

Application of magnetic deep eutectic solvent as an efficient catalyst in the synthesis of new 1,2,3-triazole-nicotinonitrile hybrids via a cooperative vinylogous anomeric based oxidation

Monireh Navazeni, Mohammad Ali Zolfigol*, Morteza Torabi, Ardashir Khazaei*

Department of Organic Chemistry, Faculty of Chemistry and Petroleum Sciences, Bu-Ali Sina University, Hamedan, Iran

zolfi@basu.ac.ir; mzolfigol@yahoo.com (M.A. Zolfigol), khazaeiardeshir@gmail.com (A. Khazaei)

- 1: FT-IR spectrum of 1,1'-(naphthalene-1,5-diyl)bis(3-(3-(triethoxysilyl)propyl)urea)
- 2: ¹H NMR spectrum of 1,1'-(naphthalene-1,5-diyl)bis(3-(3-(triethoxysilyl)propyl)urea)
- 3: FT-IR spectrum of 2-amino-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)-4-phenylnicotinonitrile
- 4: ¹H NMR spectrum of 2-amino-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)-4-phenylnicotinonitrile
- 5: ¹³C NMR spectrum of 2-amino-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)-4-phenylnicotinonitrile
- 6: Mass spectrum of 2-amino-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)-4-phenylnicotinonitrile
- 7: FT-IR spectrum of 2-amino-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)-4-(*p*-tolyl) nicotinonitrile
- 8: ¹H NMR spectrum of 2-amino-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)-4-(*p*-tolyl) nicotinonitrile
- 9: Mass spectrum of 2-amino-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)-4-(*p*-tolyl) nicotinonitrile
- 10: FT-IR spectrum of 2-amino-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)-4-(4-methoxyphenyl) nicotinonitrile

- 11: ¹H NMR spectrum of 2-amino-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)-4-(4-methoxyphenyl) nicotinonitrile
- 12: ¹³C NMR spectrum of 2-amino-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)-4-(4-methoxyphenyl) nicotinonitrile
- 13: Mass spectrum of 2-amino-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)-4-(4-methoxyphenyl) nicotinonitrile
- 14: FT-IR spectrum of 2-amino-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)-4-(naphthalen-1-yl) nicotinonitrile
- 15: ¹H NMR spectrum of 2-amino-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)-4-(naphthalen-1-yl) nicotinonitrile
- 16: ¹³C NMR spectrum of 2-amino-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)-4-(naphthalen-1-yl) nicotinonitrile
- 17: Mass spectrum of 2-amino-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)-4-(naphthalen-1-yl) nicotinonitrile
- 18: FT-IR spectrum of 2-amino-4-(1*H*-indol-3-yl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile
- 19: ¹H NMR spectrum of 2-amino-4-(1*H*-indol-3-yl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile
- 20: ¹³C NMR spectrum of 2-amino-4-(1*H*-indol-3-yl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile
- 21: FT-IR spectrum of 2-amino-4-(4-chlorophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile
- 22: ¹H NMR spectrum of 2-amino-4-(4-chlorophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile
- 23: ¹³C NMR spectrum of 2-amino-4-(4-chlorophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile
- 24: Mass spectrum of 2-amino-4-(4-chlorophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile
- 25: FT-IR spectrum of 2-amino-4-(4-bromophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile

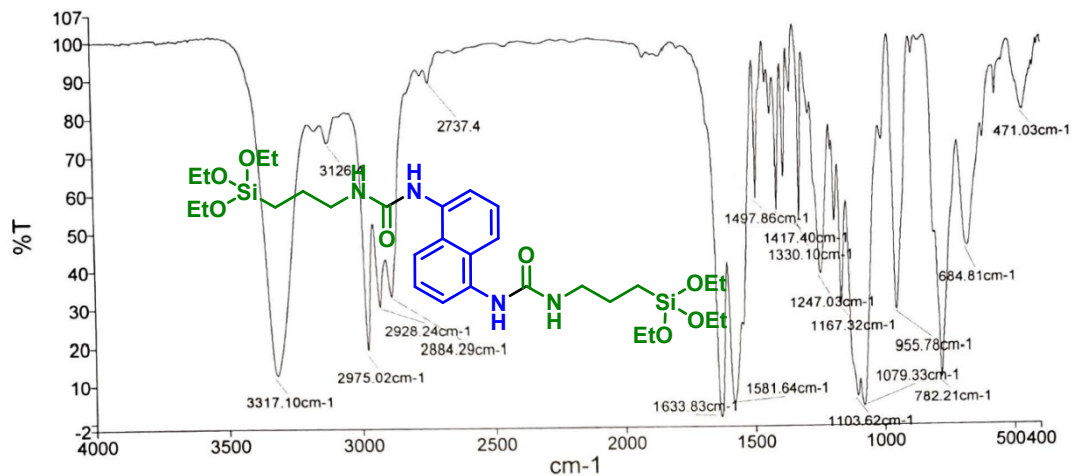
- 26: ¹H NMR spectrum of 2-amino-4-(4-bromophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile
- 27: ¹³C NMR spectrum of 2-amino-4-(4-bromophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile
- 28: Mass spectrum of 2-amino-4-(4-bromophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile
- 29: FT-IR spectrum of 2-amino-4-(3-bromophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile
- 30: ¹H NMR spectrum of 2-amino-4-(3-bromophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile
- 31: ¹³C NMR spectrum of 2-amino-4-(3-bromophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile
- 32: Mass spectrum of 2-amino-4-(3-bromophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile
- 33: ¹H NMR spectrum of 2-amino-4-(4-chloro-3-nitrophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile
- 34: ¹³C NMR spectrum of 2-amino-4-(4-chloro-3-nitrophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile
- 35: Mass spectrum of 2-amino-4-(4-chloro-3-nitrophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile
- 36: FT-IR spectrum of 2-amino-4-(4-florophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile
- 37: ¹H NMR spectrum of 2-amino-4-(4-florophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile
- 38: ¹³C NMR spectrum of 2-amino-4-(4-florophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile

- 39: Mass spectrum of 2-amino-4-(4-florophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile
- 40: FT-IR spectrum of 2-amino-4-(2-florophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile
- 41: ¹H NMR spectrum of 2-amino-4-(2-florophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile
- 42: ¹³C NMR spectrum of 2-amino-4-(2-florophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile
- 43: Mass spectrum of 2-amino-4-(2-florophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile
- 44: FT-IR spectrum of 2-amino-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)-4-(4-nitrophenyl) nicotinonitrile
- 45: ¹H NMR spectrum of 2-amino-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)-4-(4-nitrophenyl) nicotinonitrile
- 46: ¹³C NMR spectrum of 2-amino-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)-4-(4-nitrophenyl) nicotinonitrile
- 47: Mass spectrum of 2-amino-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)-4-(4-nitrophenyl) nicotinonitrile
- 48: FT-IR spectrum of 2-amino-4-(4-cyanophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile
- 49: ¹H NMR spectrum of 2-amino-4-(4-cyanophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile
- 50: ¹³C NMR spectrum of 2-amino-4-(4-cyanophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile
- 51: EDS signals of recovered Fe₃O₄@SiO₂@DES1
- 52: Mapping analysis of recovered Fe₃O₄@SiO₂@DES1
- 53: FESEM images of recovered Fe₃O₄@SiO₂@DES1

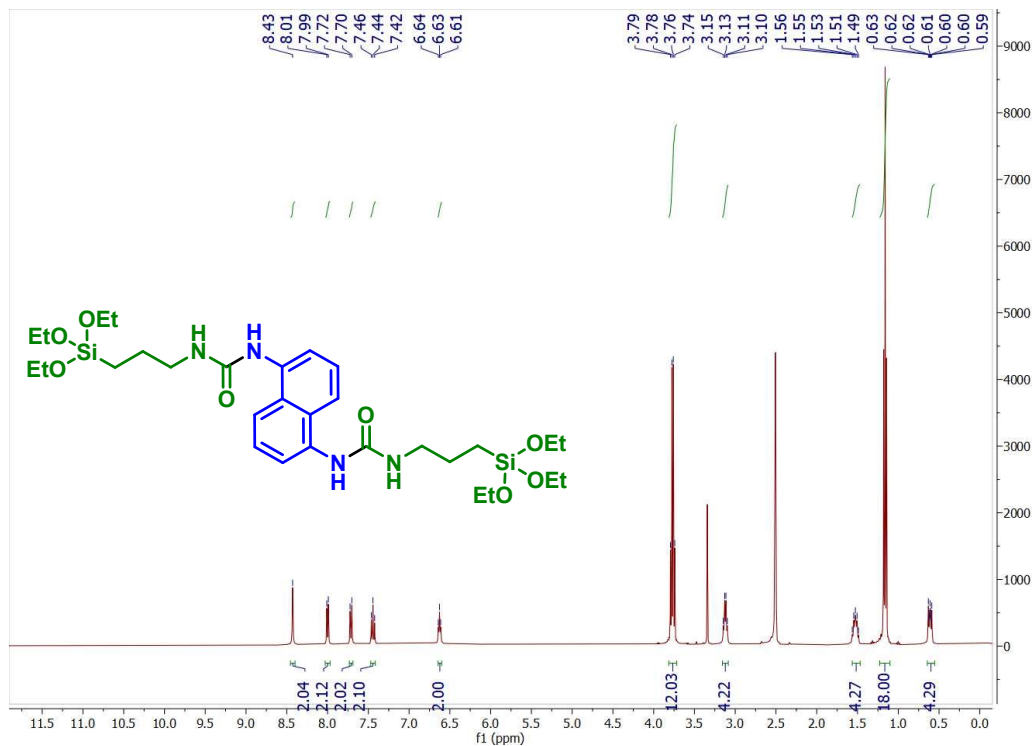
54: XRD pattern of recovered $\text{Fe}_3\text{O}_4@\text{SiO}_2@\text{DES1}$

55: The N_2 adsorption-desorption isotherms of $\text{Fe}_3\text{O}_4@\text{SiO}_2@\text{DES1}$ (a) and recovered $\text{Fe}_3\text{O}_4@\text{SiO}_2@\text{DES1}$ (b)

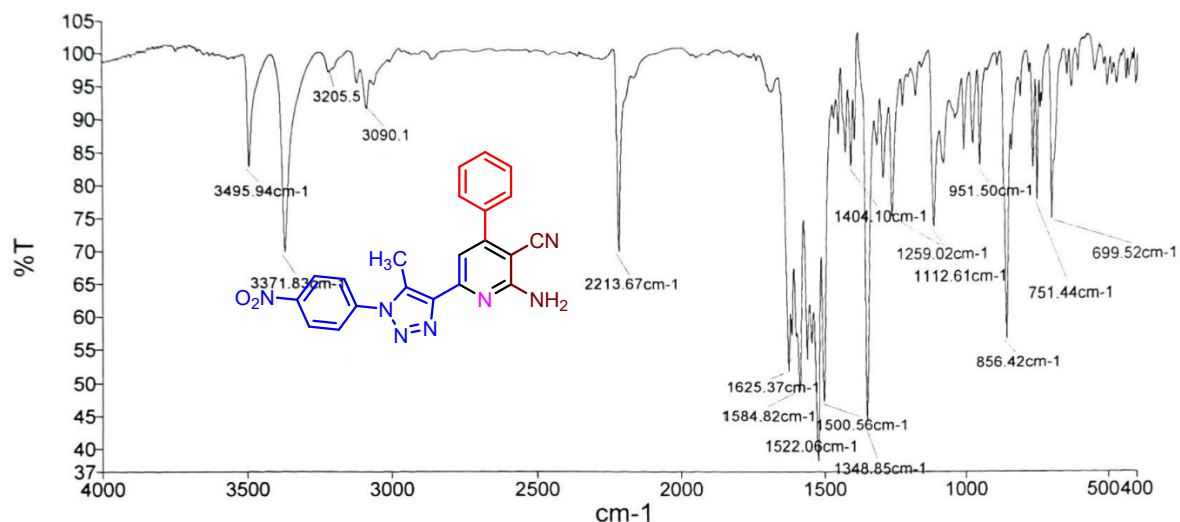
1: FT-IR spectrum of 1,1'-(naphthalene-1,5-diyl)bis(3-(3-(triethoxysilyl)propyl)urea)



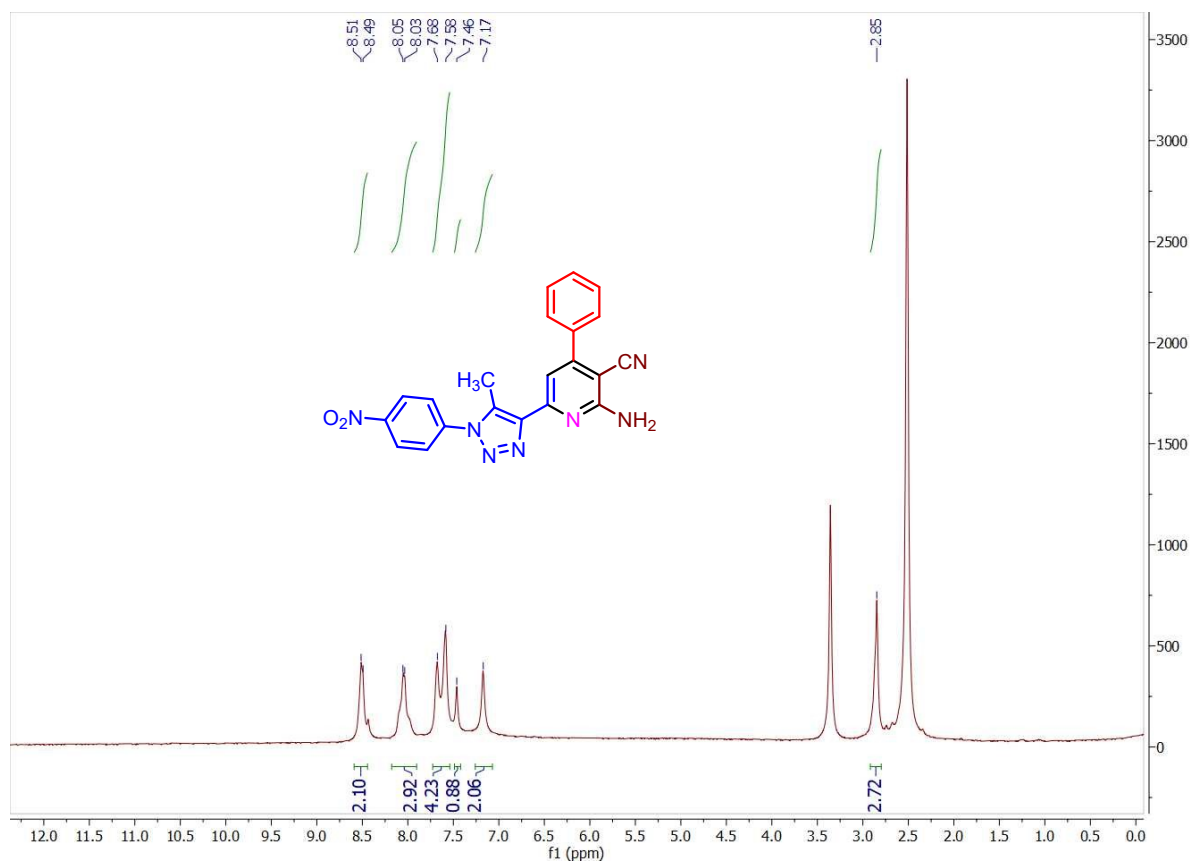
2: ^1H NMR spectrum of 1,1'-(naphthalene-1,5-diyl)bis(3-(3-(triethoxysilyl)propyl)urea)



