Sulfonated carbon-encapsulated iron nanoparticles as efficient magnetic nanocatalyst for highly selective synthesis of benzimidazoles

Electronic Supplmenetary Information (ESI)

Artur Kasprzak[a]*, Michał Bystrzejewski[b], Magdalena Poplawska[a]

*e-mail: akasprzak@ch.pw.edu.pl

[a] Faculty of Chemistry, Warsaw University of Technology, 00-664 Warsaw, Poland
[b] Faculty of Chemistry, University of Warsaw, 02-093 Warsaw, Poland

Table of contents

1. FT-IR and TGA data for NANOCAT-G4 (Fe@-Ph-SO_3H)

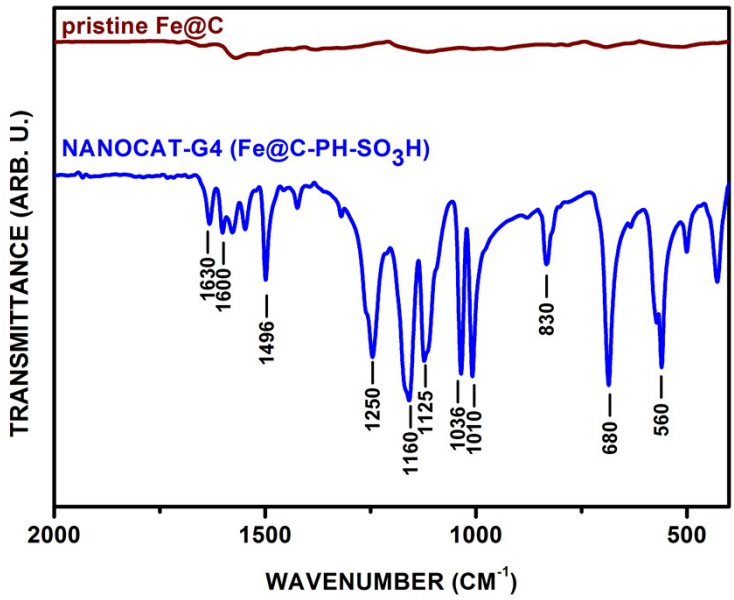

Fig. S1. FT-IR spectrum of **NANOCAT-G4** (Fe@C-Ph-SO_3H). FT-IR spectrum of pristine Fe@C is also presented

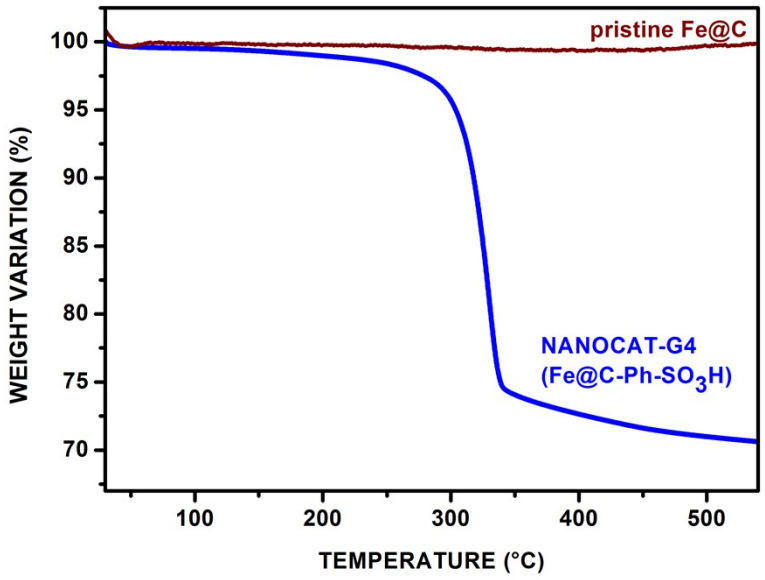

Fig. S2. TGA curve (in nitrogen) of **NANOCAT-G4** (Fe@C-Ph-SO_3H). TGA curve (in nitrogen) of pristine Fe@C is also presented

First weight loss on the TGA curve (Fig. S2) between ca. 50 °C–120 °C, is related to the desorption of moisture. The next weight loss, which is not observed for pristine Fe@C, starts at ca. 150 °C and is completed at ca. 500 °C, which is clearly attributed to the decomposition of covalently attached moieties. Content of introduced organic moiety for **NANOCAT-G4** was calculated as follows: $C_F = (WL_{500}\text{-}M)$, where C_F is the content of introduced organic moiety [wt%], WL_{500} is the observed weight loss up to 500°C, M is a content of moisture in the sample (weight loss up to 120°C).[1–3]

2. NMR and FT-IR for the benzimidazoles obtained (1-13)

The spectral data for the obtained benzimidazoles are consistent with the literature. The references are given in the figure description.

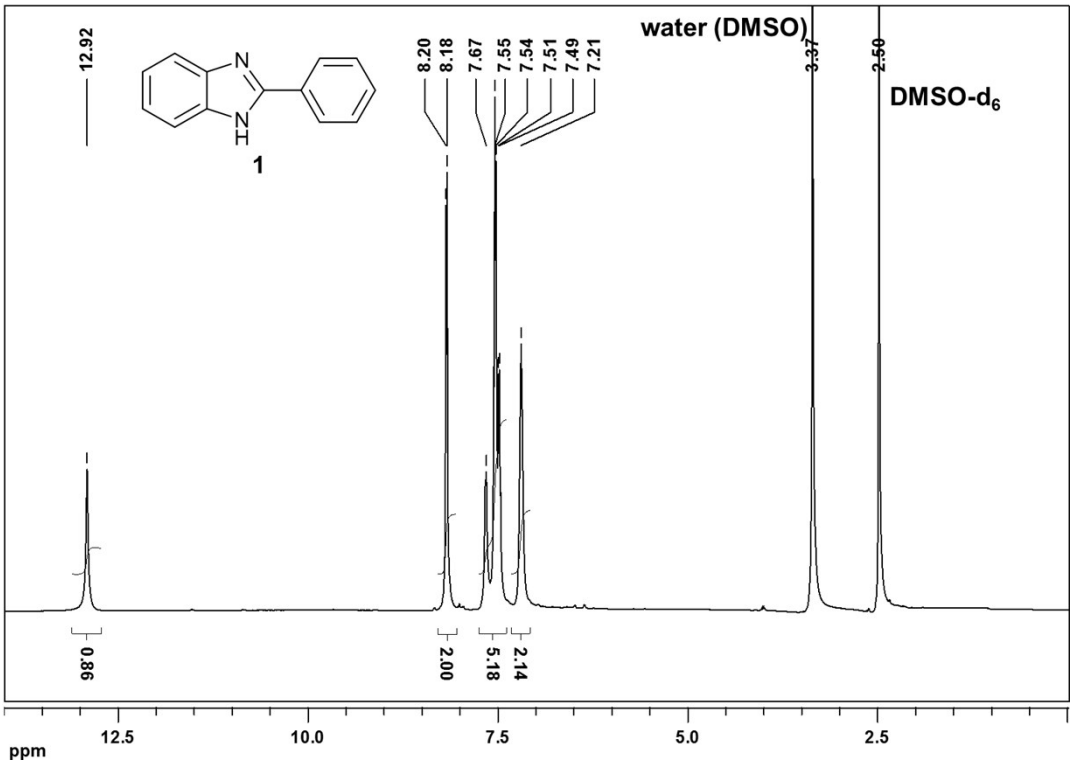

Fig. S3. ^{1}H NMR spectrum (500 MHz, DMSO-d_6) of 2-phenylbenzimidazole (**1**)[4]

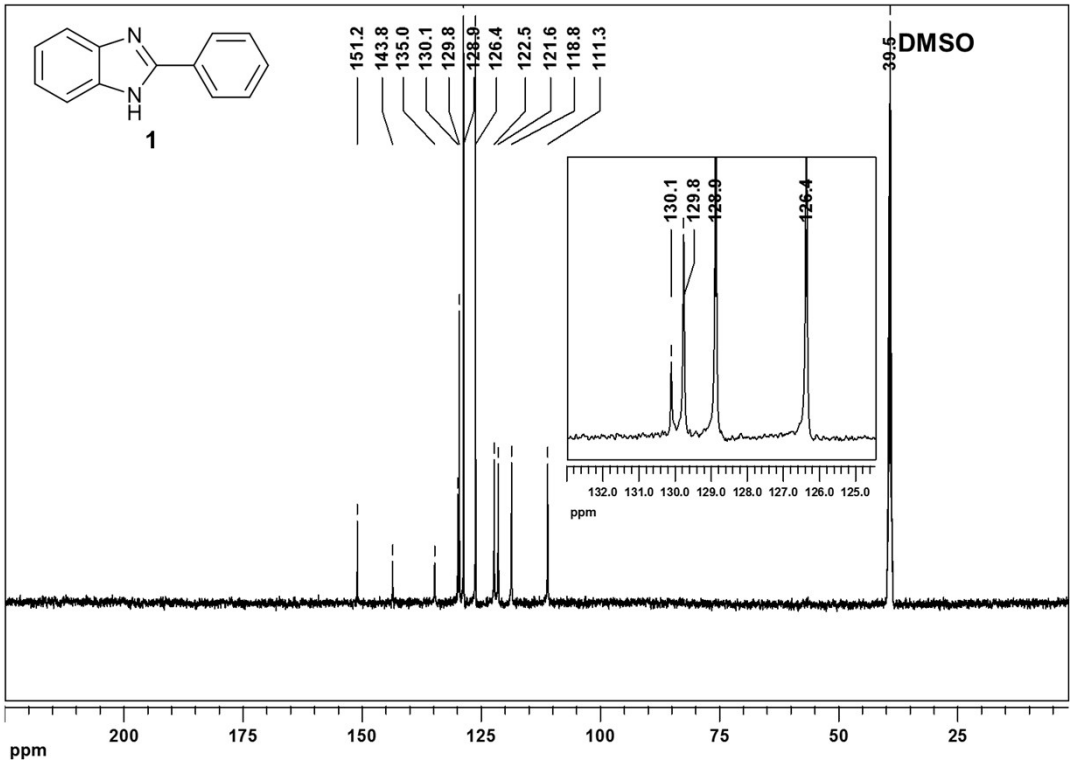

Fig. S4. ^{13}C NMR spectrum (125 MHz, DMSO-d_6) of 2-phenylbenzimidazole (**1**)[4]

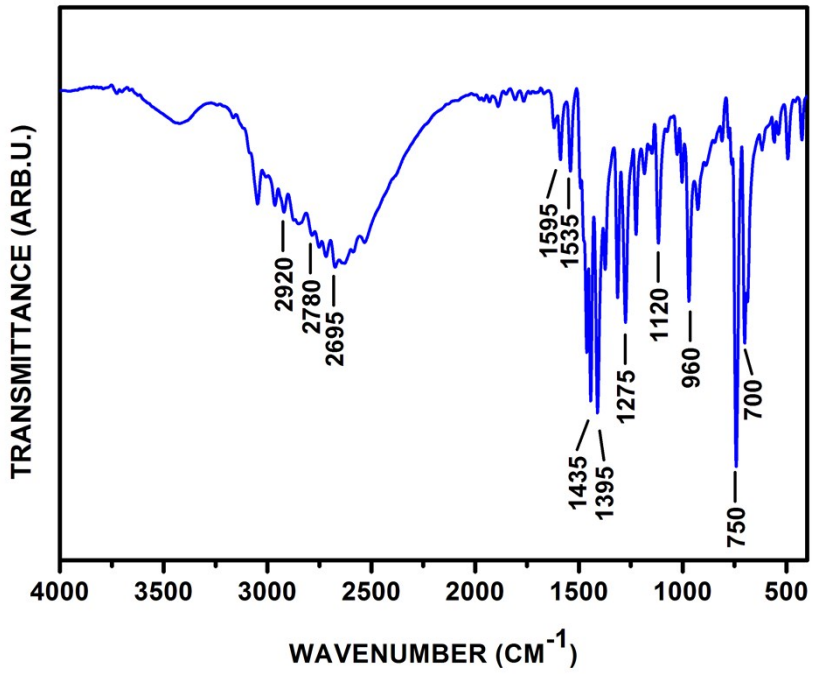

Fig. S5. FT-IR spectrum of 2-phenylbenzimidazole (**1**)[5]

