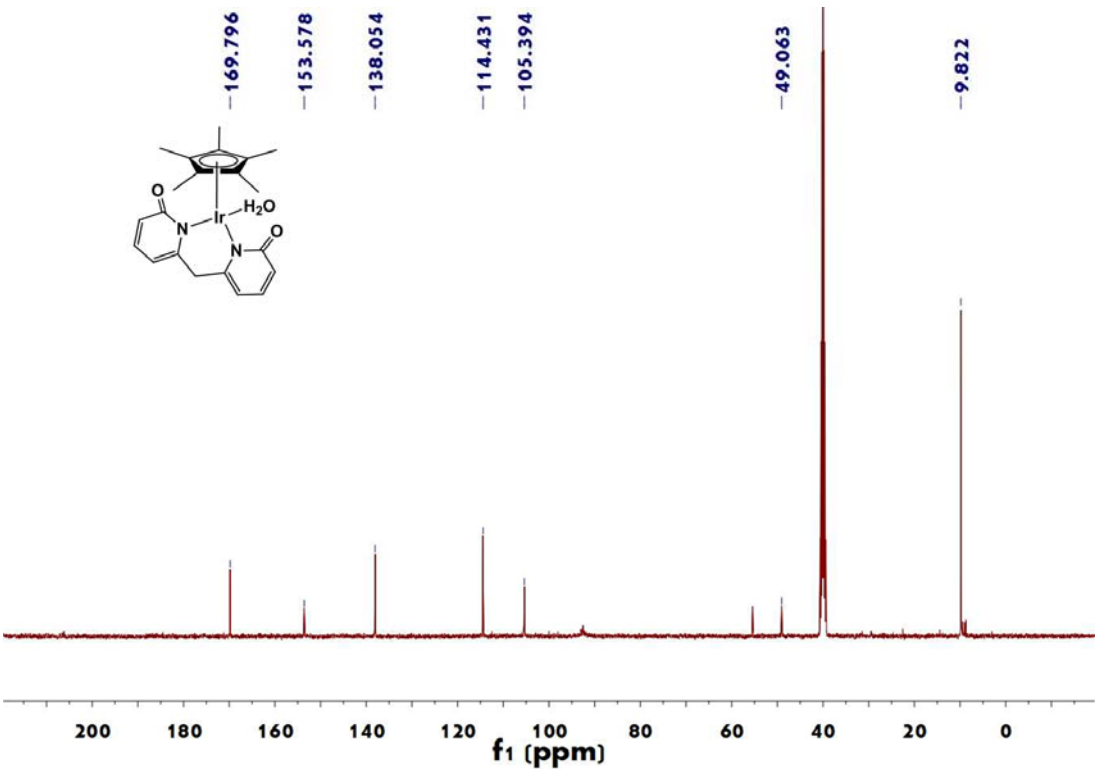

Figure S27. ^{13}C NMR spectrum of complex **3** in d_6-DMSO.