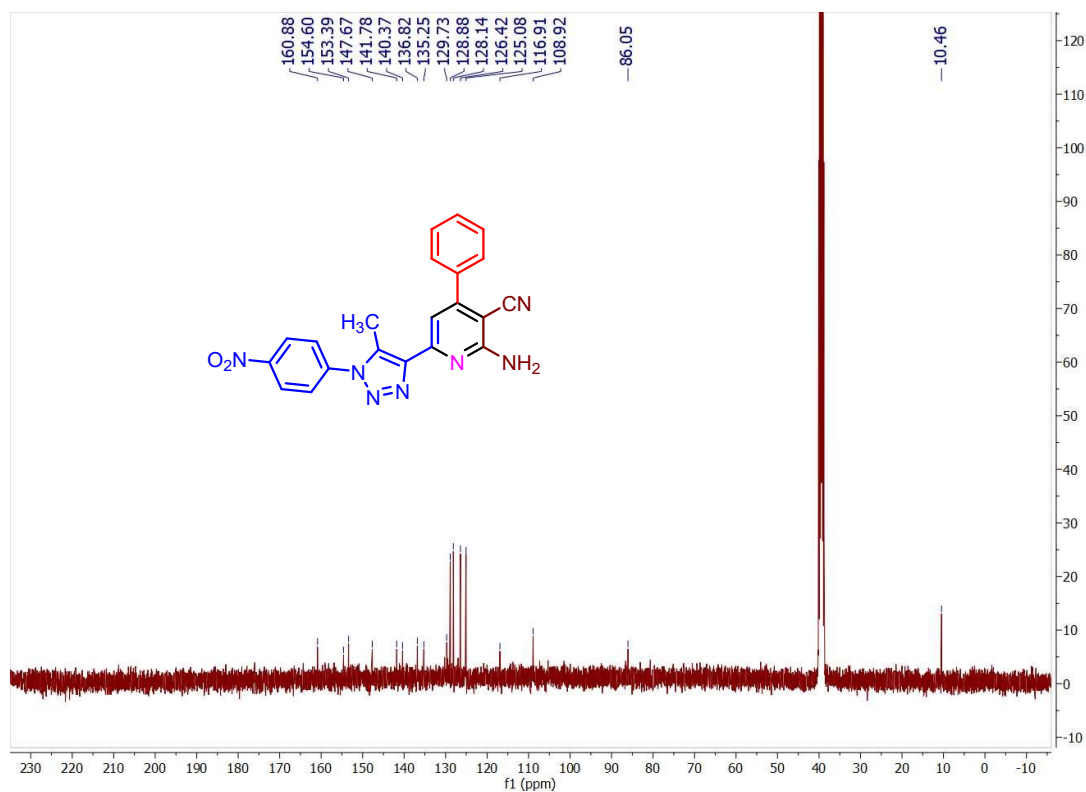
3: FT-IR spectrum of 2-amino-6-(5-methyl-1-(4-nitrophenyl)-1H-1,2,3-triazol-4-yl)-4-phenylnicotinonitrile



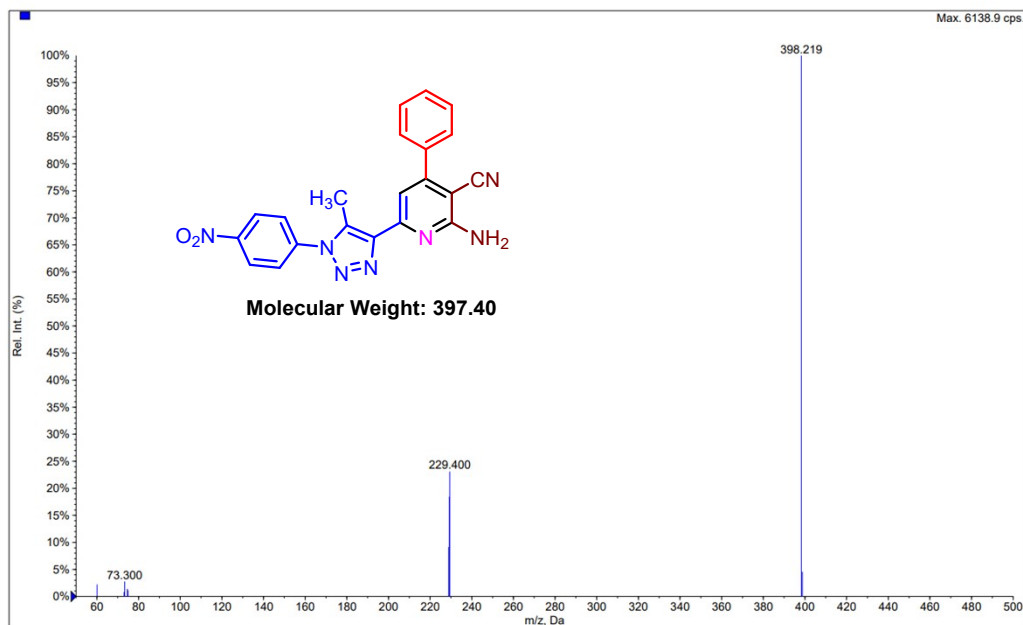
4: ¹H NMR spectrum of 2-amino-6-(5-methyl-1-(4-nitrophenyl)-1H-1,2,3-triazol-4-yl)-4-phenylnicotinonitrile



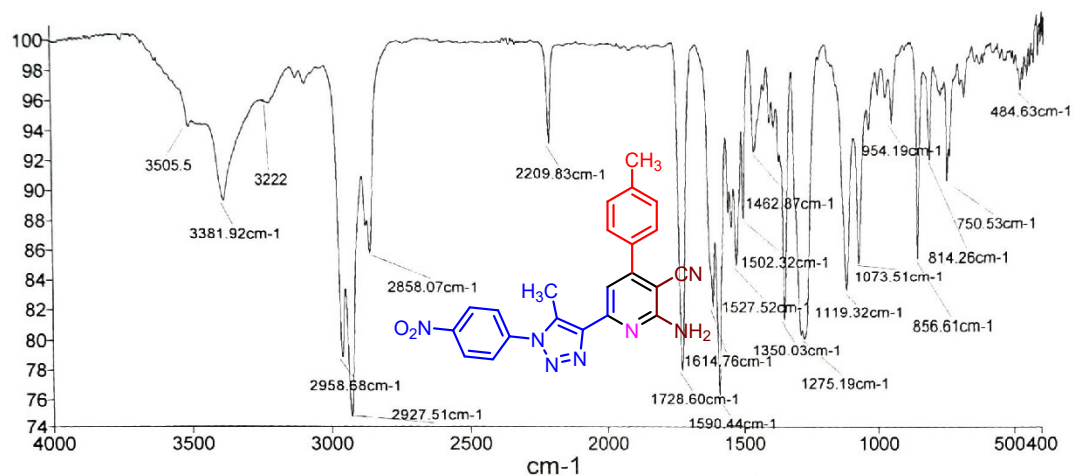
5: ^{13}C NMR spectrum of 2-amino-6-(5-methyl-1-(4-nitrophenyl)-1H-1,2,3-triazol-4-yl)-4-phenylnicotinonitrile



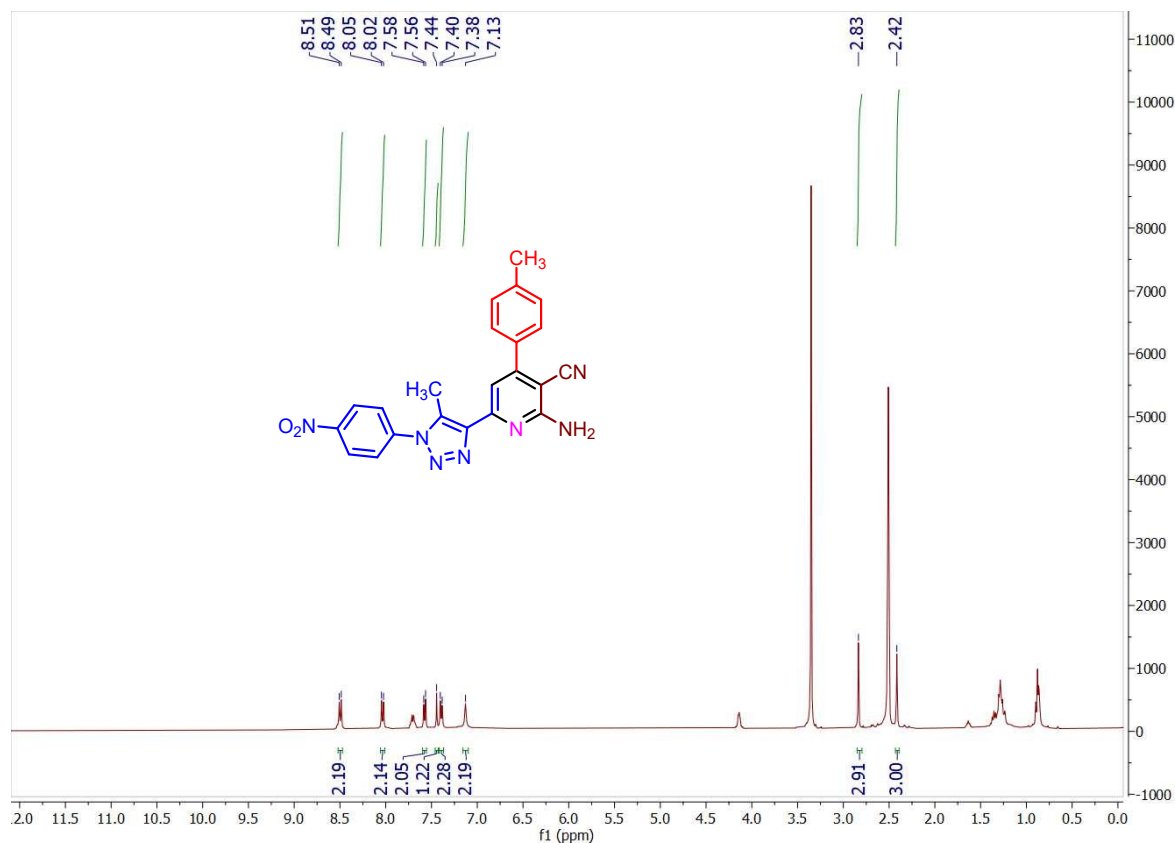
6: Mass spectrum of 2-amino-6-(5-methyl-1-(4-nitrophenyl)-1H-1,2,3-triazol-4-yl)-4-phenylnicotinonitrile



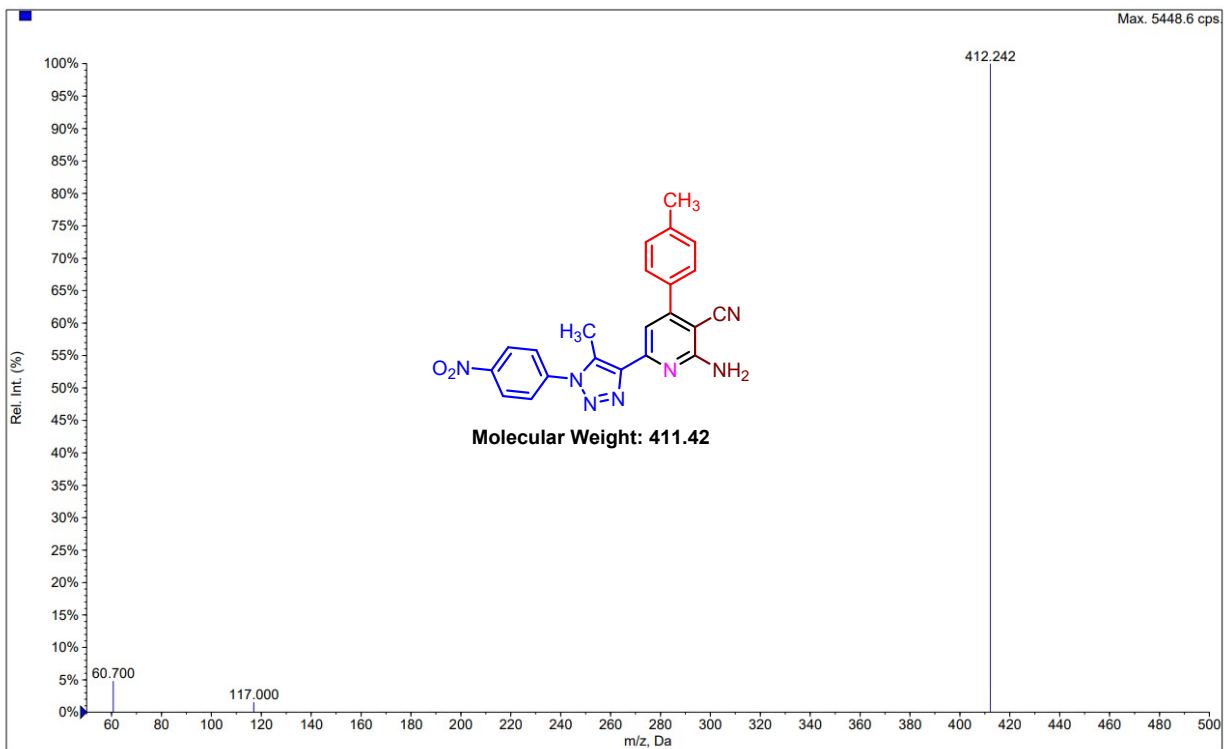
7: FT-IR spectrum of 2-amino-6-(5-methyl-1-(4-nitrophenyl)-1H-1,2,3-triazol-4-yl)-4-(*p*-tolyl) nicotinonitrile