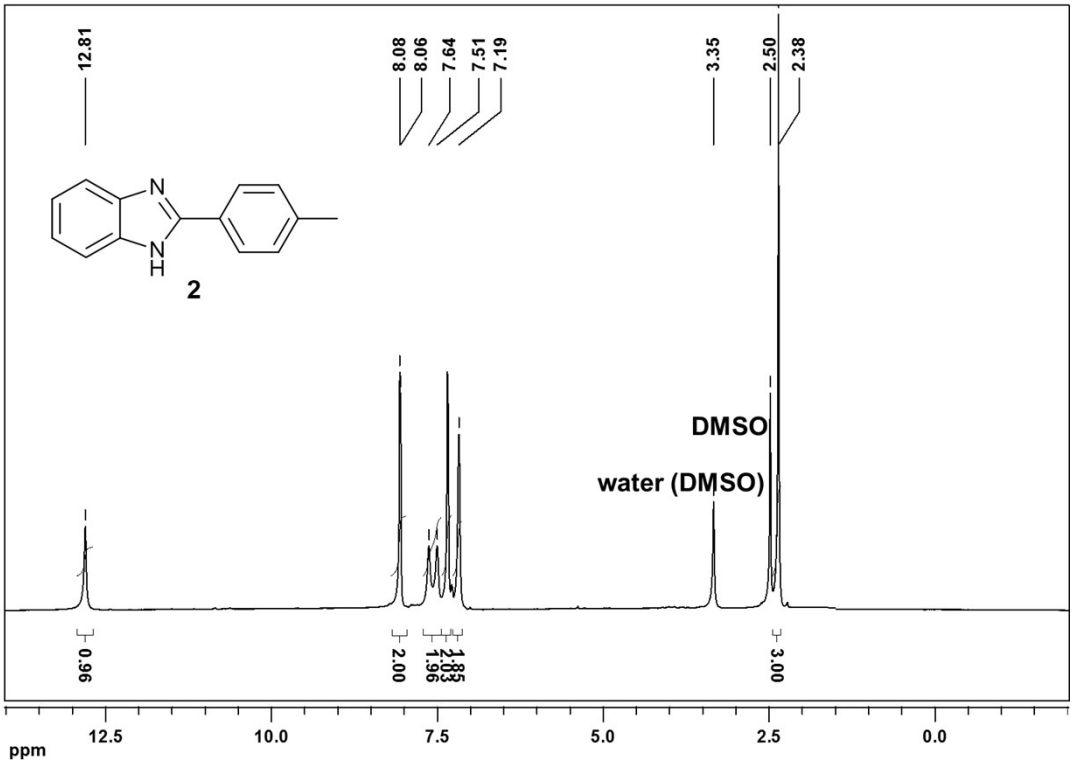

Fig. S6. ^{1}H NMR spectrum (500 MHz, DMSO-d_6) of 2-(4-tolyl)benzimidazole (**2**)[4]

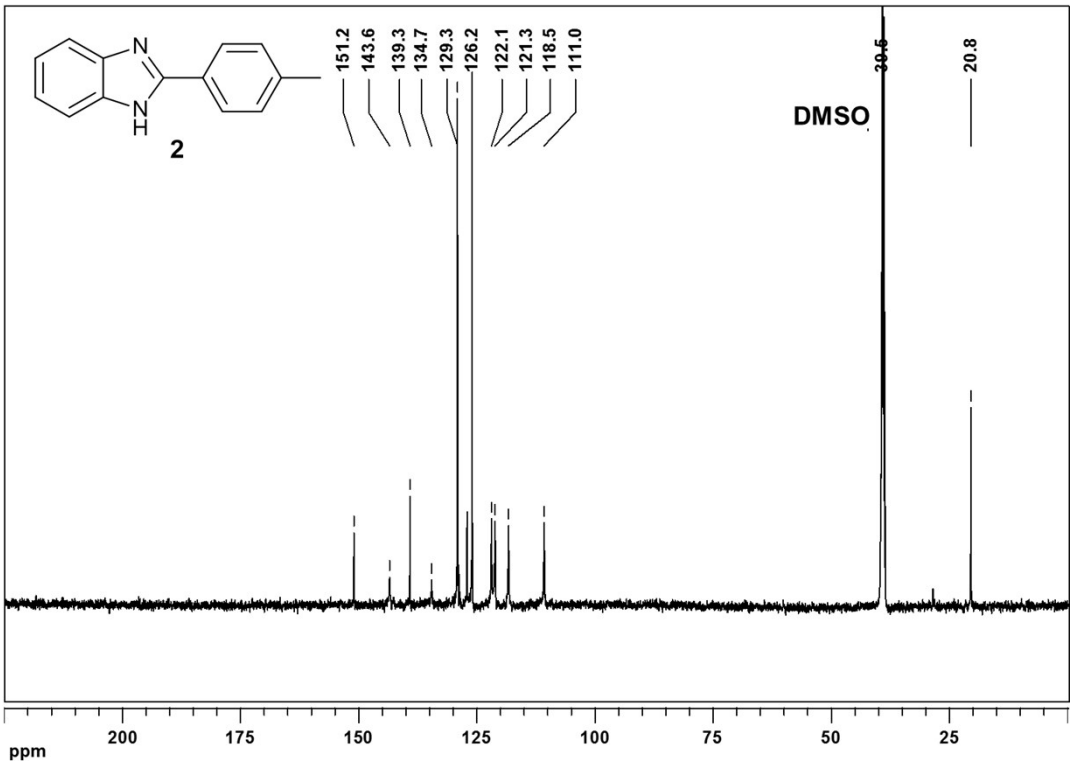

Fig. S7. ^{13}C NMR spectrum (125 MHz, DMSO-d_6) of 2-(4-tolyl)benzimidazole (**2**)[4]

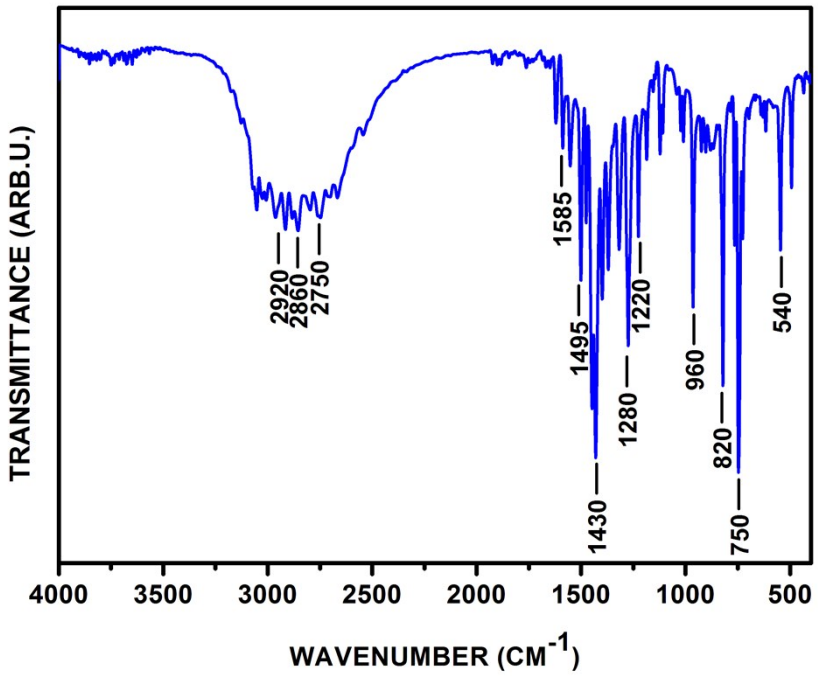

Fig. S8. FT-IR spectrum of 2-(4-tolyl)benzimidazole (**2**)[6]

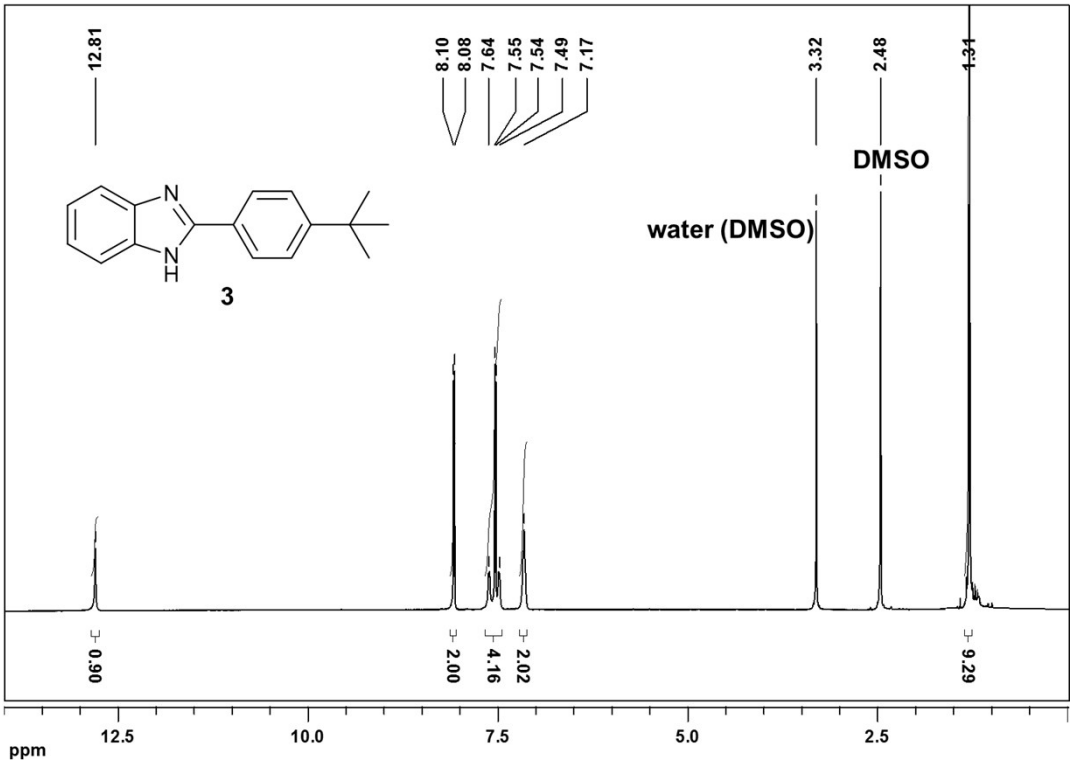

Fig. S9. ^{1}H NMR spectrum (500 MHz, DMSO-d_6) of 2-(4-tert-butylphenyl)benzimidazole (**3**)[4]

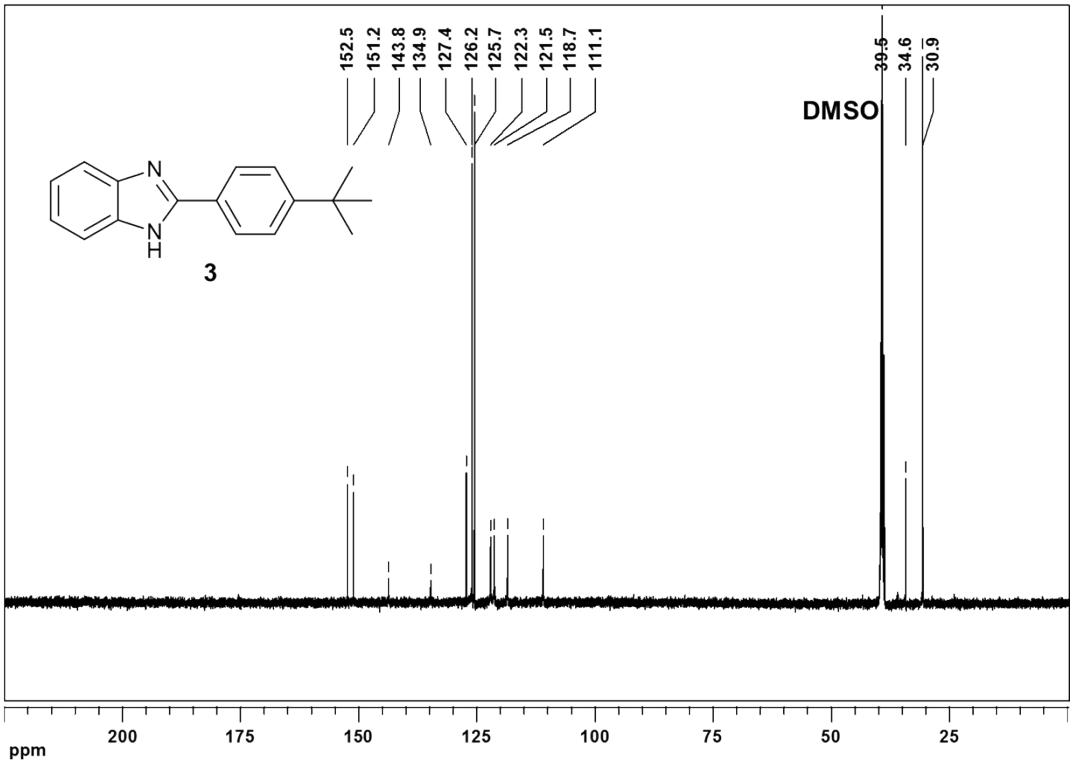

Fig. S10. ^{13}C NMR spectrum (125 MHz, DMSO-d_6) of 2-(4-tert-butylphenyl)benzimidazole (**3**)[4]

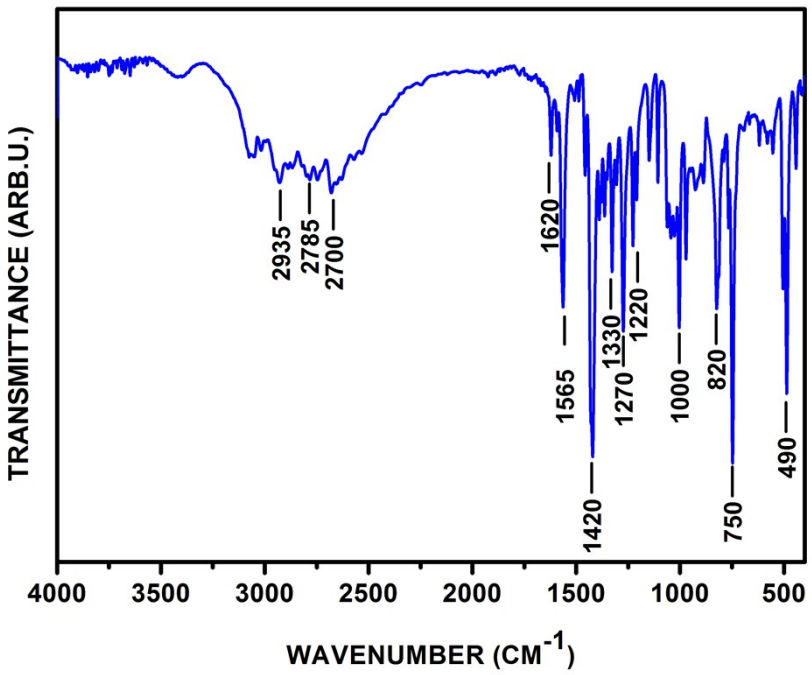

Fig. S11. FT-IR spectrum of 2-(4-tert-butylphenyl)benzimidazole (**3**)[6]