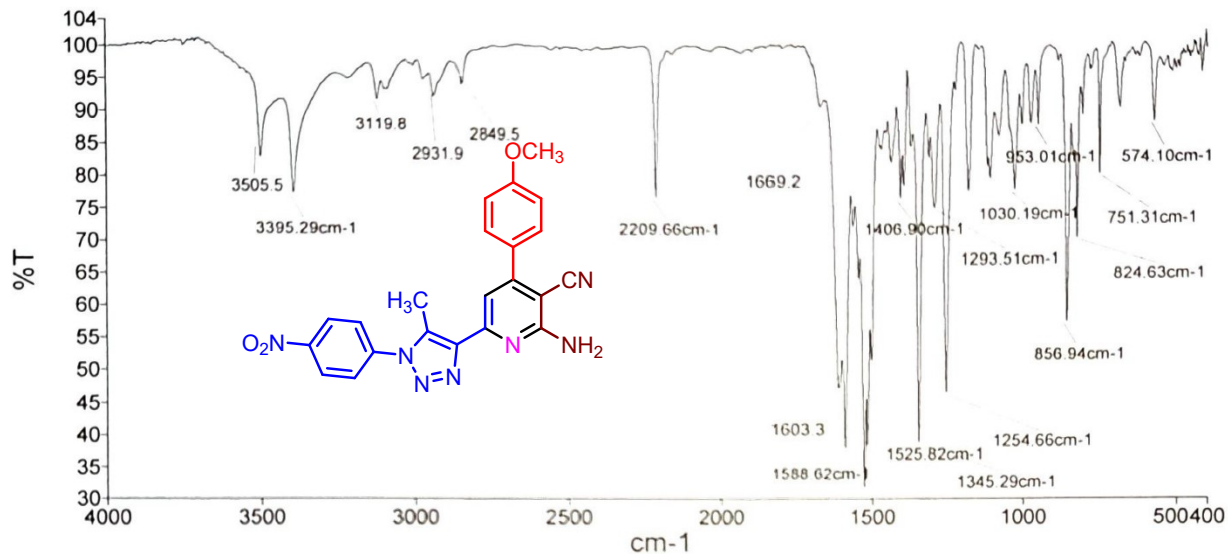
8: ¹H NMR spectrum of 2-amino-6-(5-methyl-1-(4-nitrophenyl)-1H-1,2,3-triazol-4-yl)-4-(*p*-tolyl) nicotinonitrile



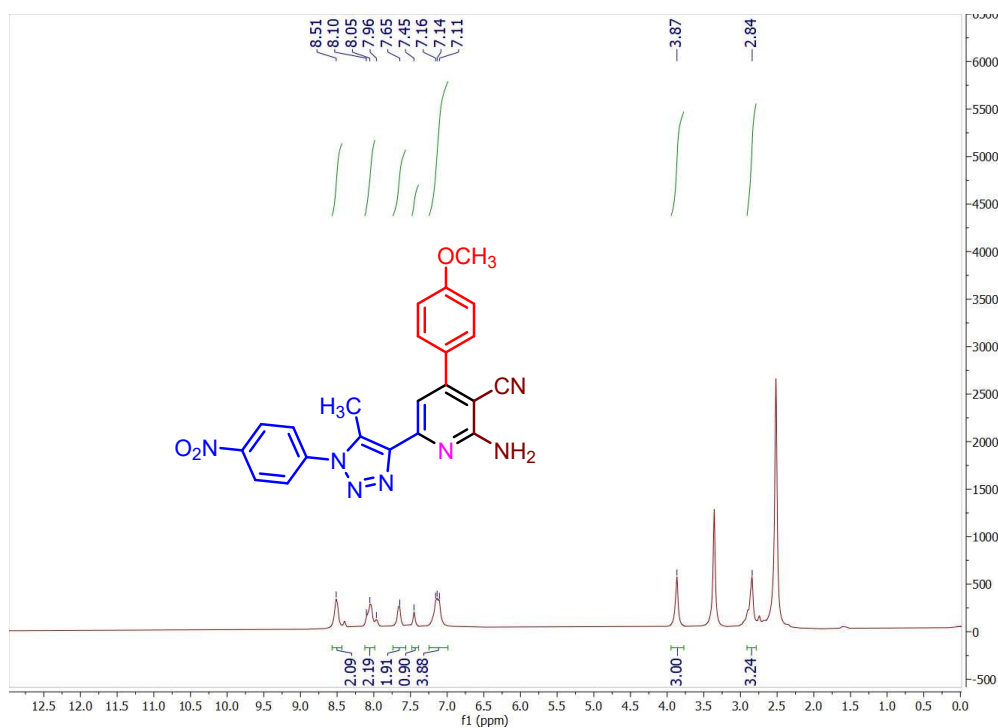
9: Mass spectrum of 2-amino-6-(5-methyl-1-(4-nitrophenyl)-1H-1,2,3-triazol-4-yl)-4-(*p*-tolyl) nicotinonitrile



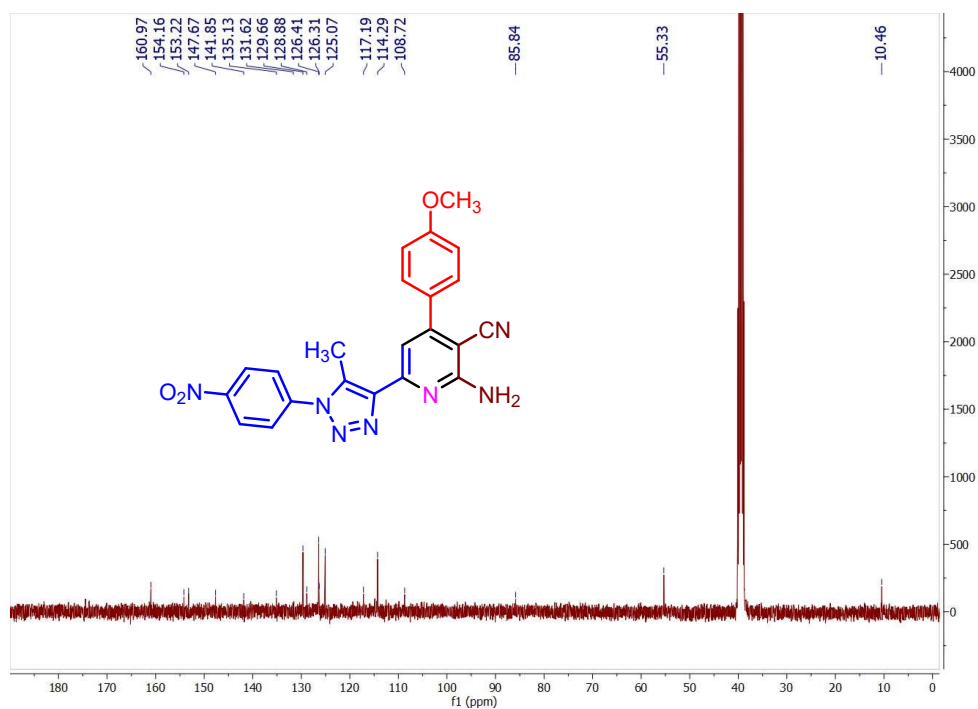
10: FT-IR spectrum of 2-amino-6-(5-methyl-1-(4-nitrophenyl)-1H-1,2,3-triazol-4-yl)-4-(4-methoxyphenyl) nicotinonitrile



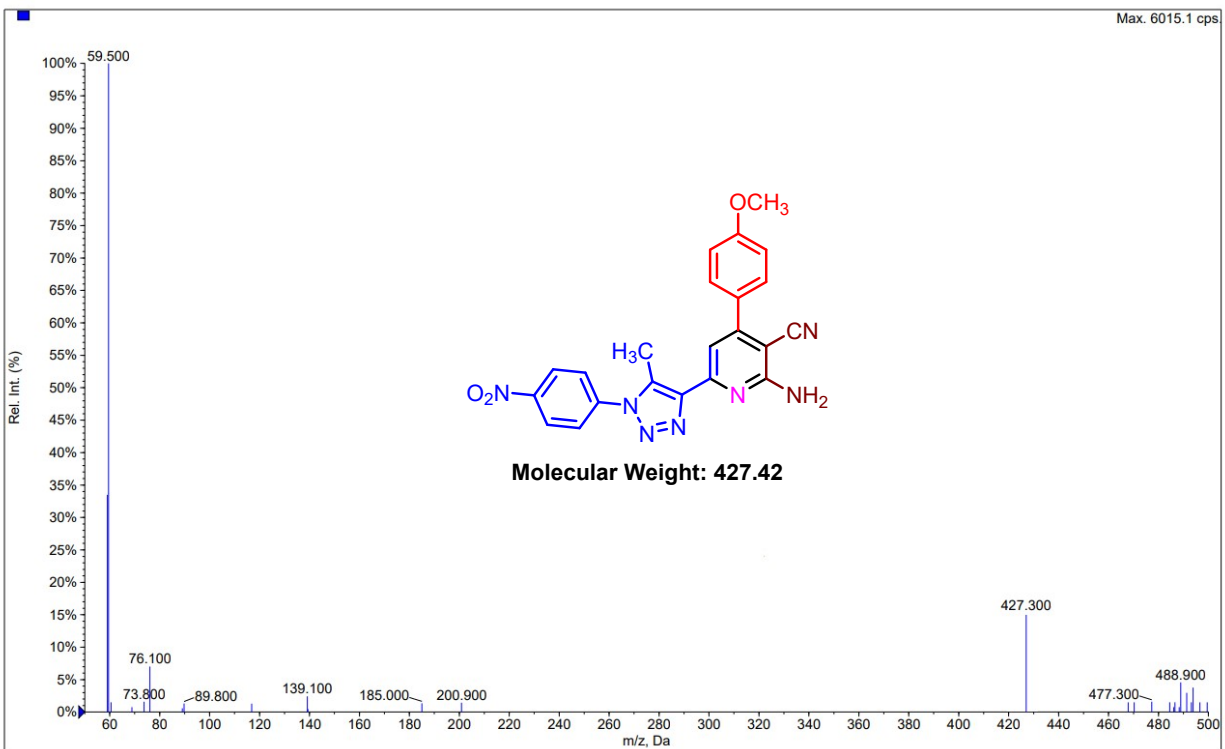
11: ^1H NMR spectrum of 2-amino-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)-4-(4-methoxyphenyl) nicotinonitrile



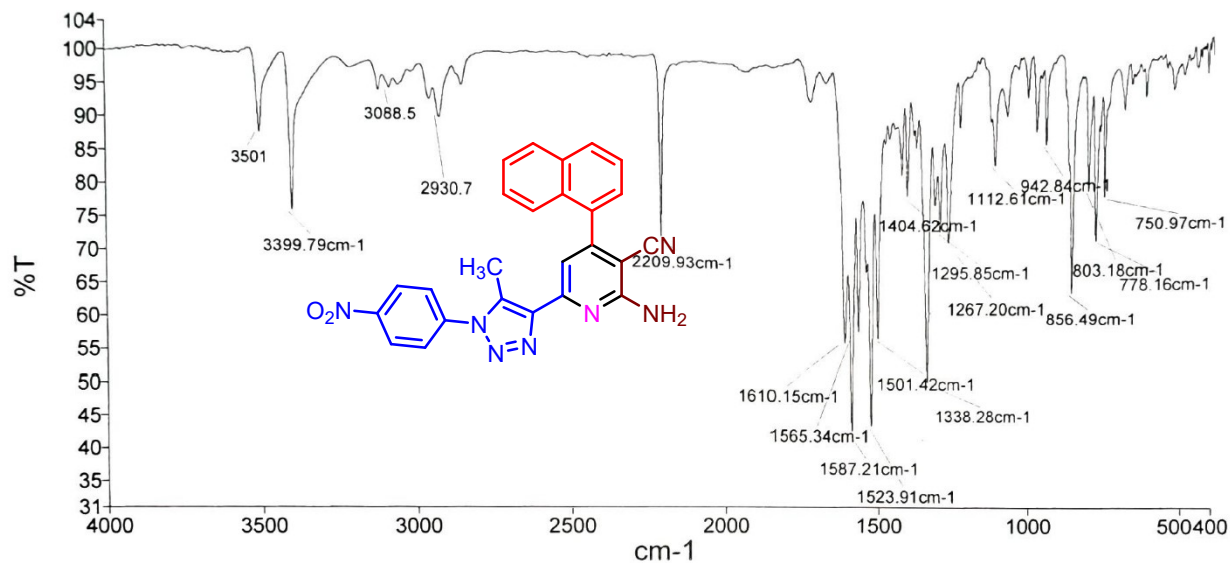
12: ^{13}C NMR spectrum of 2-amino-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)-4-(4-methoxyphenyl) nicotinonitrile