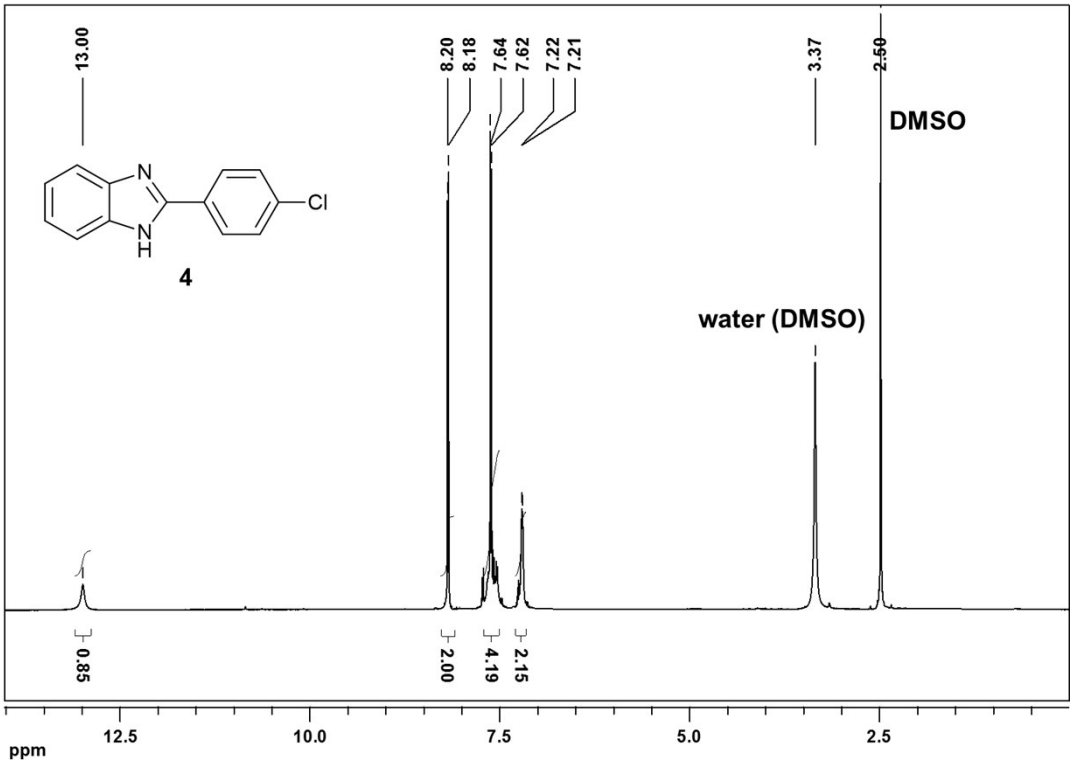

Fig. S12. ^{1}H NMR spectrum (500 MHz, DMSO-d_6) of 2-(4-chlorophenyl)benzimidazole (**4**)[4]

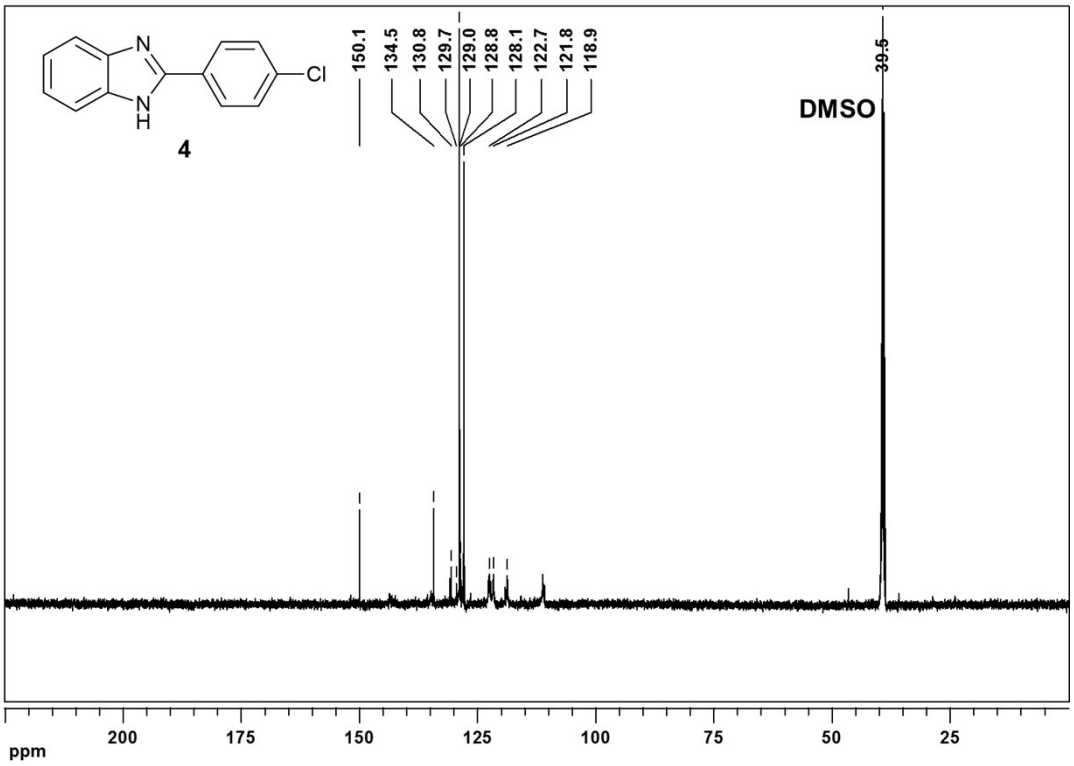

Fig. S13. ^{13}C NMR spectrum (125 MHz, DMSO-d_6) of 2-(4-chlorophenyl)benzimidazole (**4**)[4]

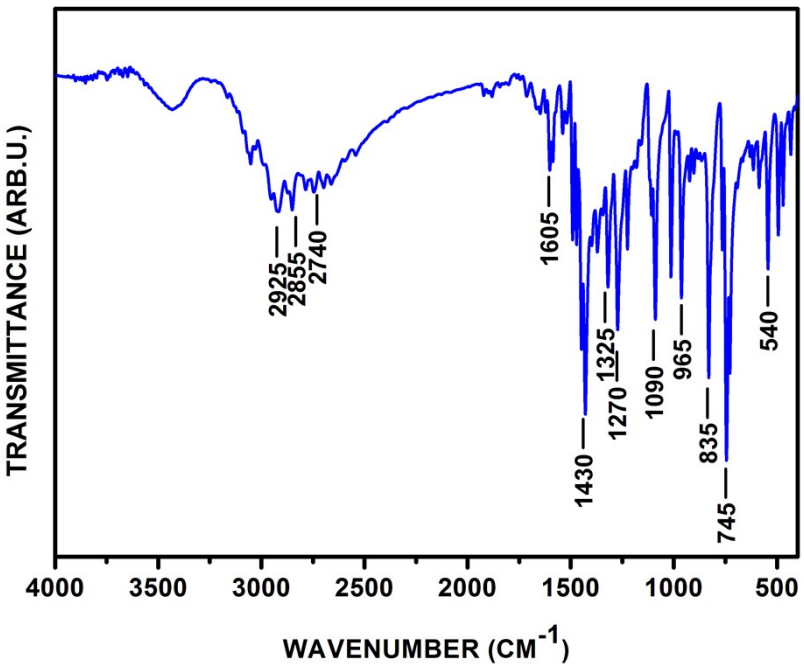

Fig. S14. FT-IR spectrum of 2-(4-chlorophenyl)benzimidazole (**4**)[7]

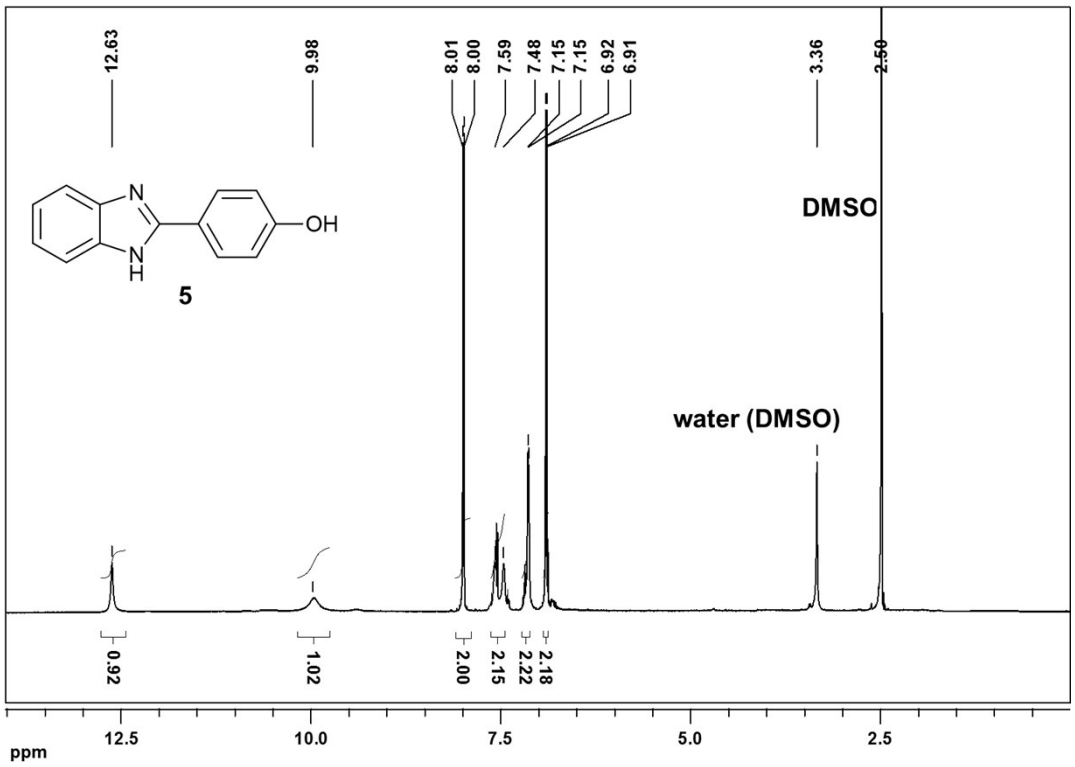

Fig. S15. ^{1}H NMR spectrum (500 MHz, DMSO-d_6) of 2-(4-hydroxyphenyl)benzimidazole (**5**)[8]

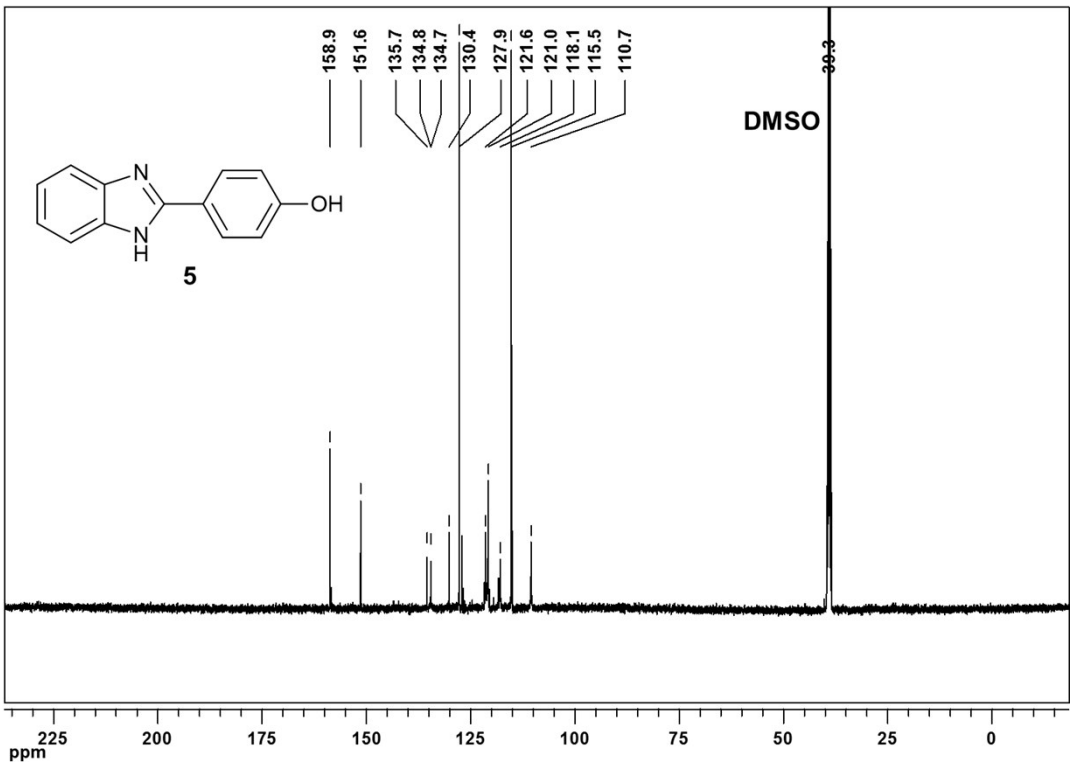

Fig. S16. ^{13}C NMR spectrum (125 MHz, DMSO-d_6) of 2-(4-hydroxyphenyl)benzimidazole (**5**)[8]

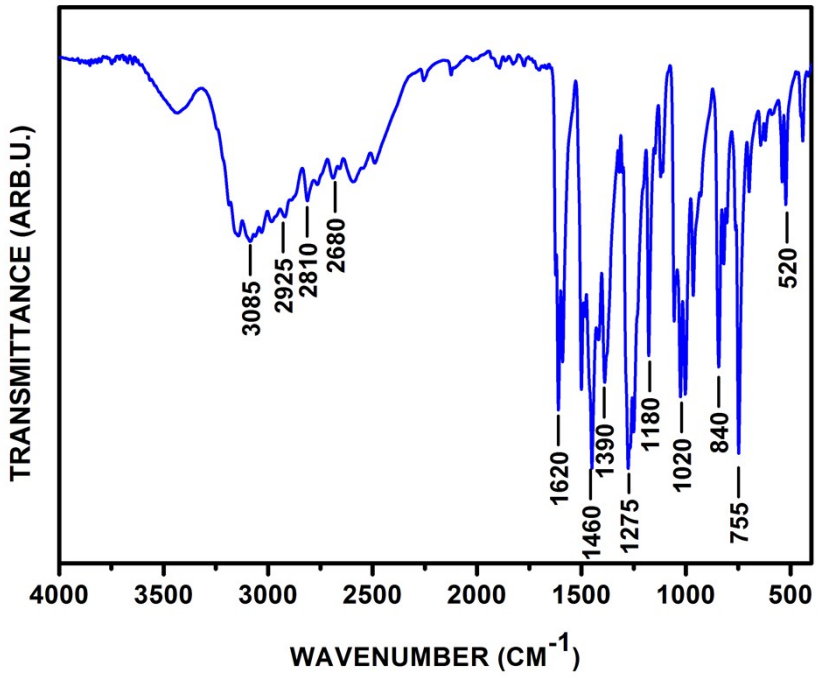

Fig. S17. FT-IR spectrum of 2-(4-hydroxyphenyl)benzimidazole (**5**)[8]

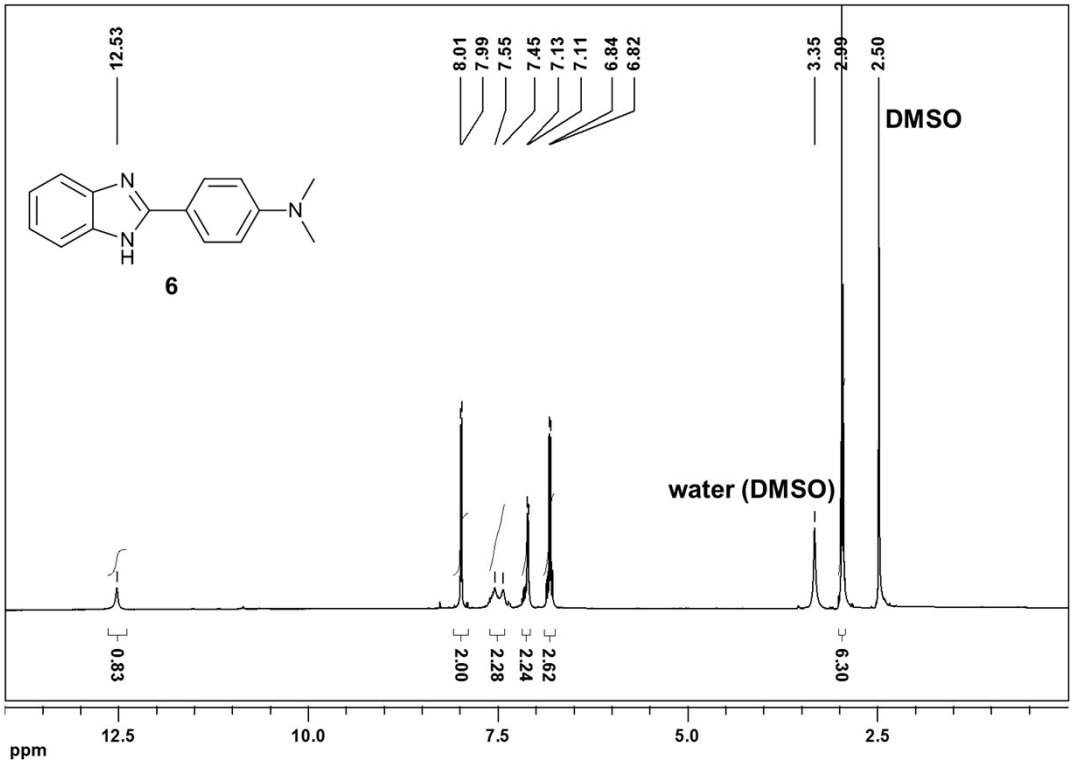

Fig. S18. ^{1}H NMR spectrum (500 MHz, DMSO-d_6) of 2-(4-dimethylaminophenyl)benzimidazole (**6**)[7]

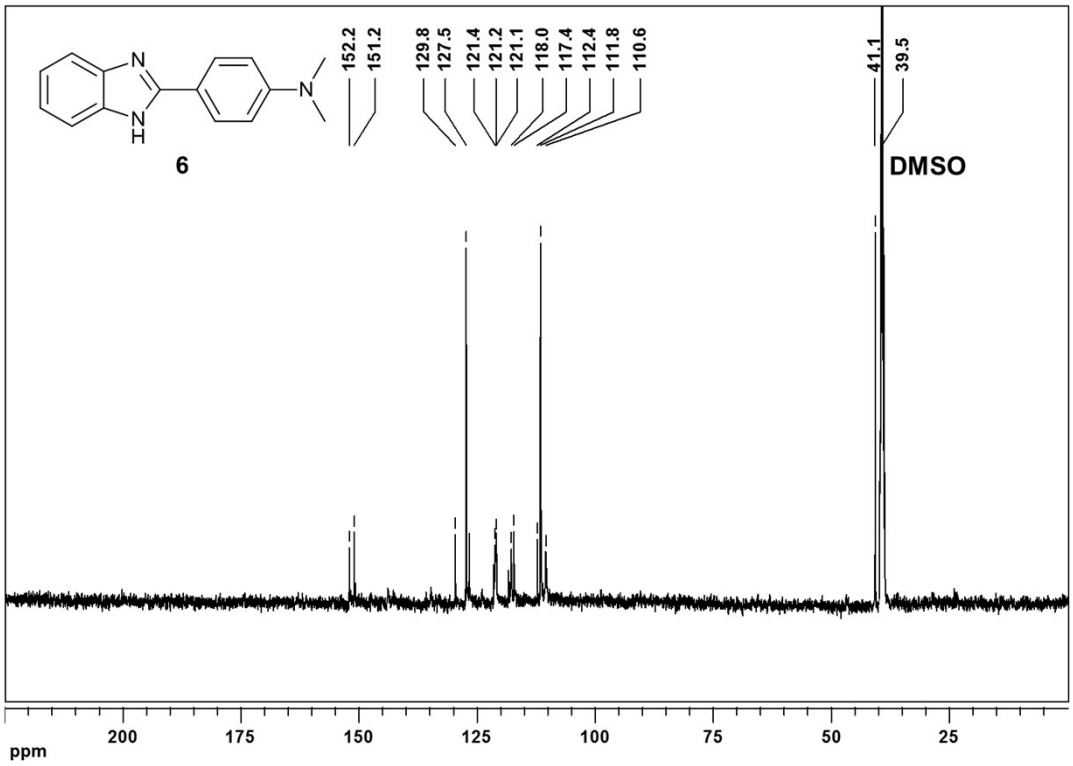

Fig. S19. ^{13}C NMR spectrum (125 MHz, DMSO-d_6) of 2-(4-dimethylaminophenyl)benzimidazole (**6**)[7]

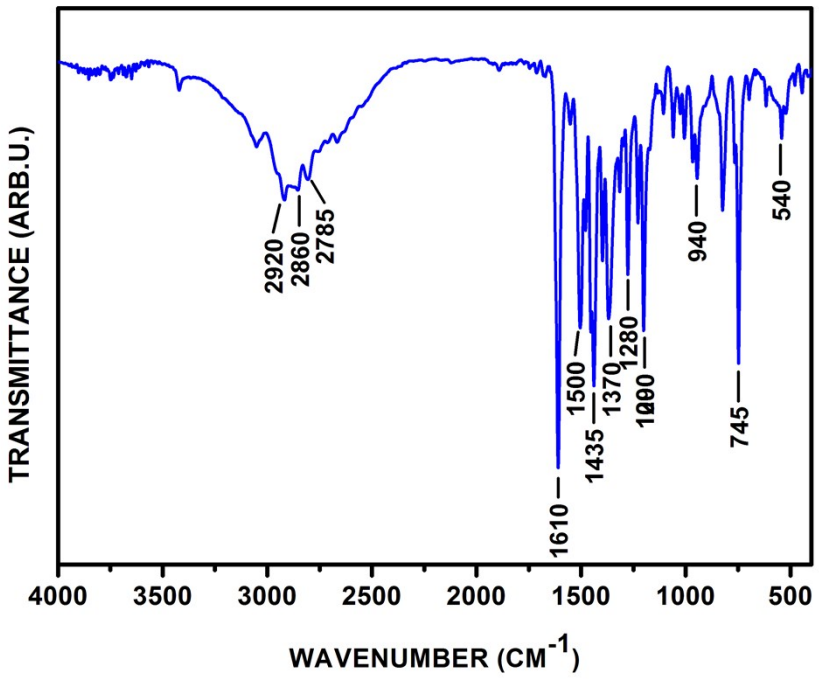

Fig. S20. FT-IR spectrum of 2-(4-dimethylaminophenyl)benzimidazole (**6**)[7]

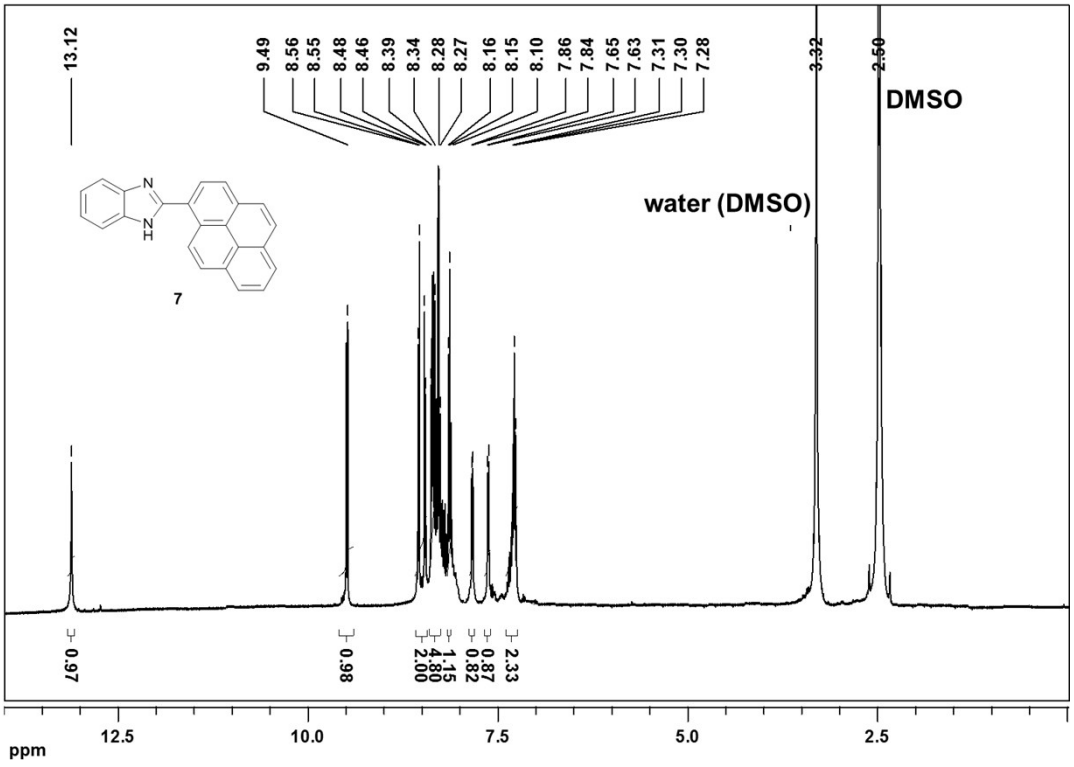

Fig. S21. ^{1}H NMR spectrum (500 MHz, DMSO-d_6) of 2-(1-pyrenyl)benzimidazole (**7**)[9]