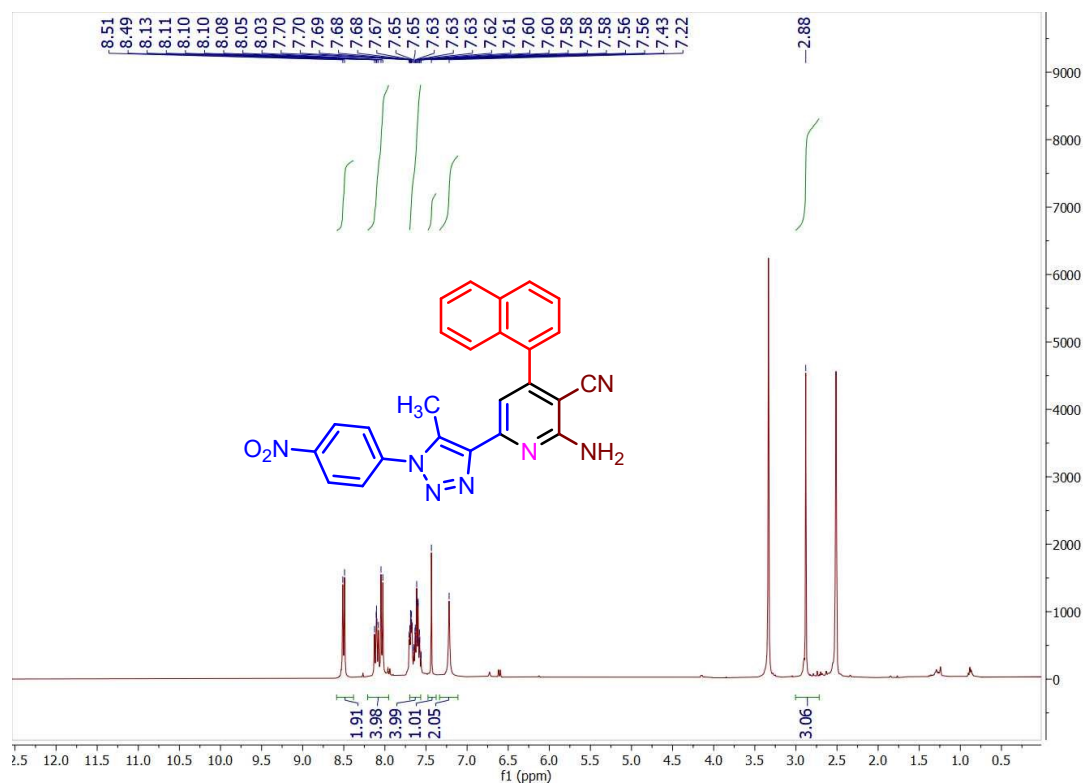
13: Mass spectrum of 2-amino-6-(5-methyl-1-(4-nitrophenyl)-1H-1,2,3-triazol-4-yl)-4-(4-methoxyphenyl) nicotinonitrile



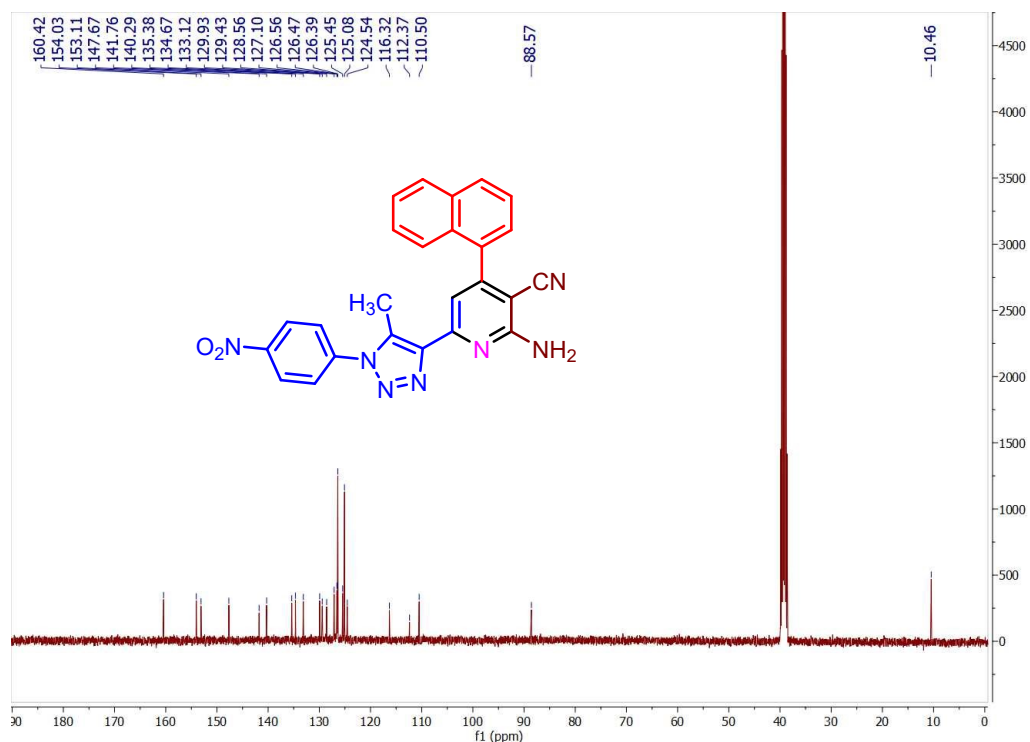
14: FT-IR spectrum of 2-amino-6-(5-methyl-1-(4-nitrophenyl)-1H-1,2,3-triazol-4-yl)-4-(naphthalen-1-yl) nicotinonitrile



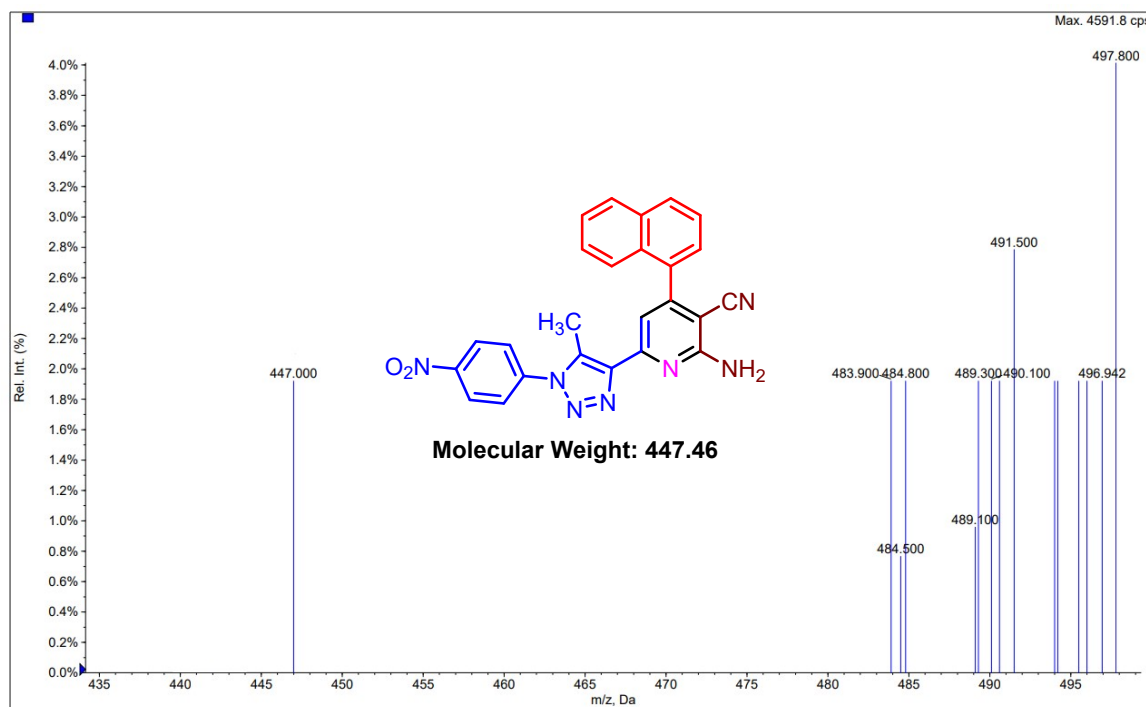
15: ^1H NMR spectrum of 2-amino-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)-4-(naphthalen-1-yl) nicotinonitrile



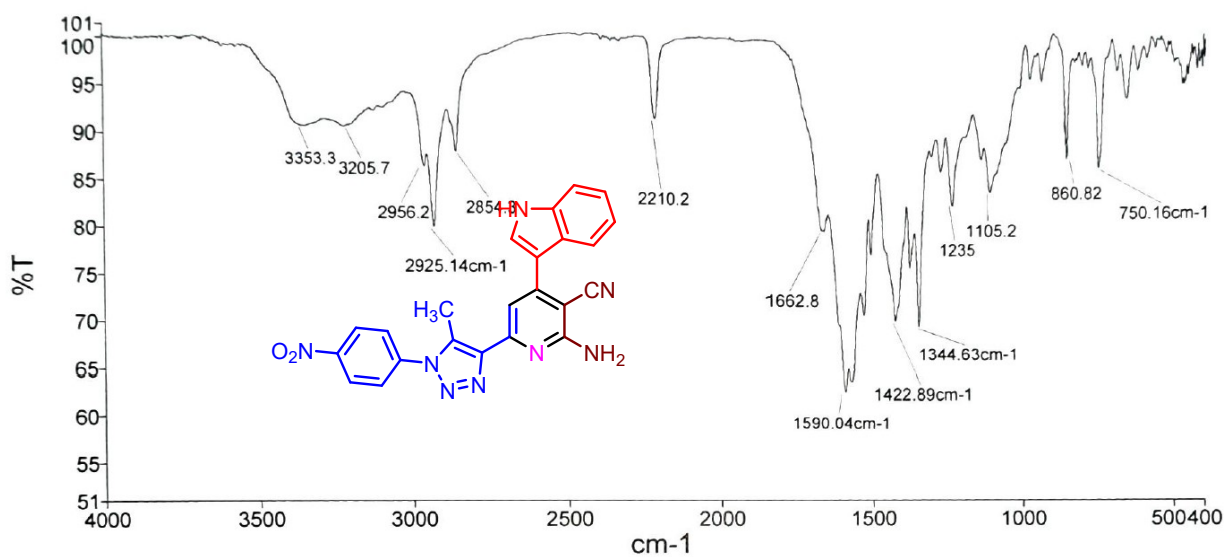
16: ^{13}C NMR spectrum of 2-amino-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl)-4-(naphthalen-1-yl) nicotinonitrile



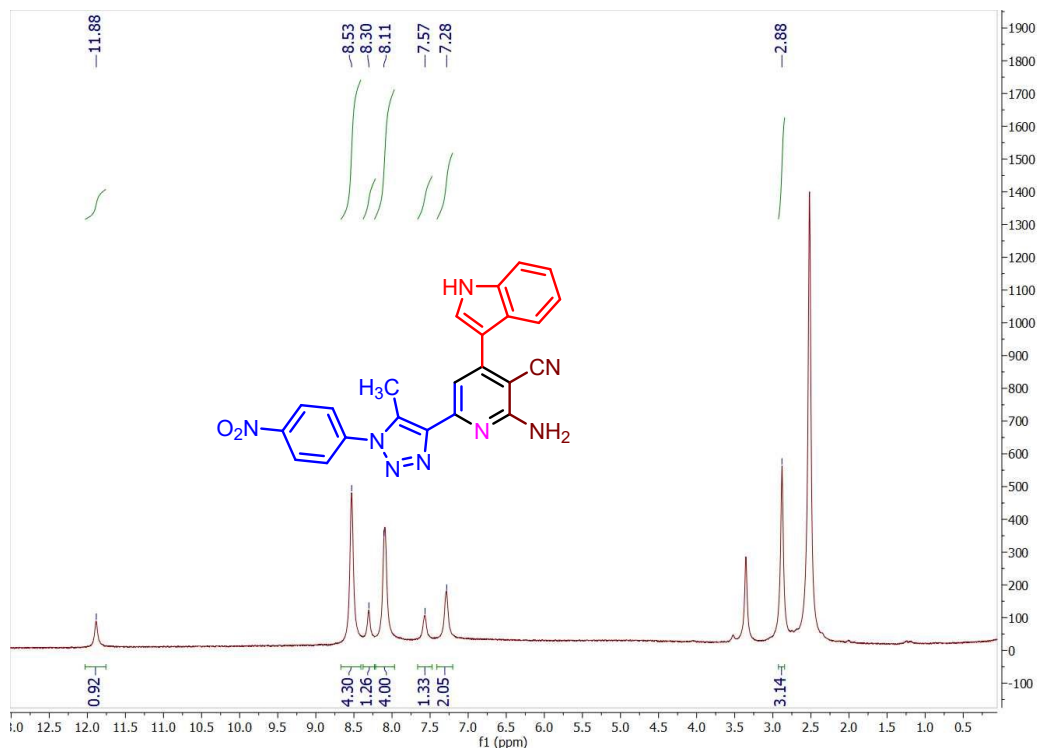
17: Mass spectrum of 2-amino-6-(5-methyl-1-(4-nitrophenyl)-1H-1,2,3-triazol-4-yl)-4-(naphthalen-1-yl) nicotinonitrile



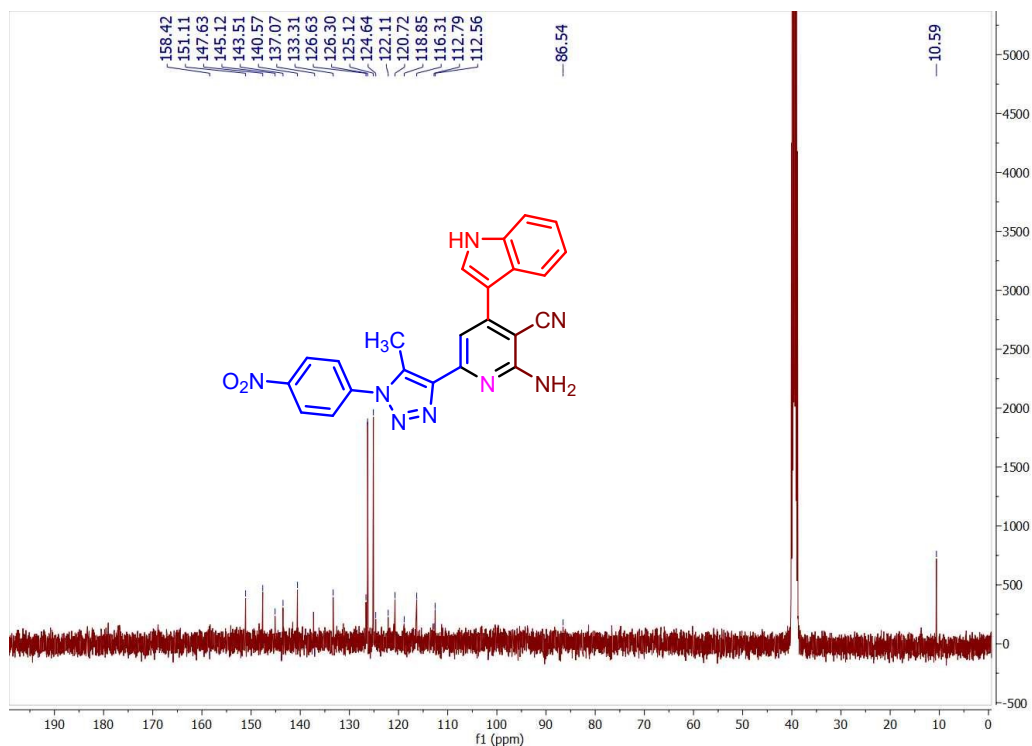
18: FT-IR spectrum of 2-amino-4-(1H-indol-3-yl)-6-(5-methyl-1-(4-nitrophenyl)-1H-1,2,3-triazol-4-yl) nicotinonitrile



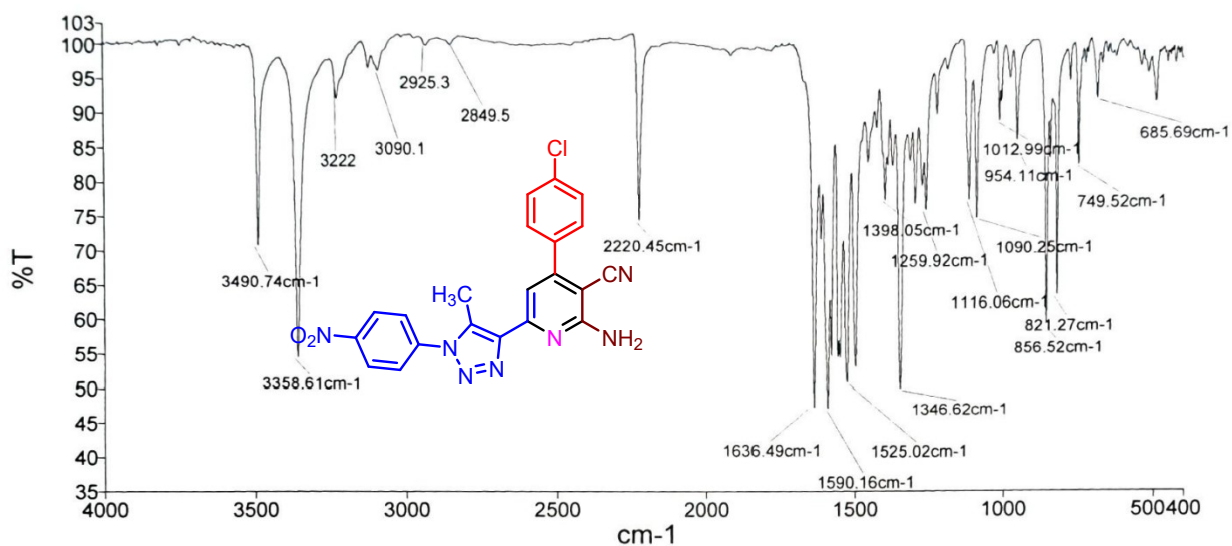
19: ^1H NMR spectrum of 2-amino-4-(1*H*-indol-3-yl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile



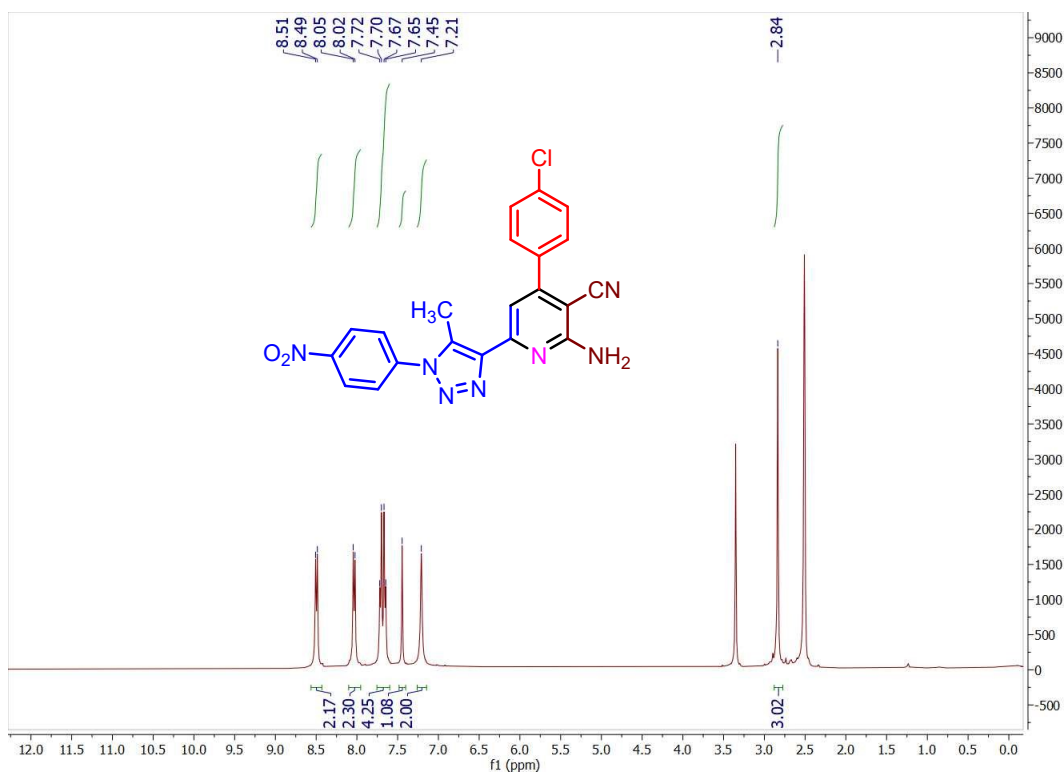
20: ^{13}C NMR spectrum of 2-amino-4-(1*H*-indol-3-yl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile



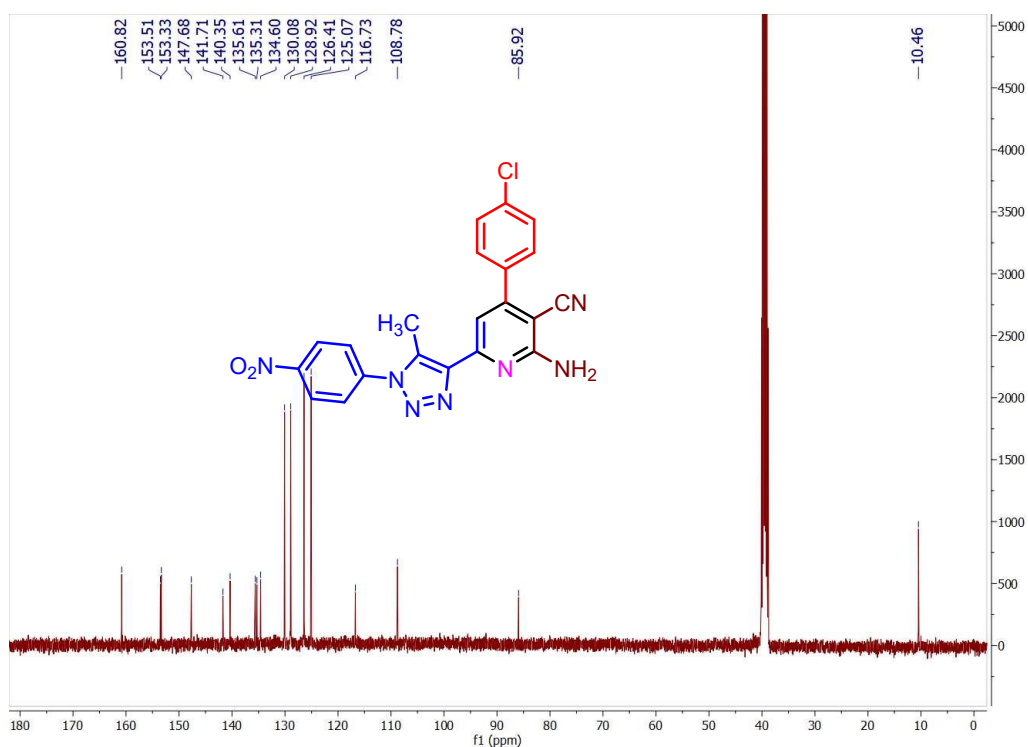
21: FT-IR spectrum of 2-amino-4-(4-chlorophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile



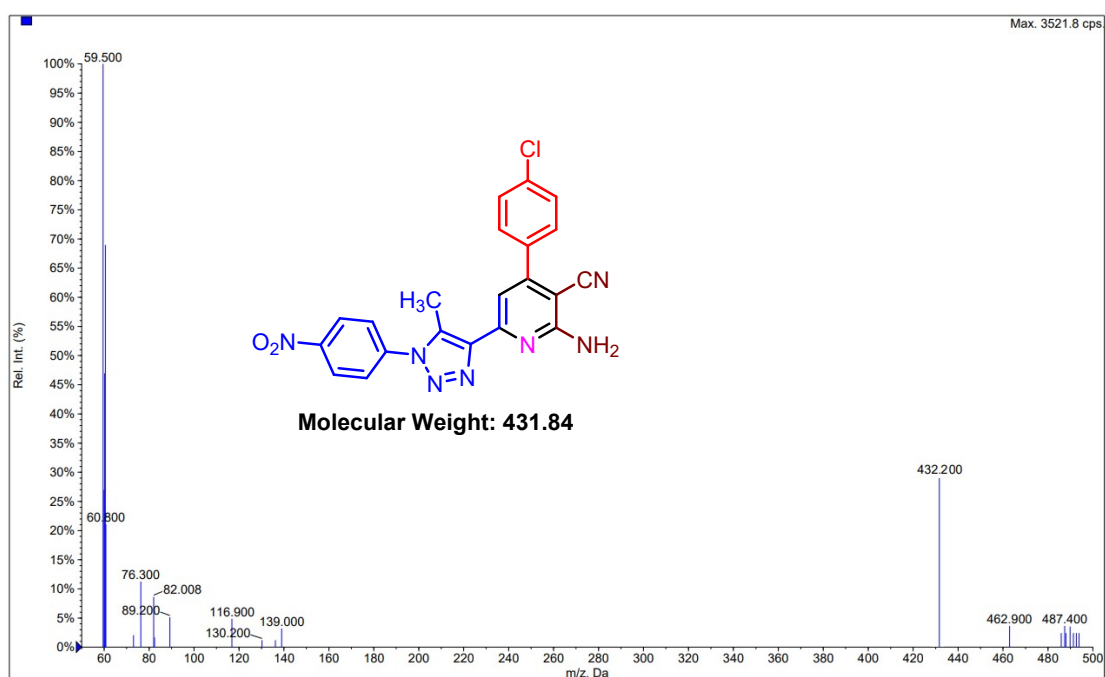
22: ¹H NMR spectrum of 2-amino-4-(4-chlorophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile



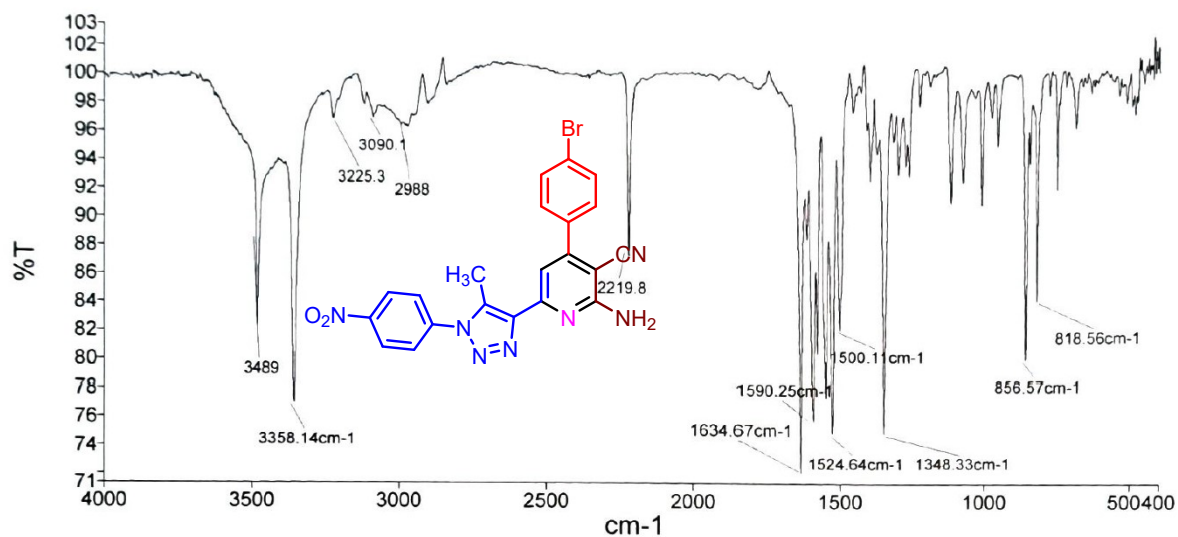
23: ^{13}C NMR spectrum of 2-amino-4-(4-chlorophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile



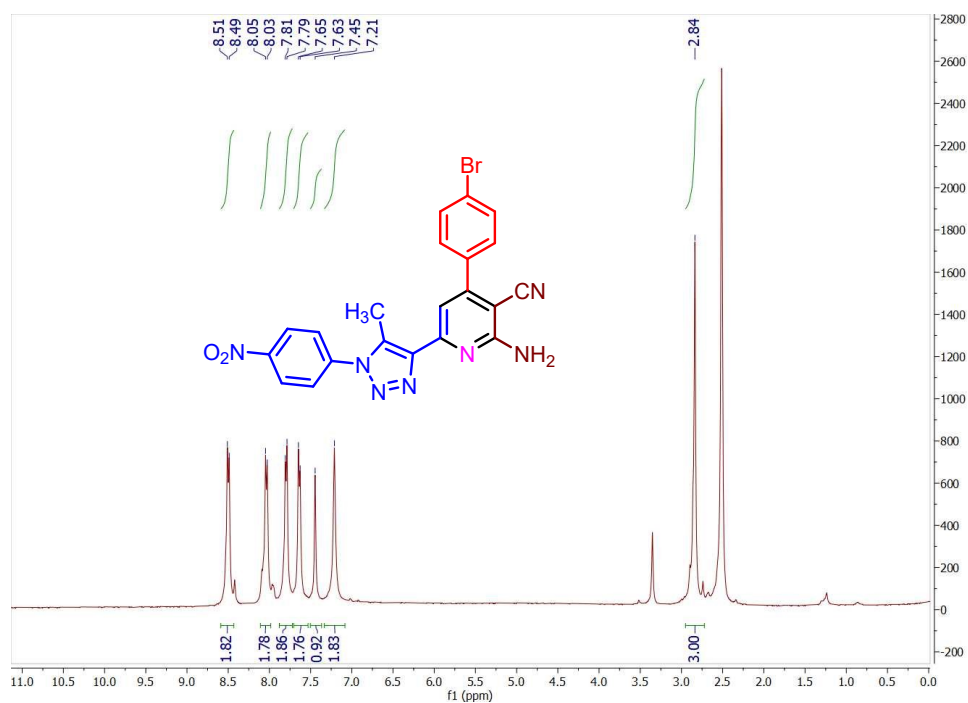
24: Mass spectrum of 2-amino-4-(4-chlorophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile