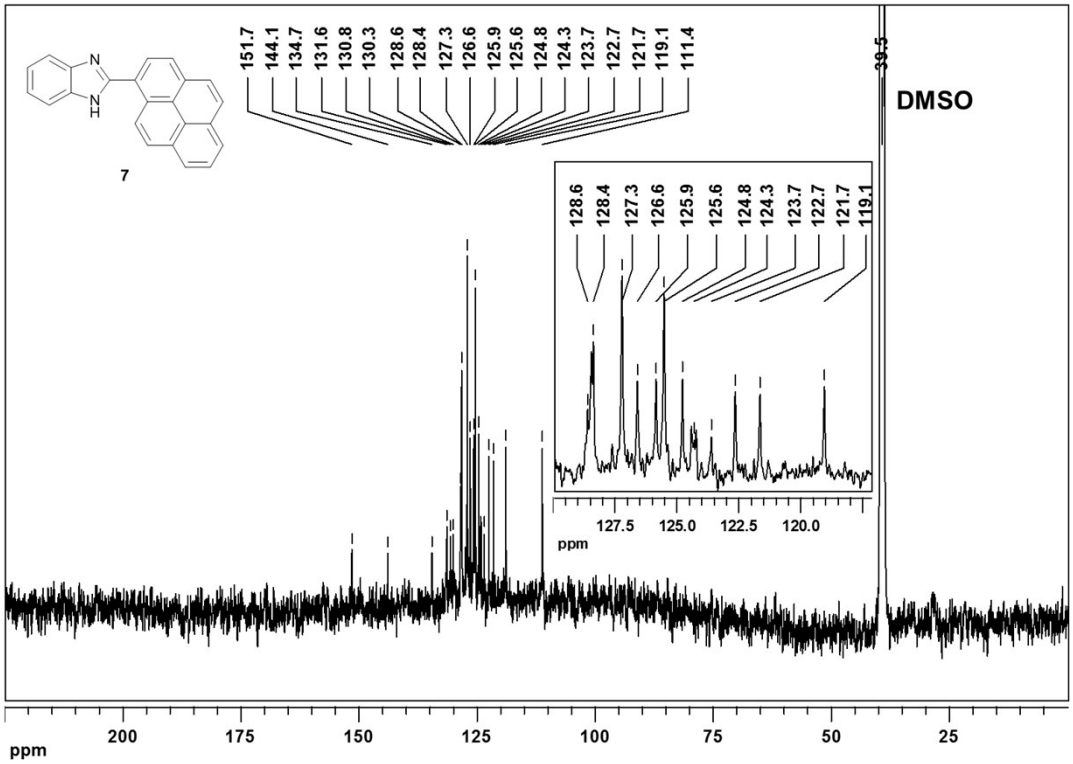

Fig. S22. ^{13}C NMR spectrum (125 MHz, DMSO-d_6) of 2-(1-pyrenyl)benzimidazole (**7**)[9]

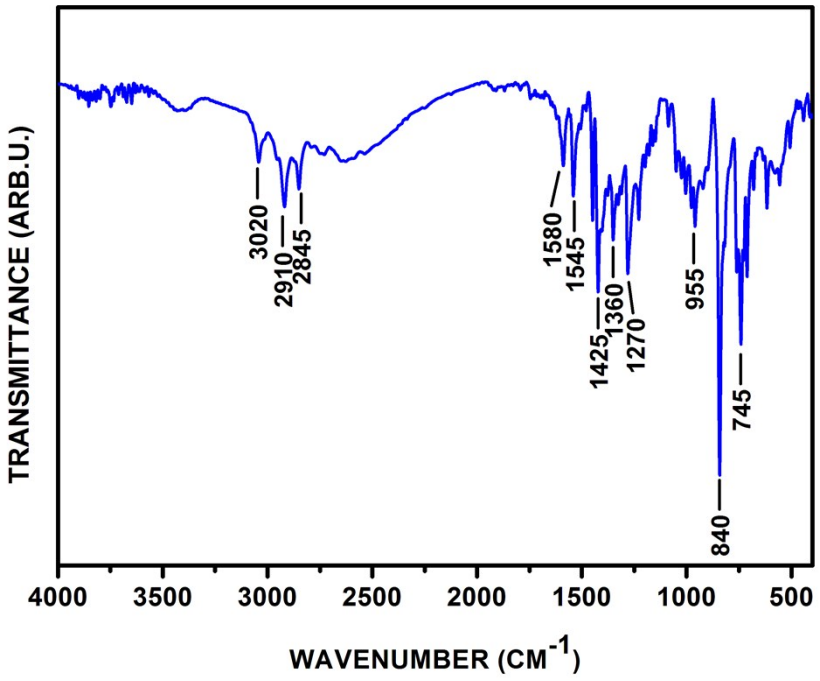

Fig. S23. FT-IR spectrum of 2-(1-pyrenyl)benzimidazole (**7**)[9]

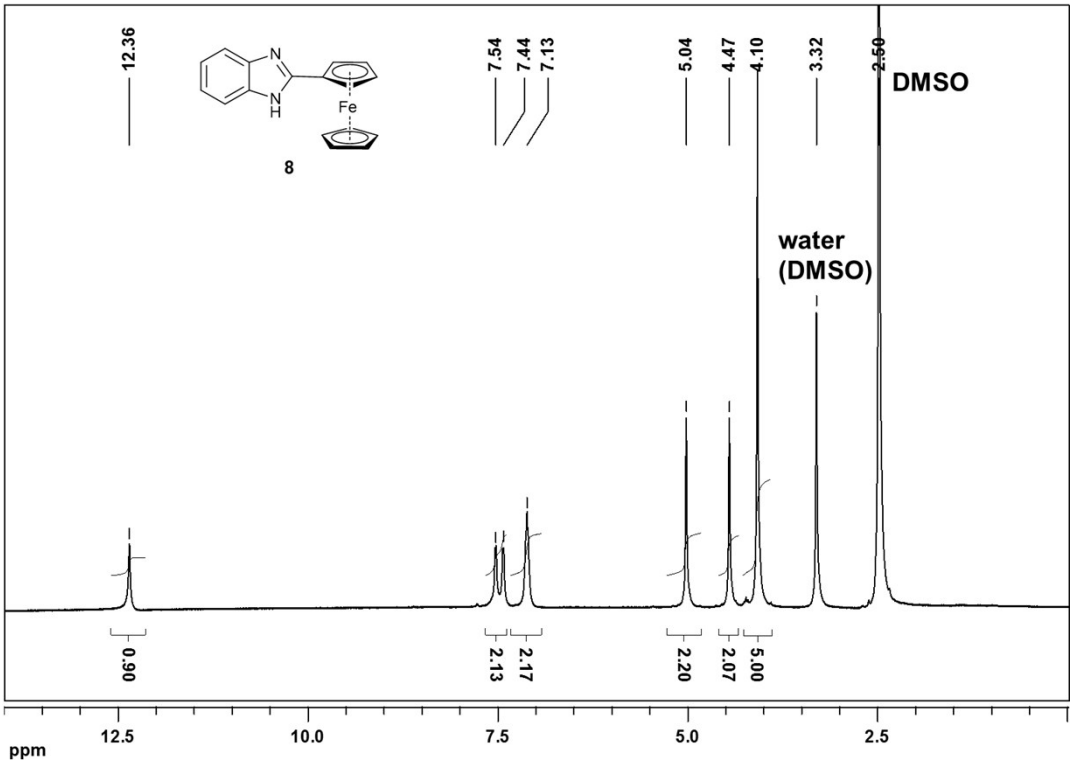

Fig. S24. ^{1}H NMR spectrum (500 MHz, DMSO-d_6) of 2-(ferrocenyl)benzimidazole (**8**)[10]

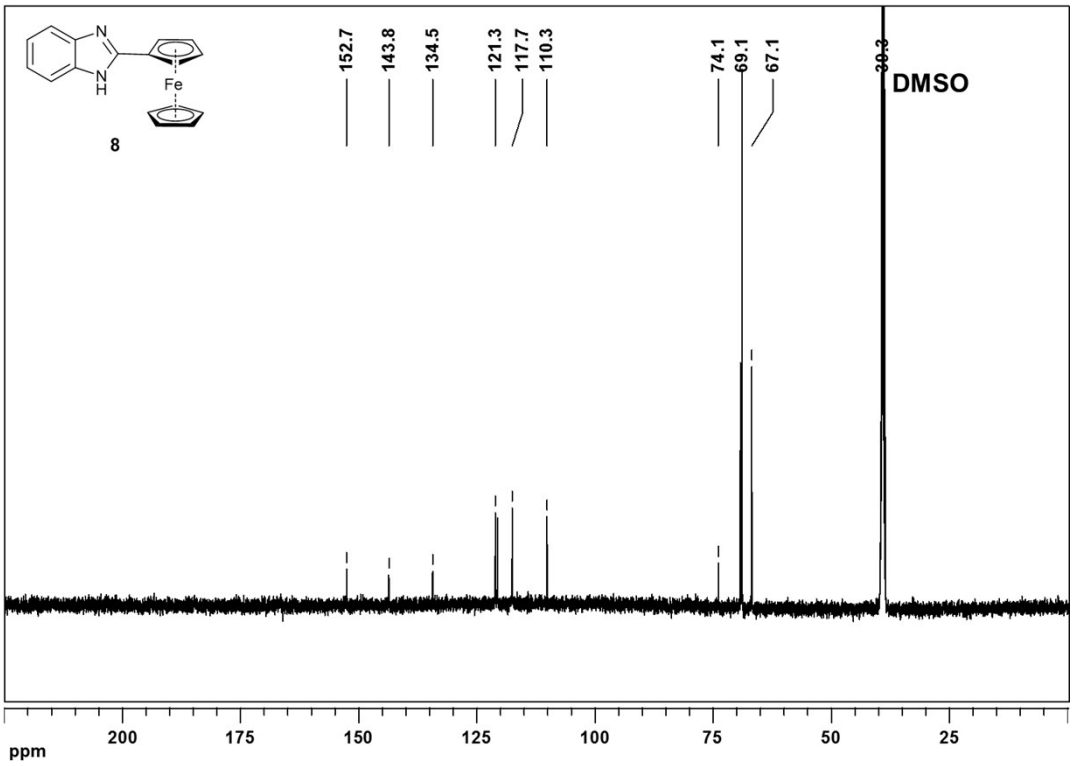

Fig. S25. ^{13}C NMR spectrum (125 MHz, DMSO-d_6) of 2-(ferrocenyl)benzimidazole (**8**)[10]

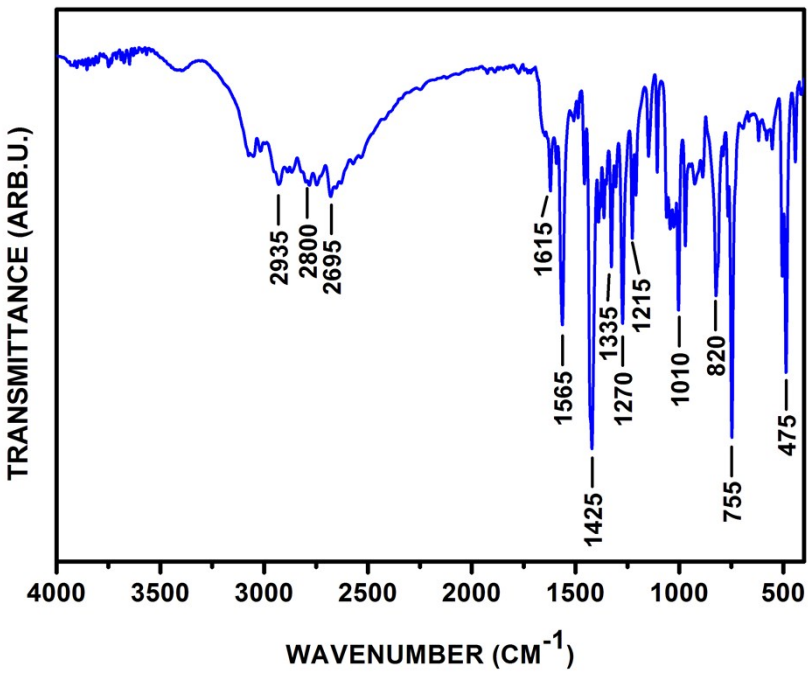

Fig. S26. FT-IR spectrum of 2-(ferrocenyl)benzimidazole (**8**)[10]

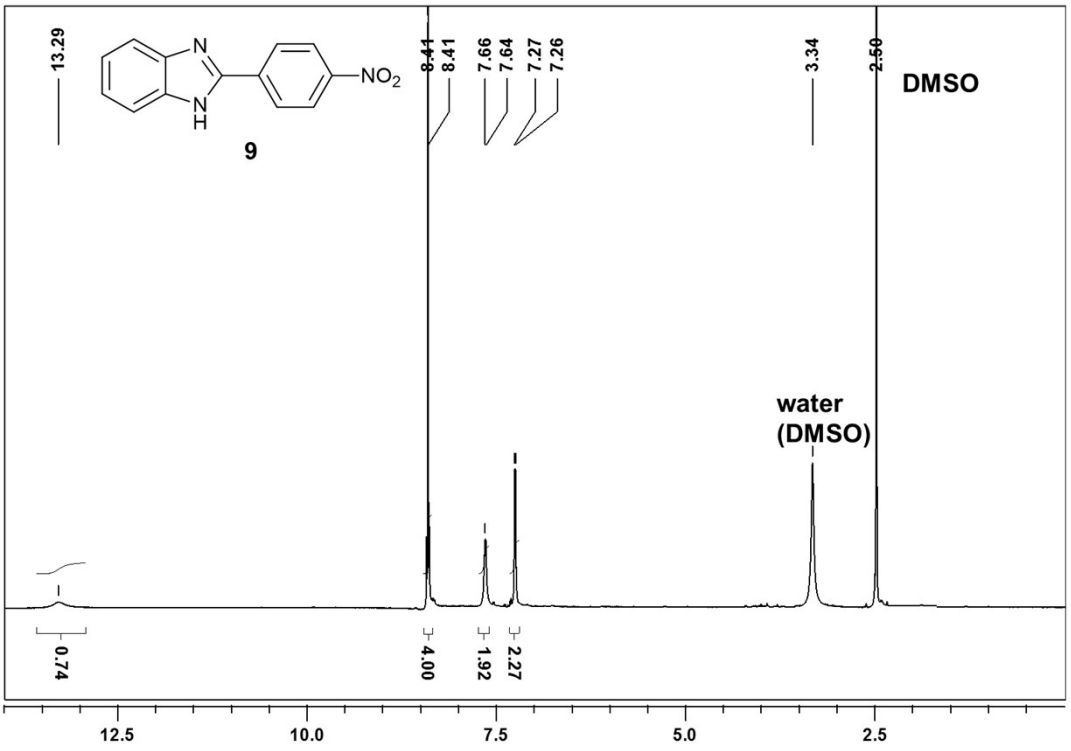

Fig. S27. ^{1}H NMR spectrum (500 MHz, DMSO-d_6) of 2-(4-nitrophenyl)benzimidazole (**9**)[4]

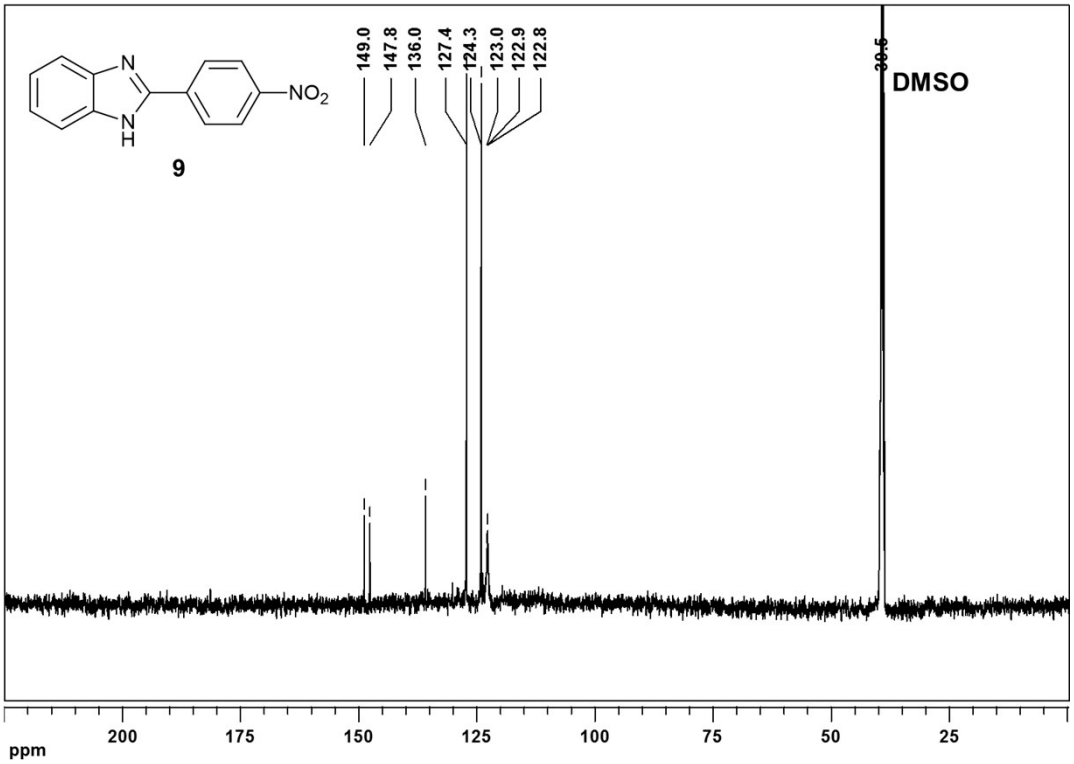

Fig. S28. ^{13}C NMR spectrum (125 MHz, DMSO-d_6) of 2-(4-nitrophenyl)benzimidazole (**9**)[4]

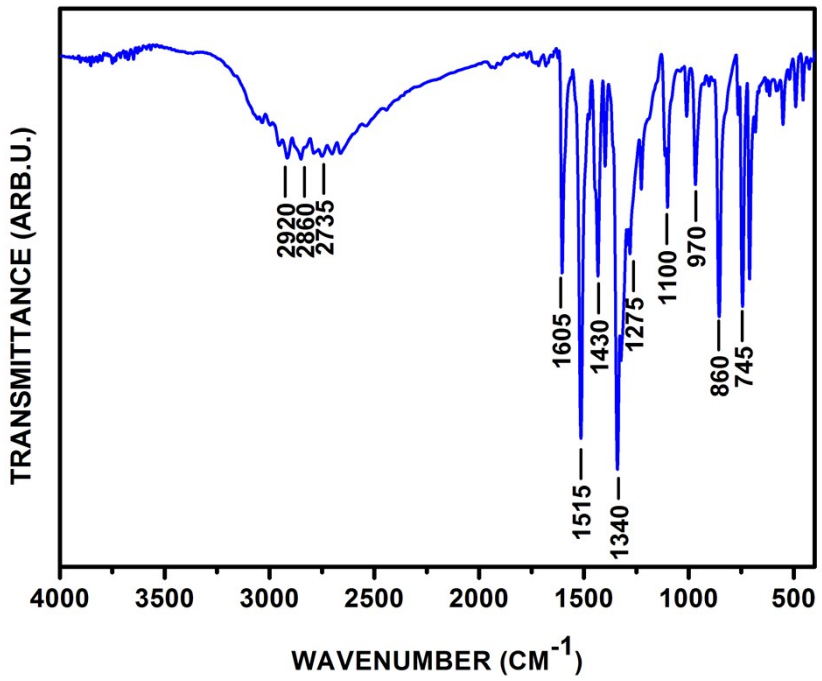

Fig. S29. FT-IR spectrum of 2-(4-nitrophenyl)benzoimidazole (**9**)[7]

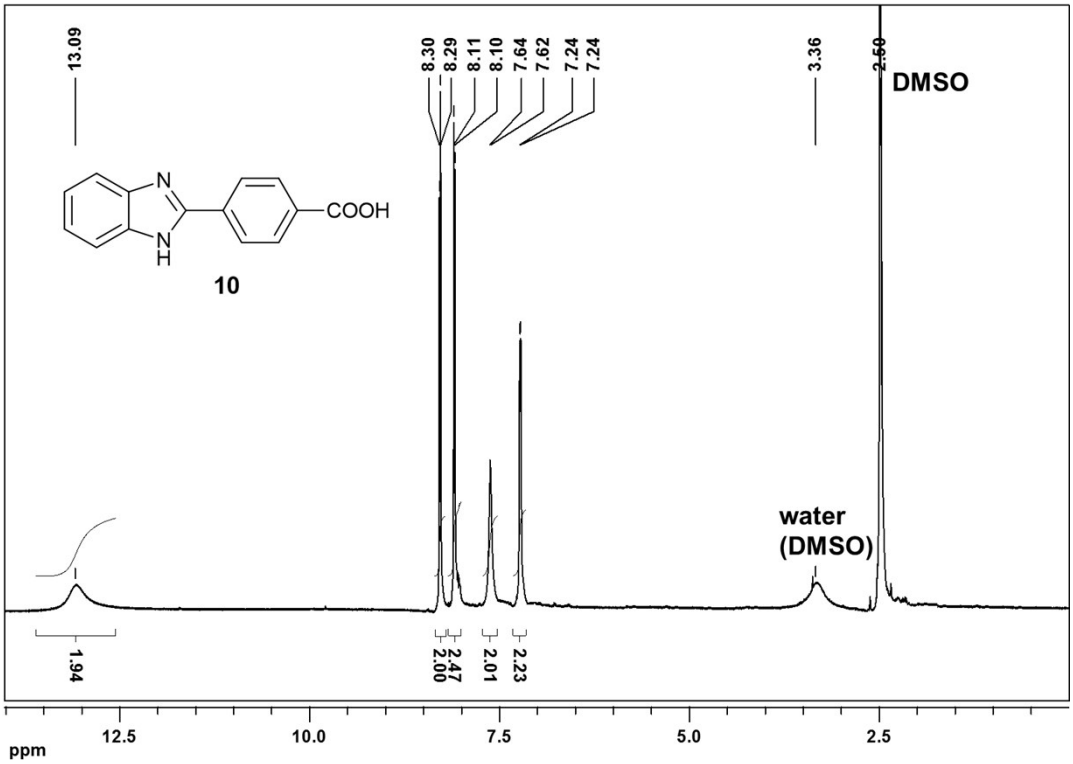

Fig. S30. ^{1}H NMR spectrum (500 MHz, DMSO-d_6) of 2-(4-carboxyphenyl)benzimidazole (**10**)[5]

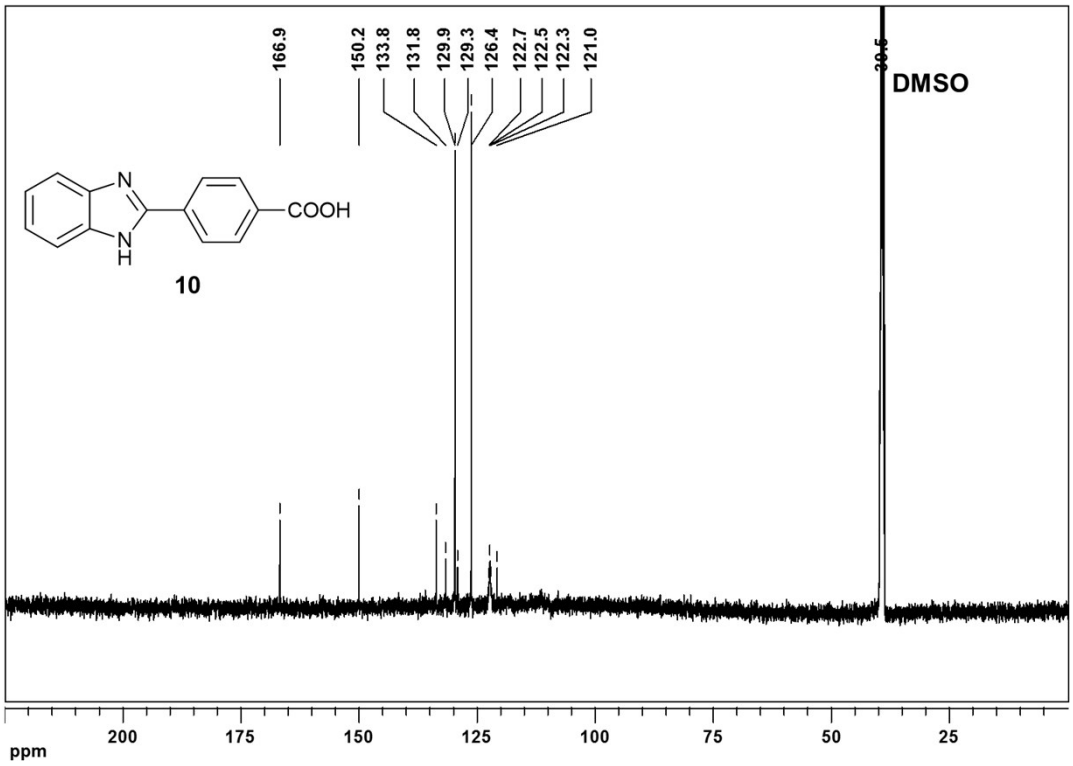

Fig. S31. ^{13}C NMR spectrum (125 MHz, DMSO-d_6) of 2-(4-carboxyphenyl)benzimidazole (**10**)[5]

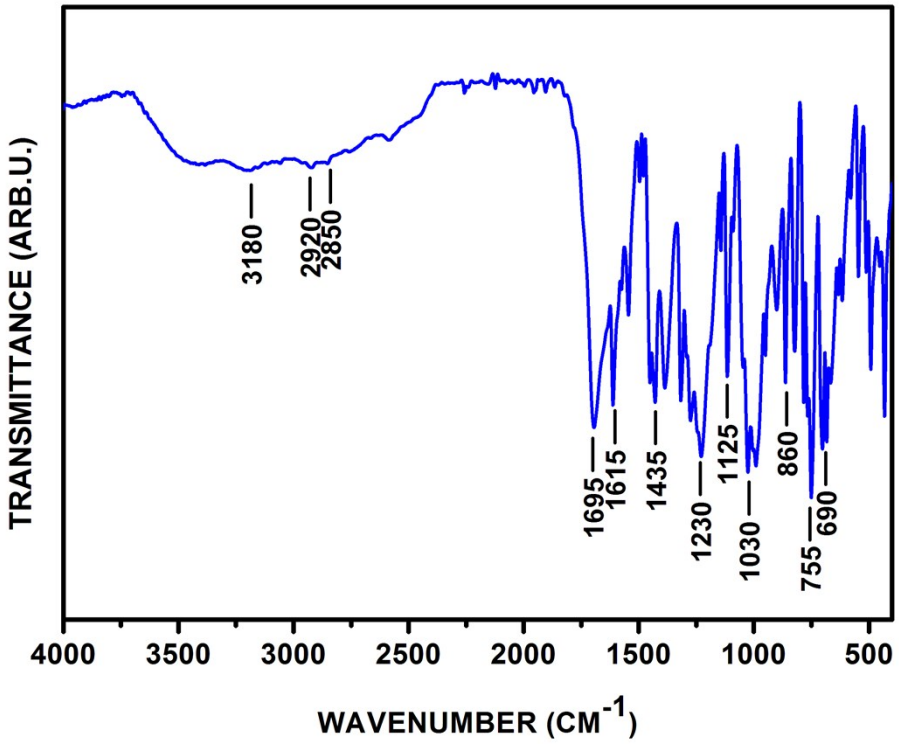

Fig. S32. FT-IR spectrum of 2-(4-carboxyphenyl)benzimidazole (**10**)[5]