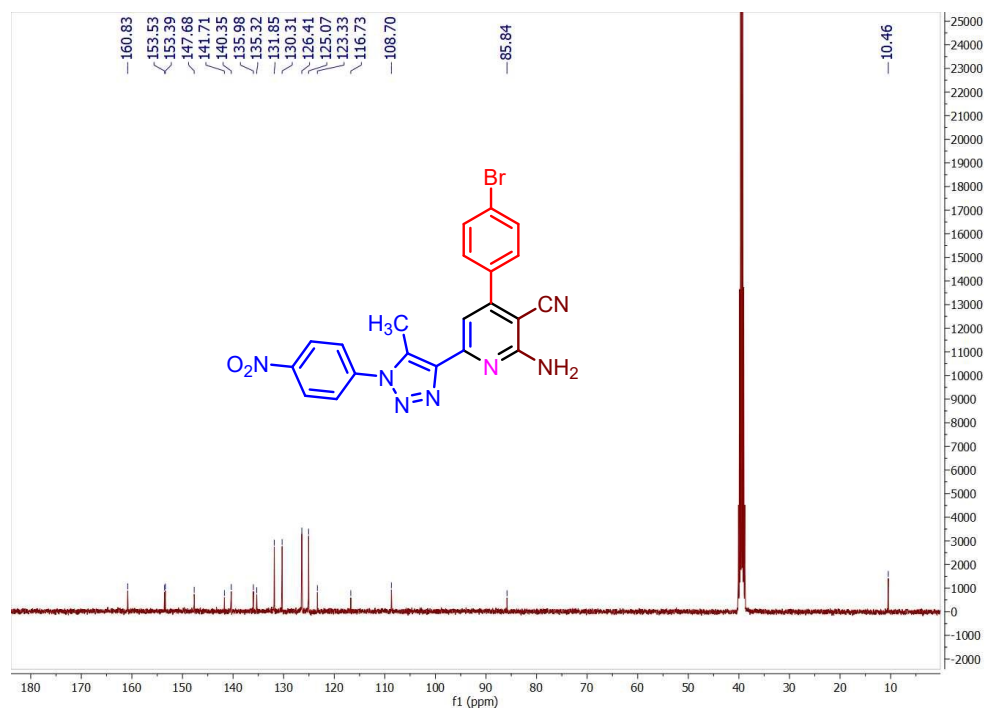
25: FT-IR spectrum of 2-amino-4-(4-bromophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1H-1,2,3-triazol-4-yl) nicotinonitrile



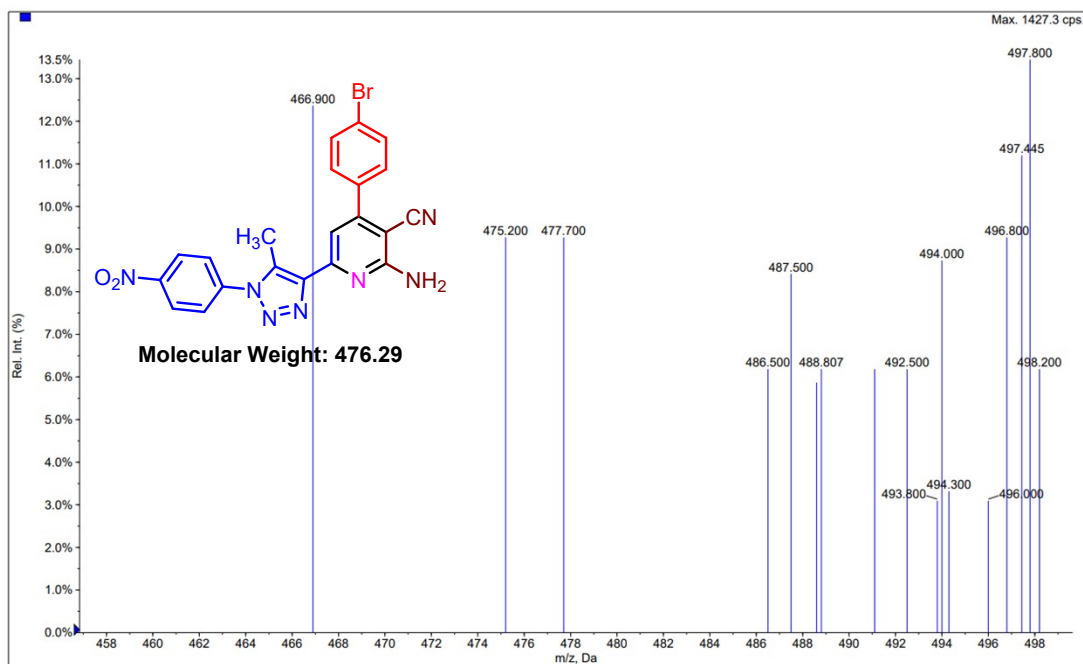
26: ¹H NMR spectrum of 2-amino-4-(4-bromophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1H-1,2,3-triazol-4-yl) nicotinonitrile



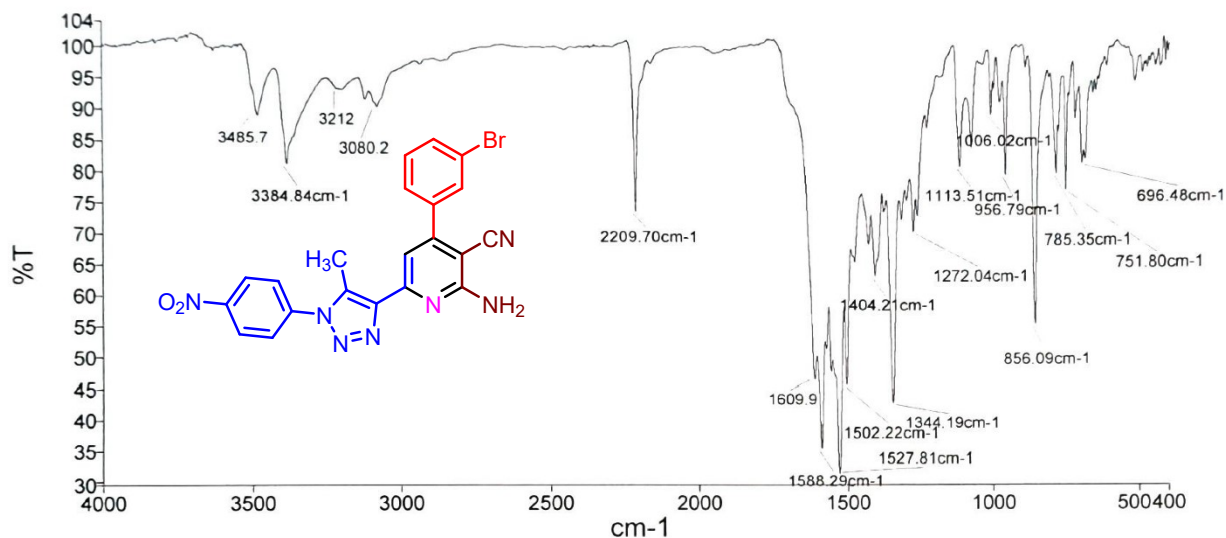
27: ^{13}C NMR spectrum of 2-amino-4-(4-bromophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile



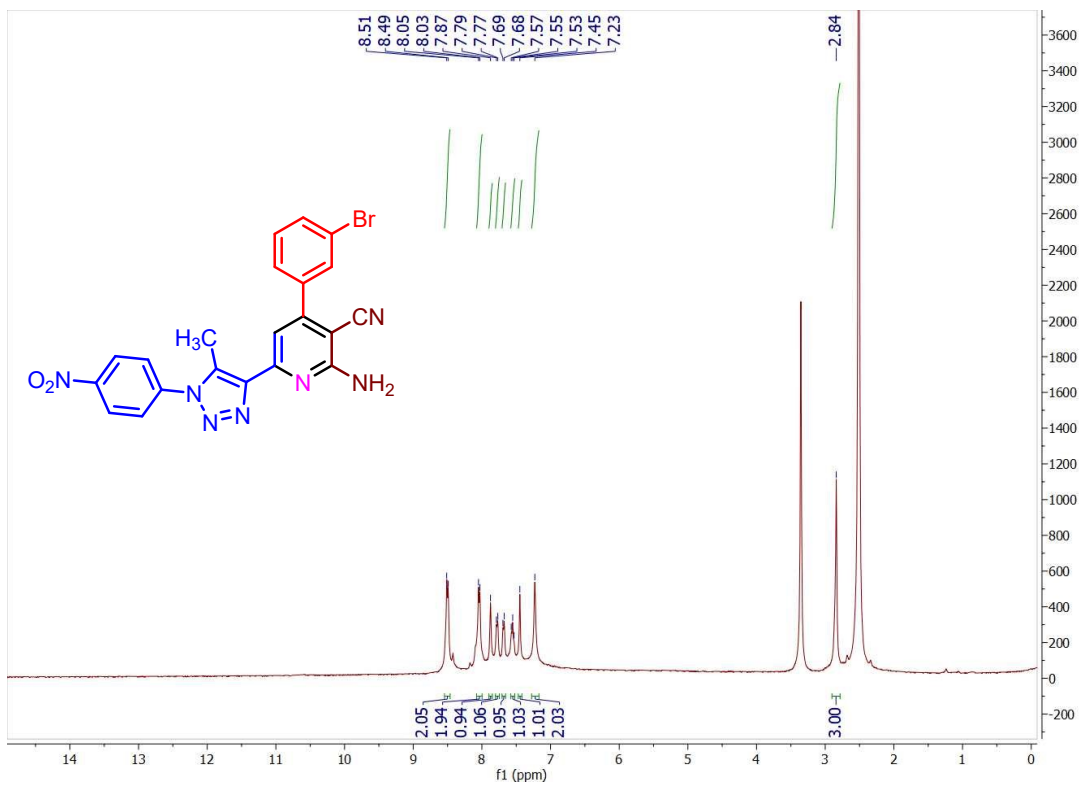
28: Mass spectrum of 2-amino-4-(4-bromophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile



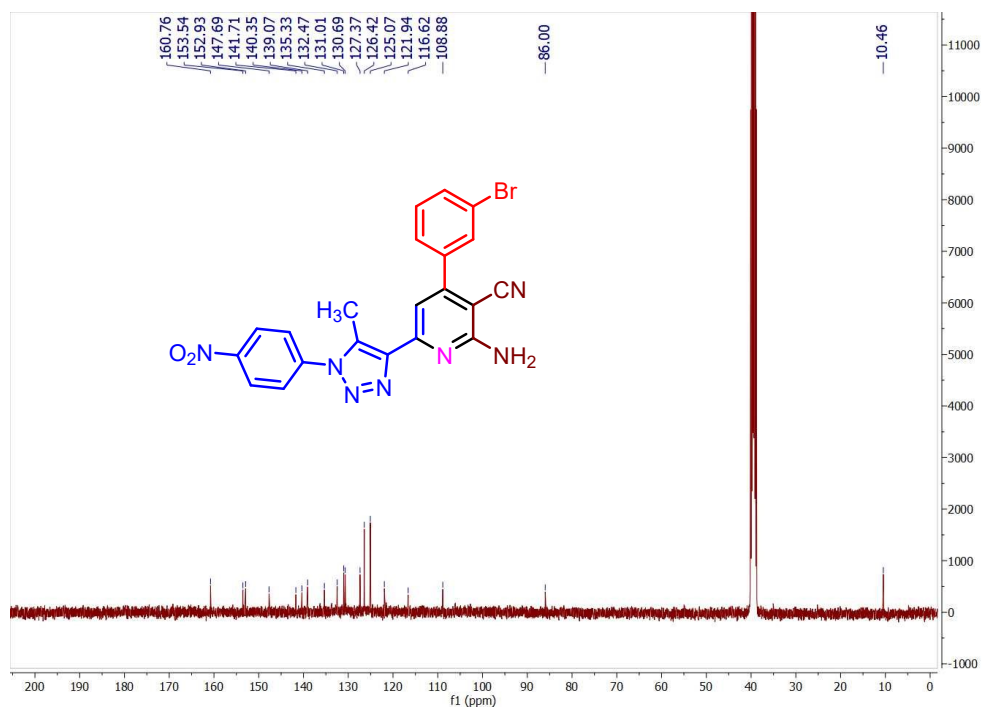
29: FT-IR spectrum of 2-amino-4-(3-bromophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1H-1,2,3-triazol-4-yl) nicotinonitrile