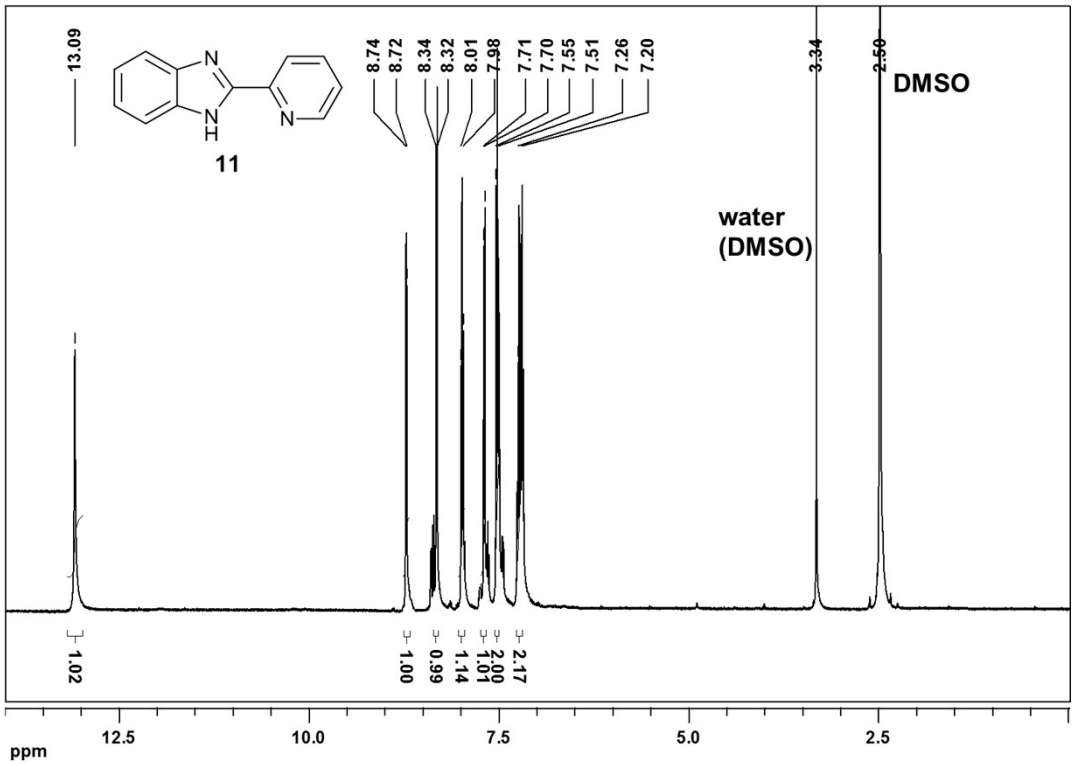

Fig. S33. ^{1}H NMR spectrum (500 MHz, DMSO-d_6) of 2-(2-pyridyl)benzimidazole (**11**)[4]

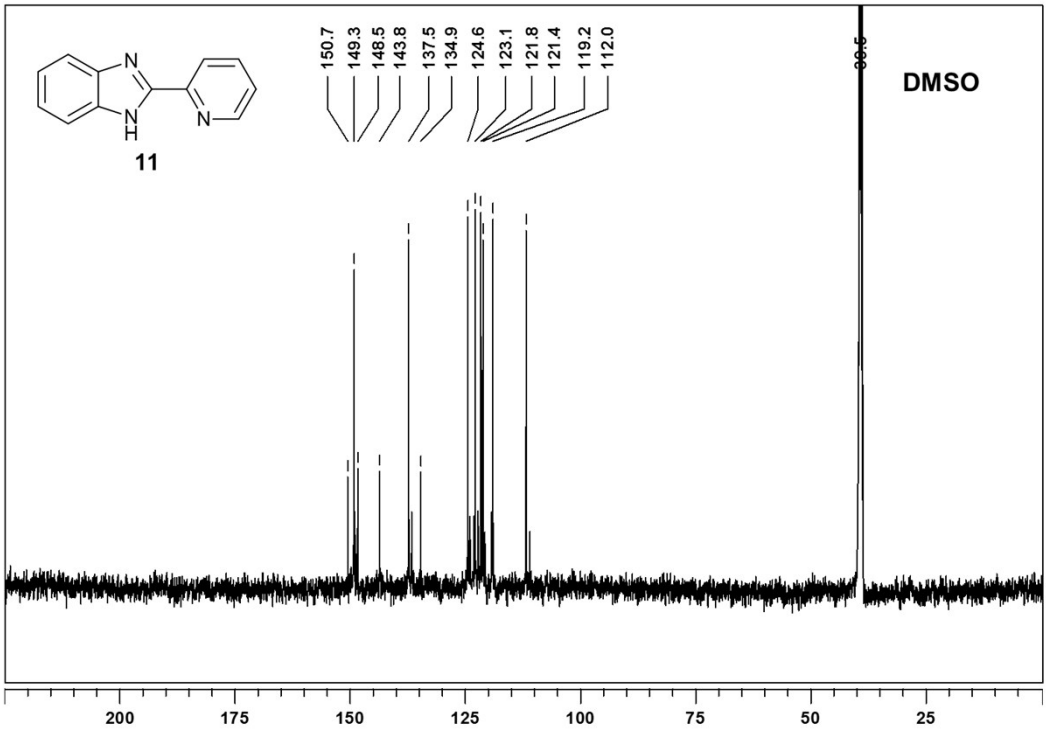

Fig. S34. ^{13}C NMR spectrum (125 MHz, DMSO-d_6) of 2-(2-pyridyl)benzimidazole (**11**)[4]

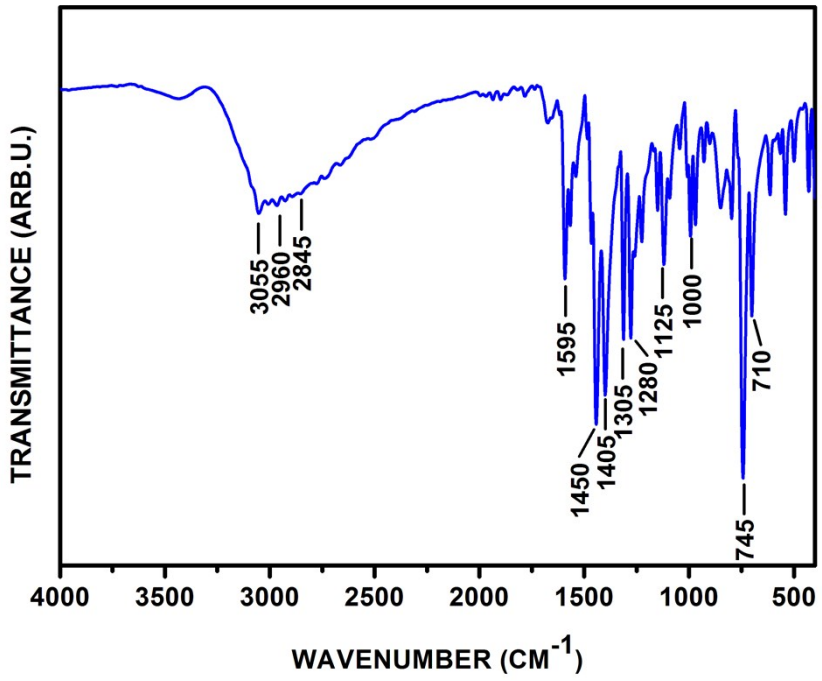

Fig. S35. FT-IR spectrum of 2-(2-pyridyl)benzimidazole (**11**)[5]

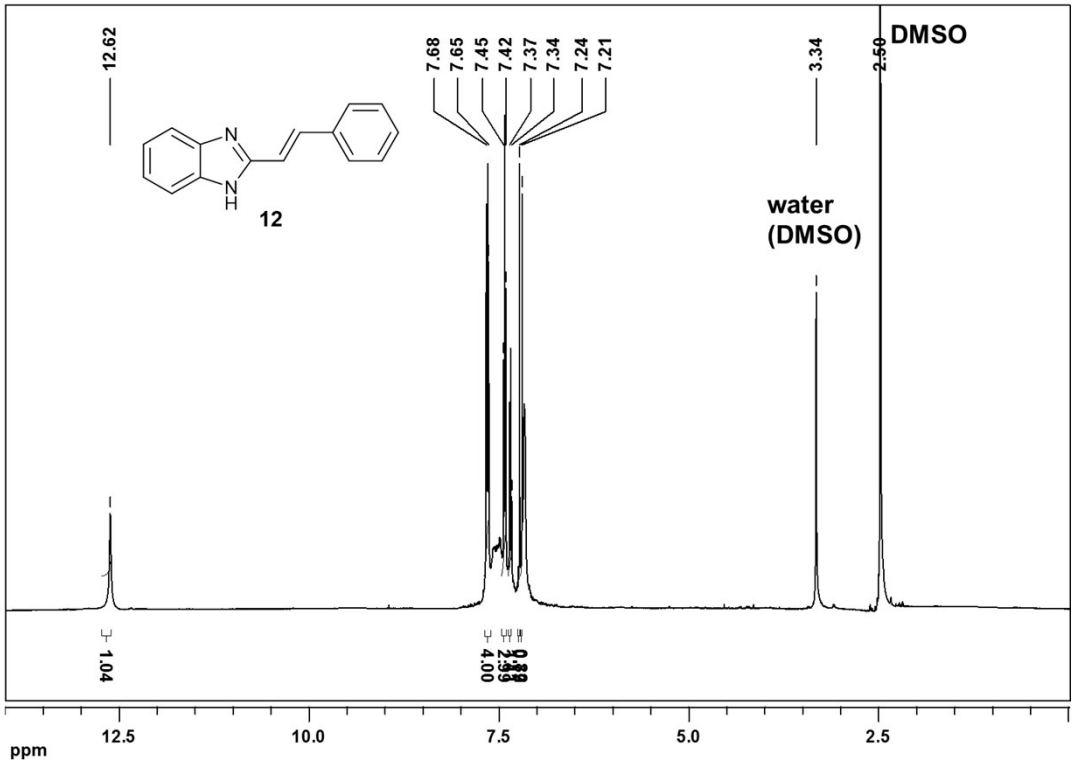

Fig. S36. ^{1}H NMR spectrum (500 MHz, DMSO-d_6) of *(E)*-2-styrylbenzimidazole (**12**)[11]

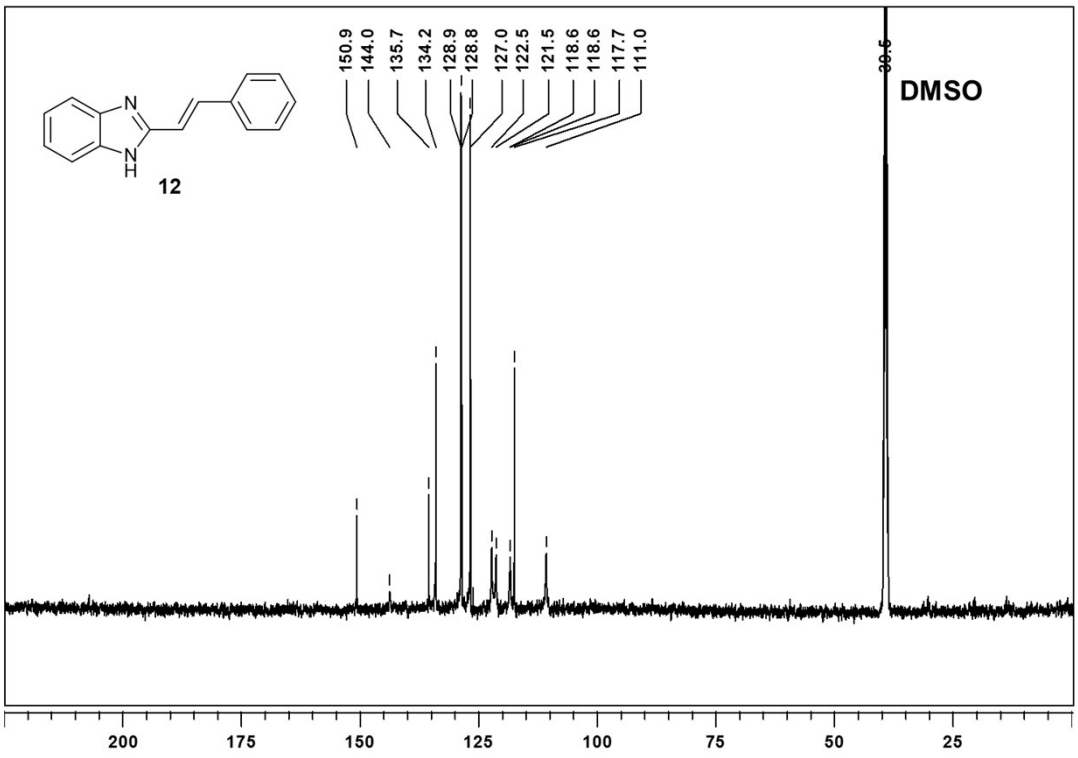

Fig. S37. ^{13}C NMR spectrum (125 MHz, DMSO-d_6) of *(E)*-2-styrylbenzimidazole (**12**)[11]