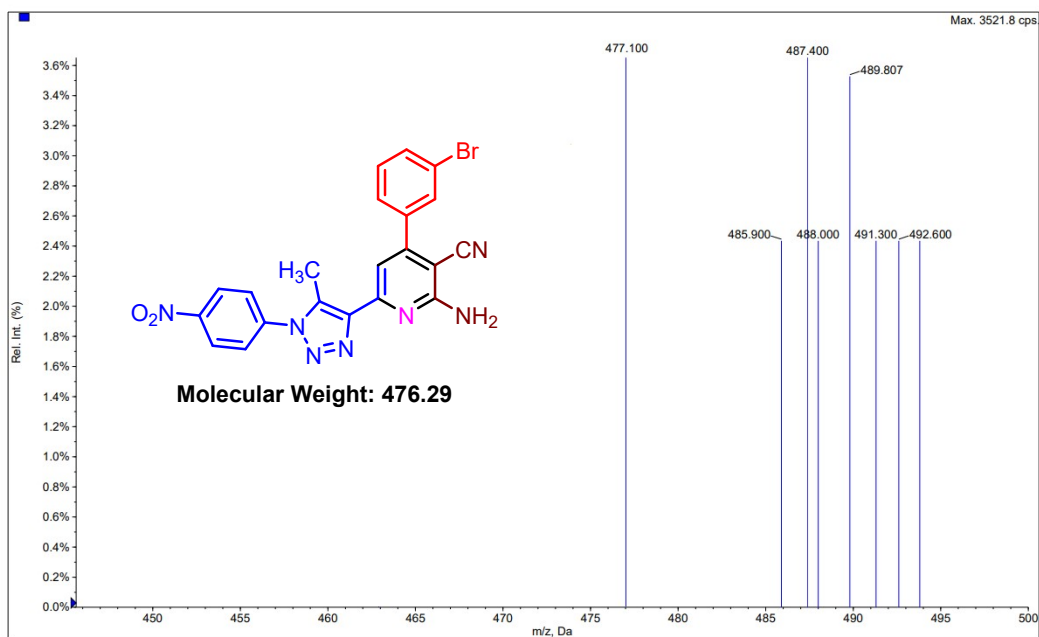
30: ¹H NMR spectrum of 2-amino-4-(3-bromophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1H-1,2,3-triazol-4-yl) nicotinonitrile



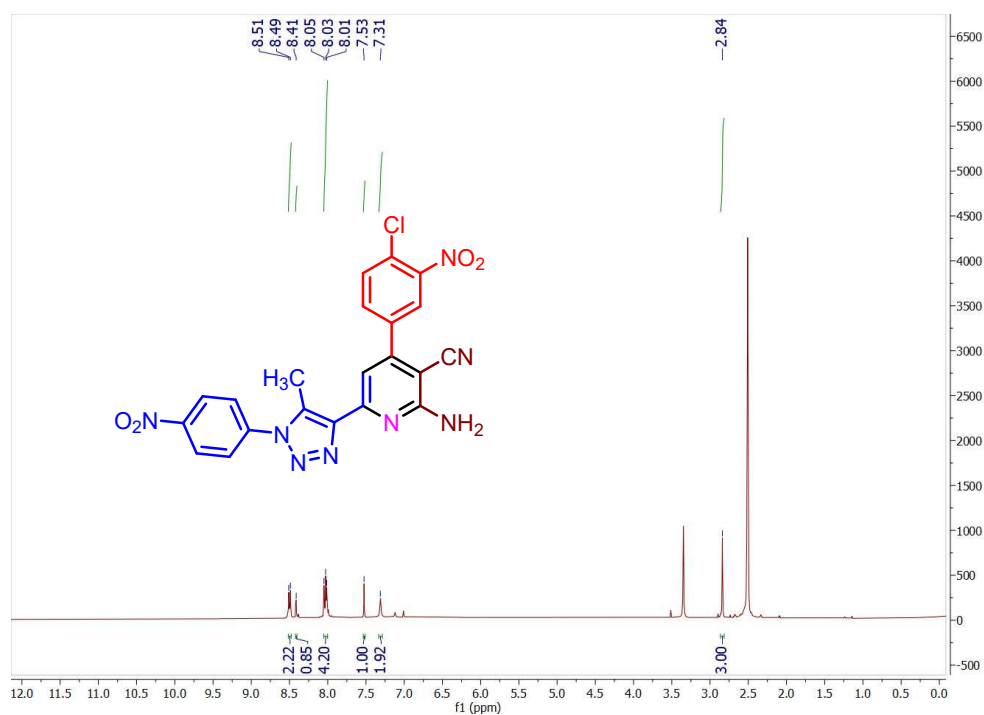
31: ^{13}C NMR spectrum of 2-amino-4-(3-bromophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile



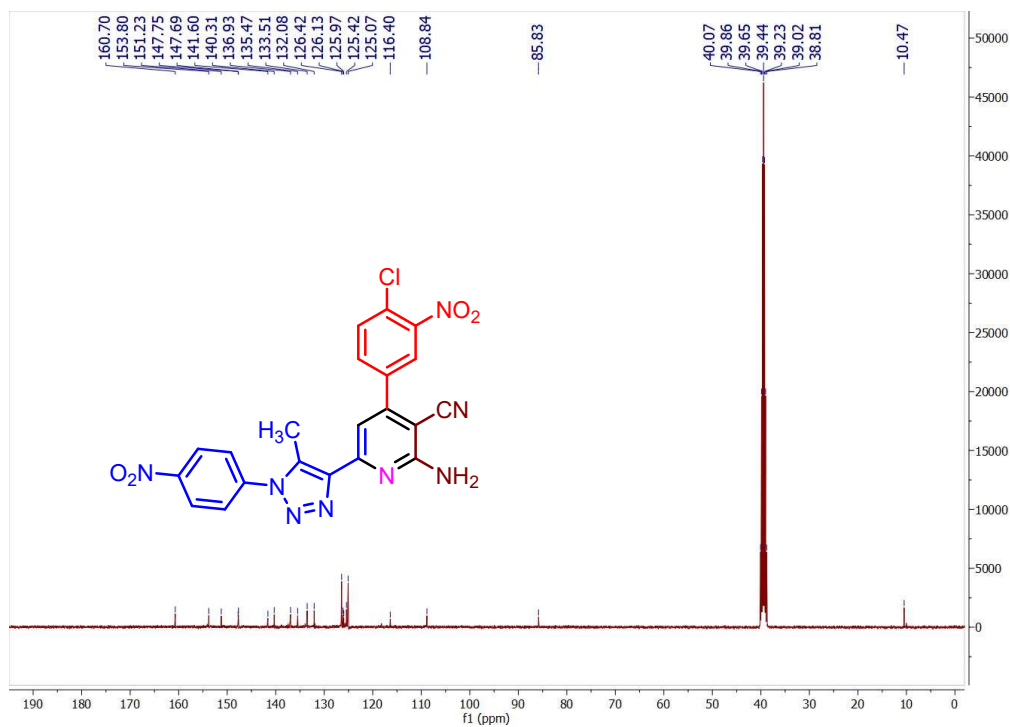
32: Mass spectrum of 2-amino-4-(3-bromophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile



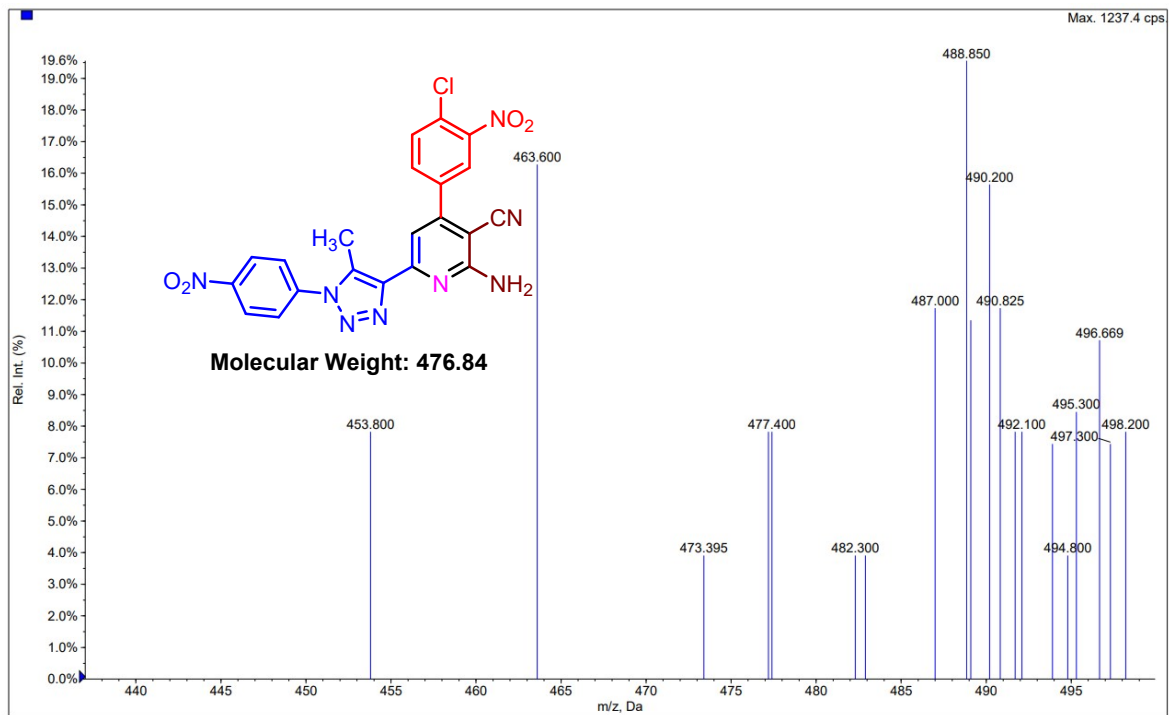
33: ¹H NMR spectrum of 2-amino-4-(4-chloro-3-nitrophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1H-1,2,3-triazol-4-yl) nicotinonitrile



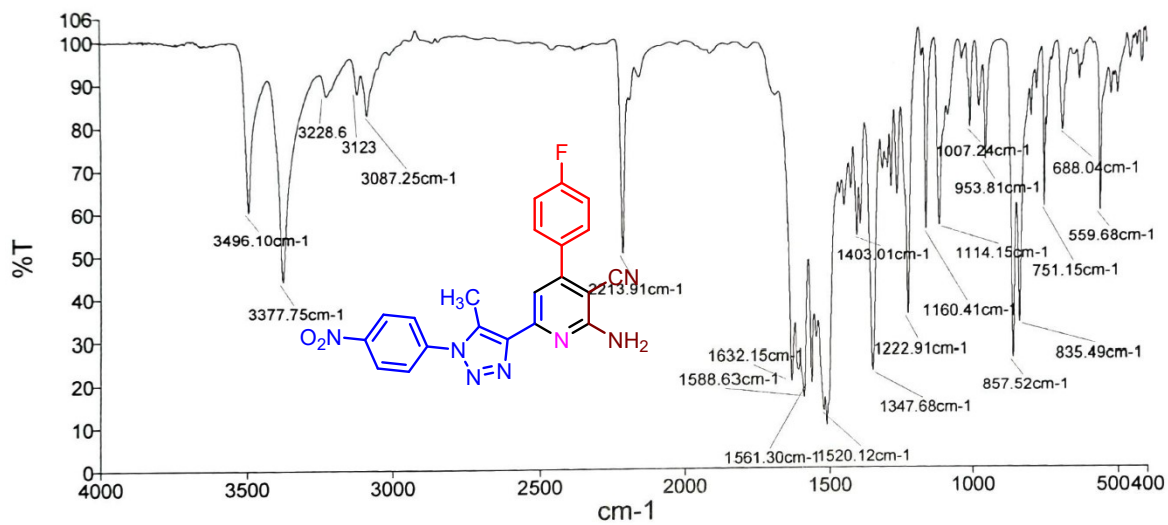
34: ¹³C NMR spectrum of 2-amino-4-(4-chloro-3-nitrophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1H-1,2,3-triazol-4-yl) nicotinonitrile



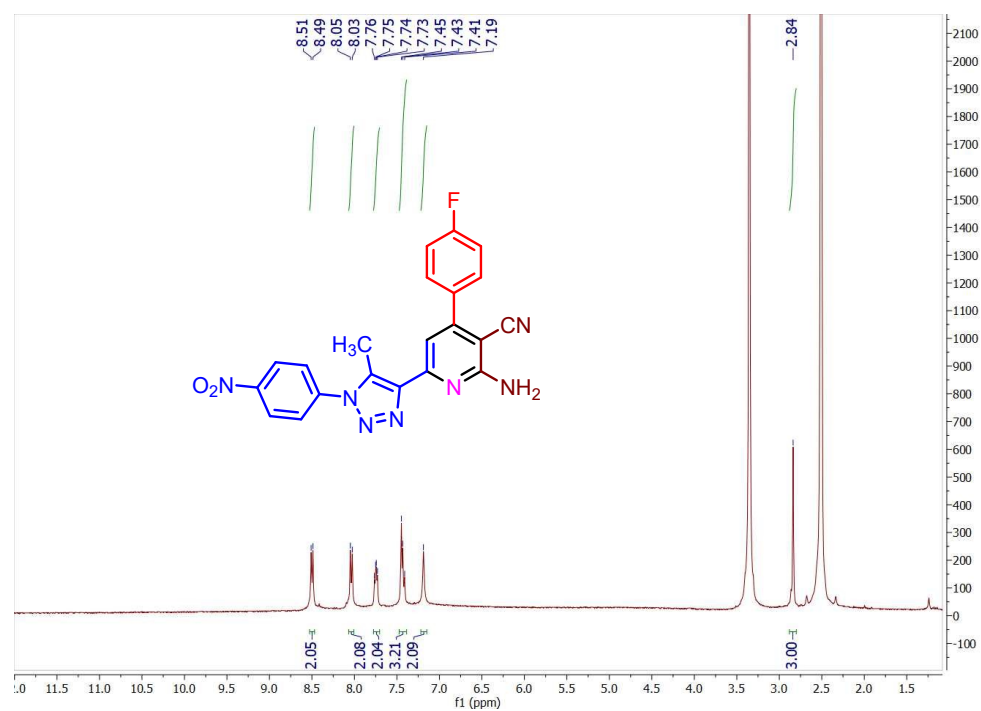
35: Mass spectrum of 2-amino-4-(4-chloro-3-nitrophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1H-1,2,3-triazol-4-yl) nicotinonitrile