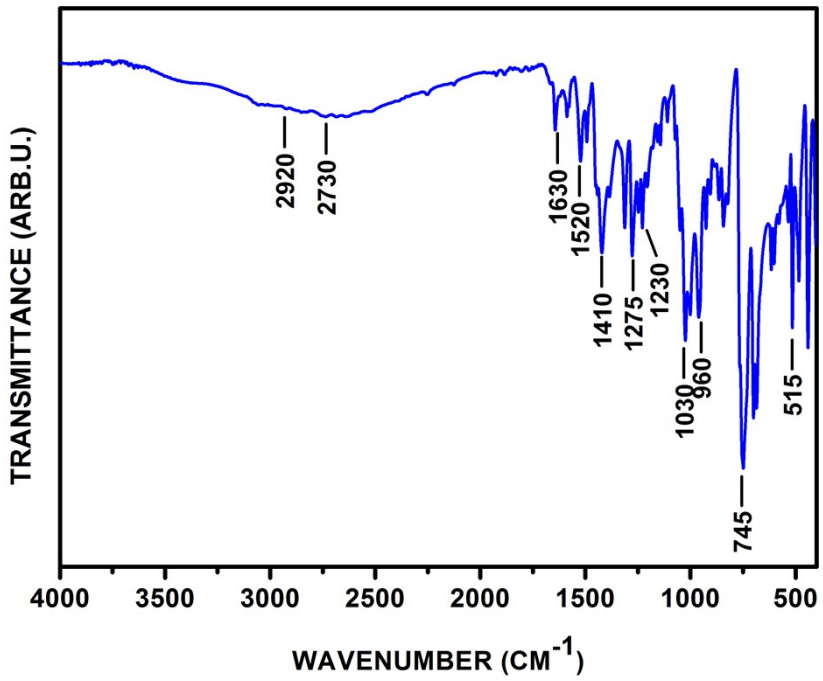

Fig. S38. FT-IR spectrum of *(E)*-2-styrylbenzimidazole (**12**)[11]

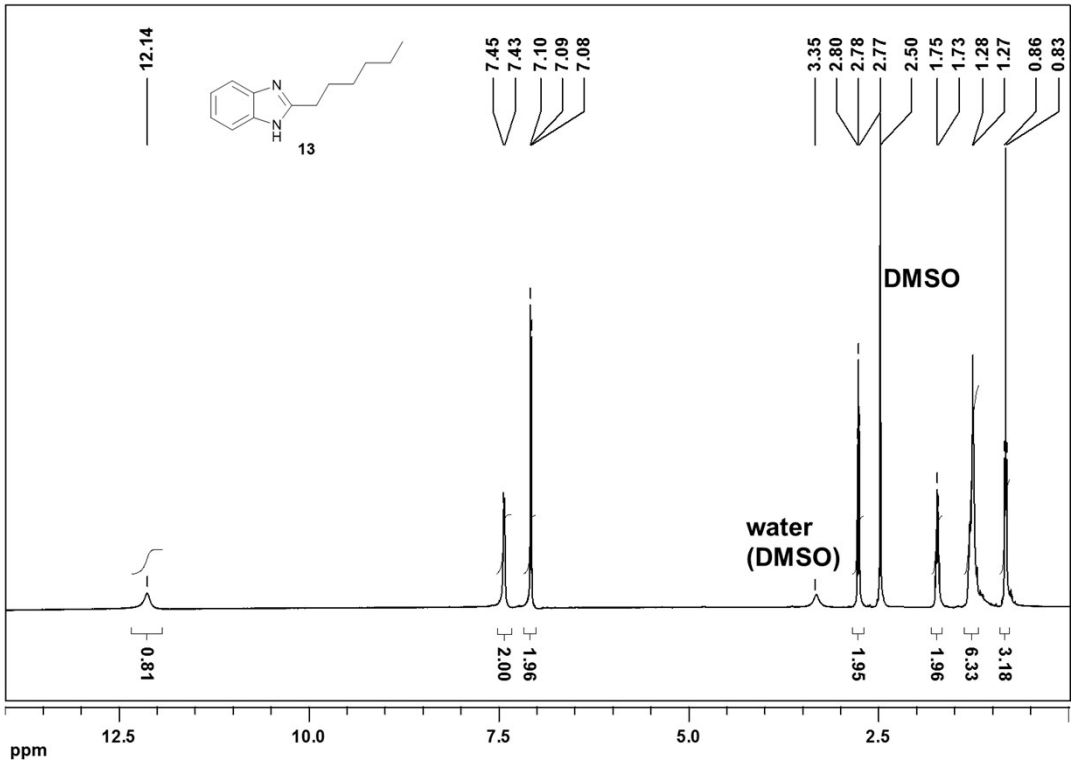

Fig. S39. ^{1}H NMR spectrum (500 MHz, DMSO-d_6) of 2-hexylbenzimidazole (**13**)[12]

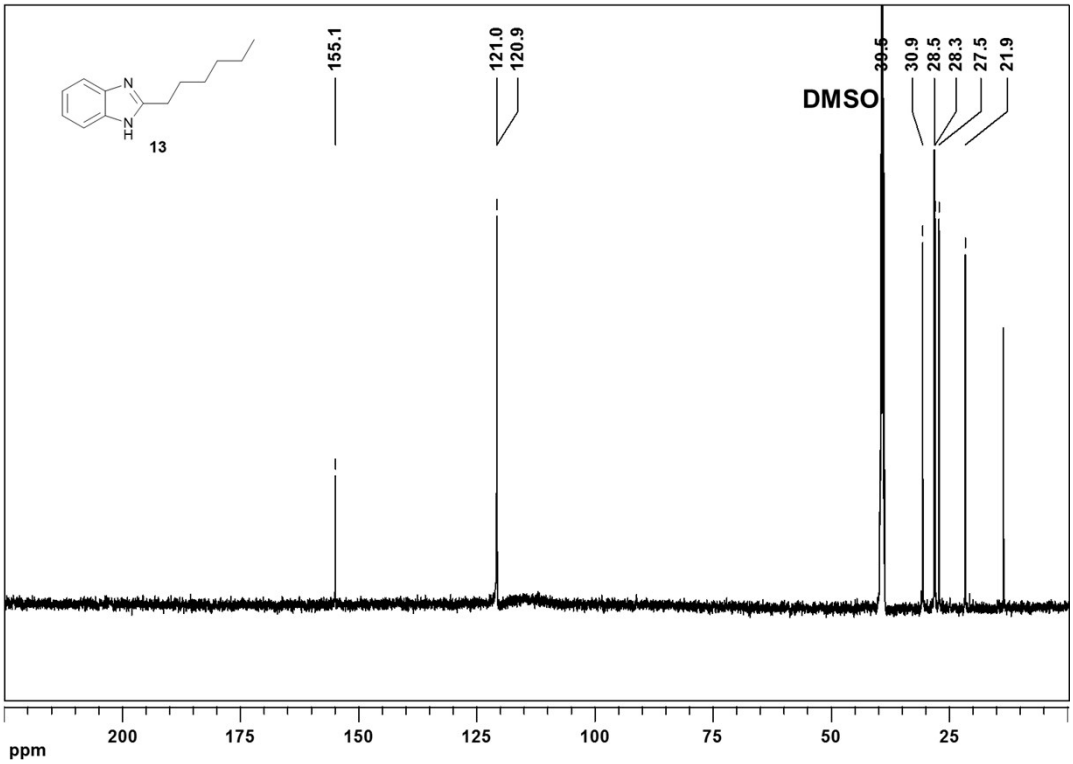

Fig. S40. ^{13}C NMR spectrum (125 MHz, DMSO-d_6) of 2-hexylbenzimidazole (**13**)[12]

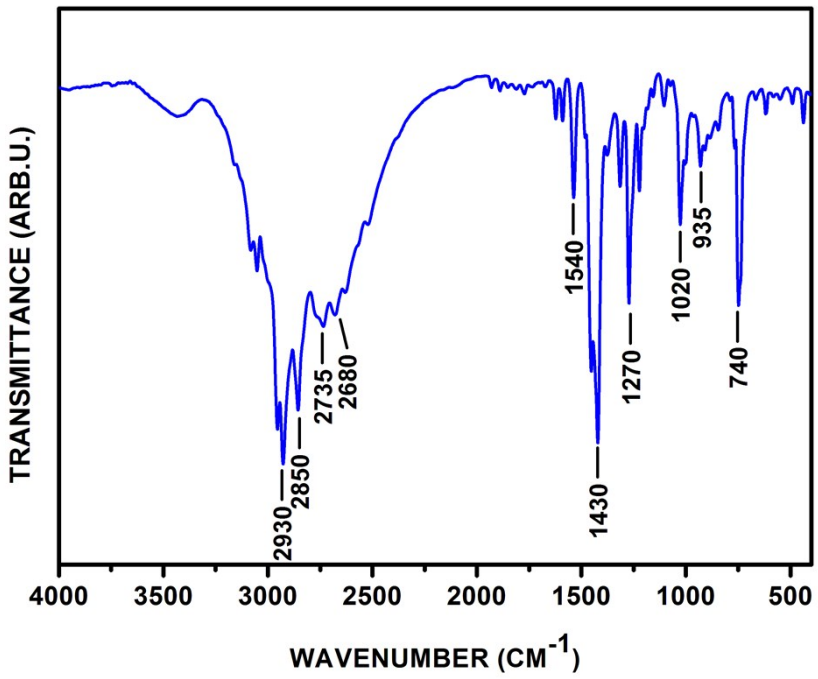

Fig. S41. FT-IR spectrum of 2-hexylbenzimidazole (**13**)[12]

3. Recyclability study on NANOCAT-G4: FT-IR data

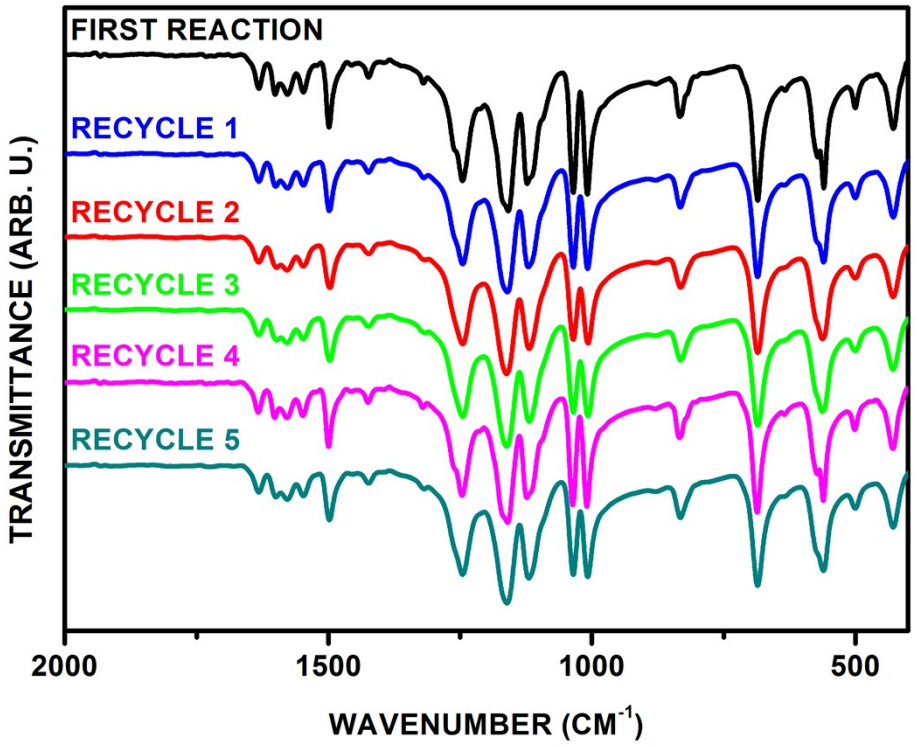

Fig. S42. Comparison of FT-IR spectra of **NANOCAT-G4** after 5 reaction cycles

References:

1 A. Kasprzak, M. Bystrzejewski, M. Koszytkowska-Stawinska and M. Poplawska, *Green Chem.*, 2017, **19**, 3510.

2 A. Kasprzak, A. M. Nowicka, J. P. Sek, M. Fronczak, M. Bystrzejewski, M. Koszytkowska-Stawinska and M. Poplawska, *Dalt. Trans.*, 2018, **47**, 30.

3 M. Poplawska, G. Z. Zukowska, S. Cudziło and M. Bystrzejewski, *Carbon N. Y.*, 2010, **48**, 1318–1320.

4 M. R. Marri, S. Peraka, A. K. Macharla, N. Mameda, S. Kodumuri and N. Nama, *Tetrahedron Lett.*, 2014, **55**, 6520–6525.

5 Y. K. Bommegowda, G. S. Lingaraju, S. Thamas, K. S. Vinay Kumar, C. S. Pradeepa Kumara, K. S. Rangappa and M. P. Sadashiva, *Tetrahedron Lett.*, 2013, **54**, 2693–2695.

6 O. Ravi, A. Shaikh, A. Upare, K. K. Singarapu and S. R. Bathula, *J. Org. Chem.*, 2017, **82**, 4422–4428.

7 Y. Kim, M. R. Kumar, N. Park, Y. Heo and S. Lee, *J. Org. Chem.*, 2011, **76**, 9577–9583.

8 G. Bai, X. Lan, X. Liu, C. Liu, L. Shi, Q. Chen and G. Chen, *Green Chem.*, 2014, **16**, 3160–3168.

9 M. Zhao, Z. Deng, J. Tang, X. Zhou, Z. Chen, X. Li, L. Yang and L.-J. Ma, *Analyst*, 2016, **141**, 2308–2312.

10 A. Benito, R. Martínez-Máñez, J. Payá, J. Soto, M. J. Tendero and E. Sinn, *J. Organomet. Chem.*, 1995, **503**, 259–263.

11 V. K. A. Kalalbandi and J. Seetharamappa, *Synth. Commun.*, 2016, **46**, 626–635.

12 K. L. Tan, R. G. Bergman and J. A. Ellman, *J. Am. Chem. Soc.*, 2002, **124**, 13964–13965.