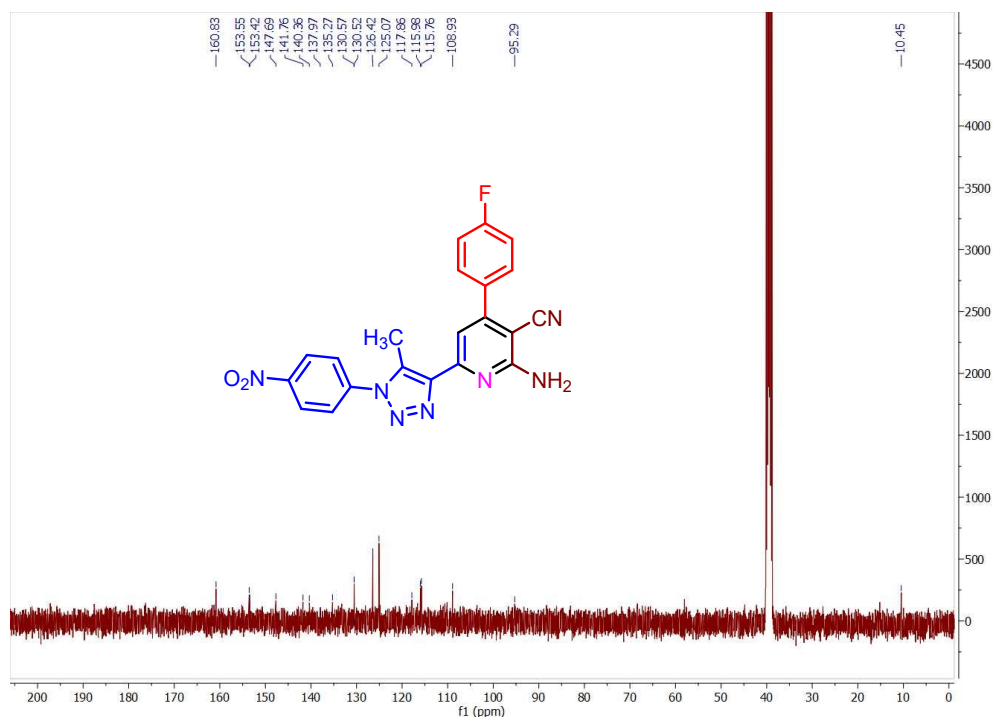
36: FT-IR spectrum of 2-amino-4-(4-fluorophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1H-1,2,3-triazol-4-yl) nicotinonitrile



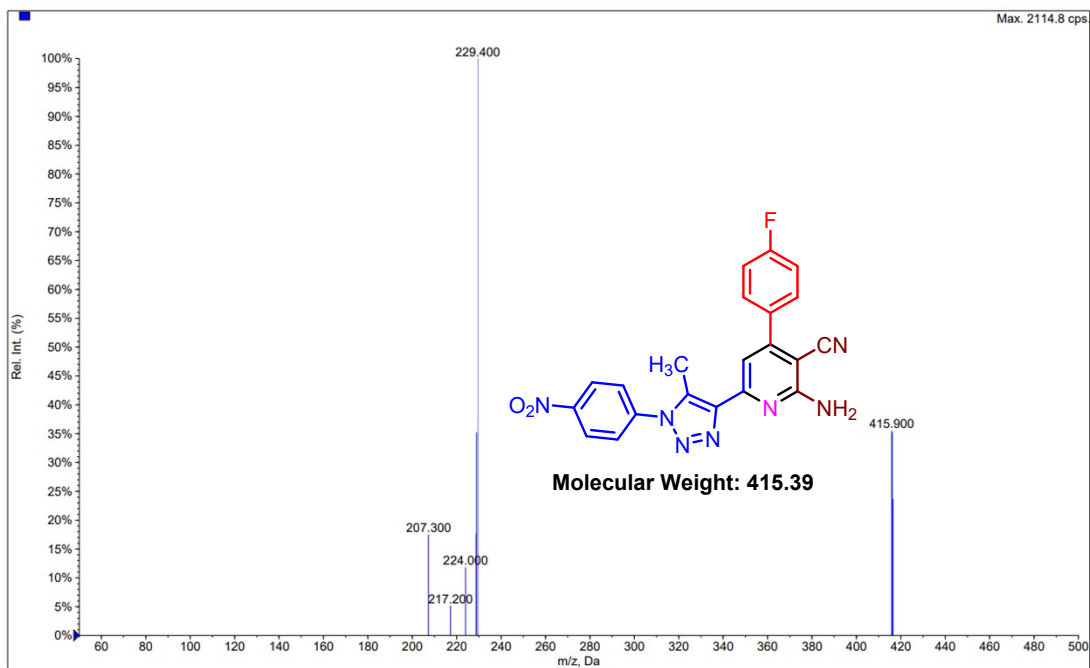
37: ^1H NMR spectrum of 2-amino-4-(4-fluorophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile



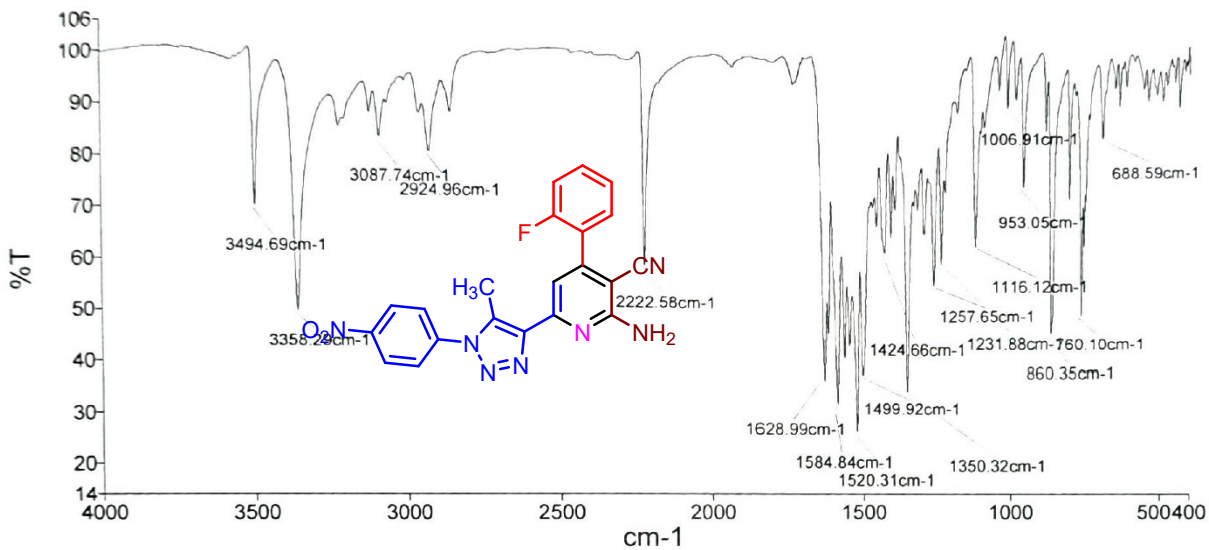
38: ^{13}C NMR spectrum of 2-amino-4-(4-fluorophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile



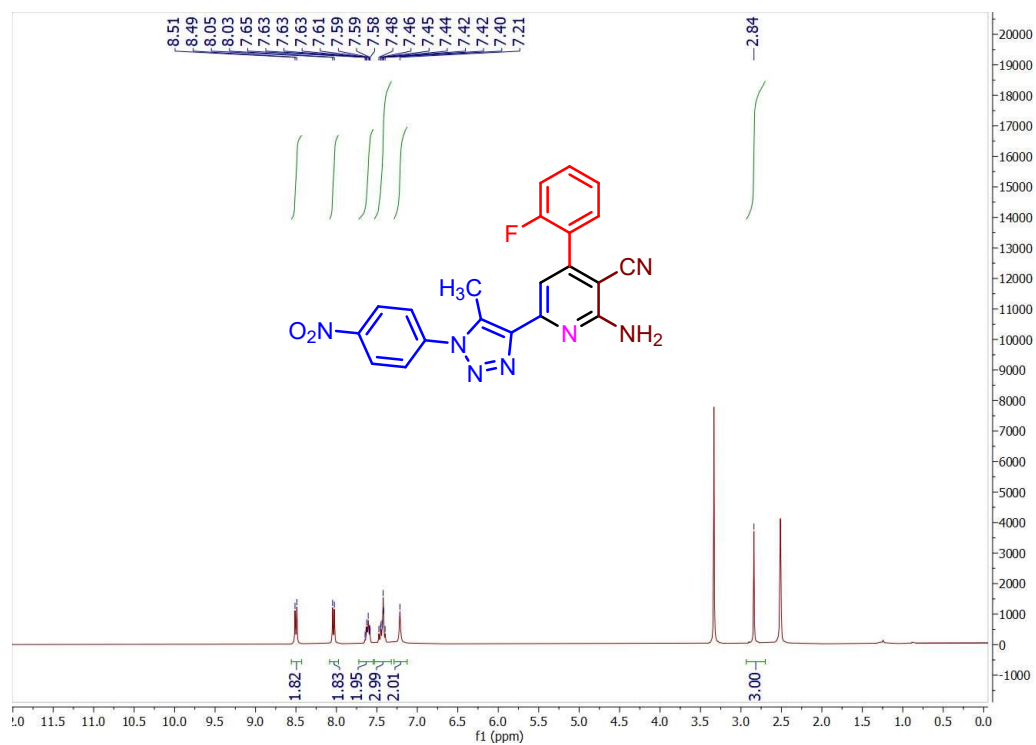
39: Mass spectrum of 2-amino-4-(4-fluorophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1H-1,2,3-triazol-4-yl) nicotinonitrile



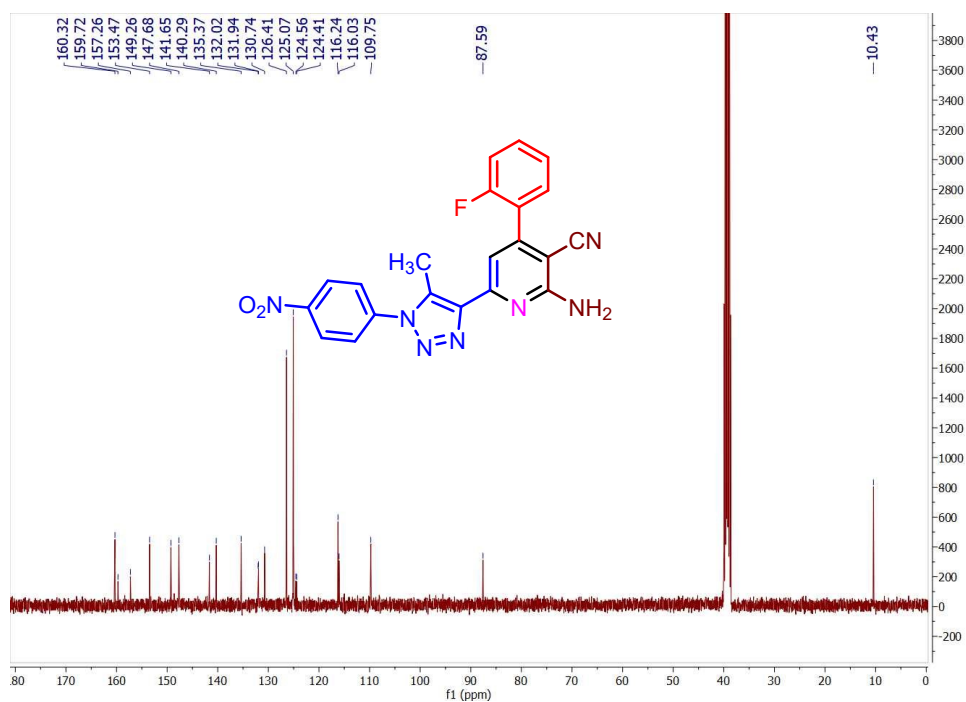
40: FT-IR spectrum of 2-amino-4-(2-fluorophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1H-1,2,3-triazol-4-yl) nicotinonitrile



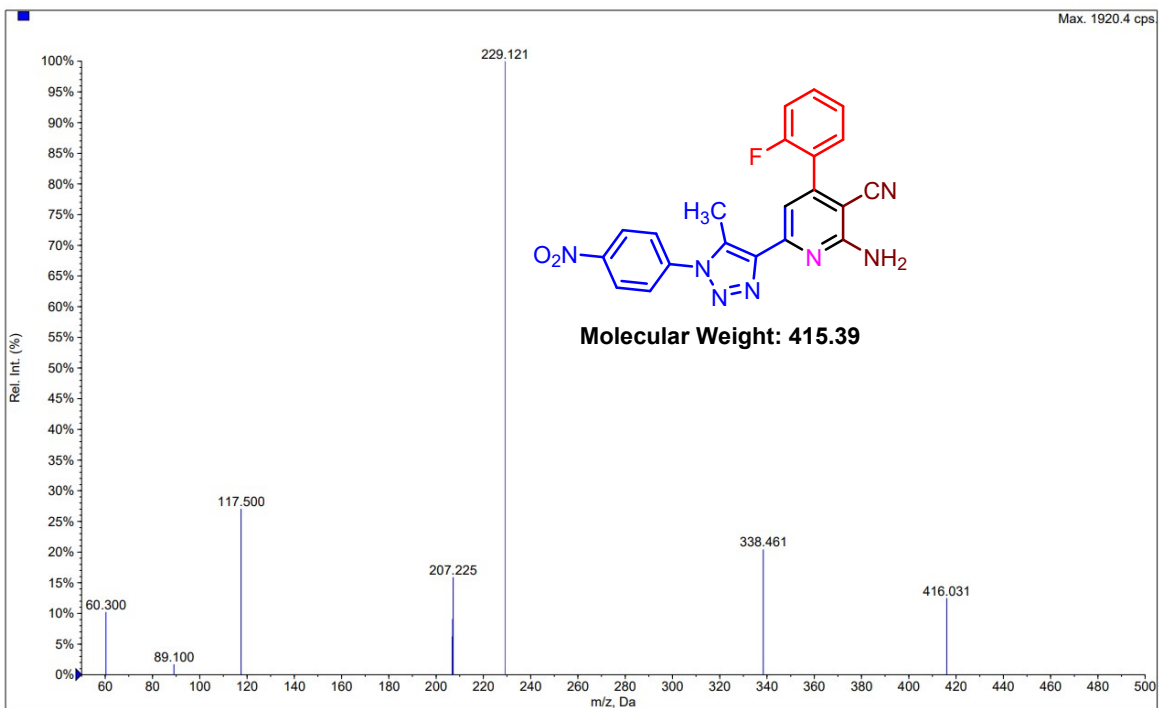
41: ^1H NMR spectrum of 2-amino-4-(2-fluorophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile



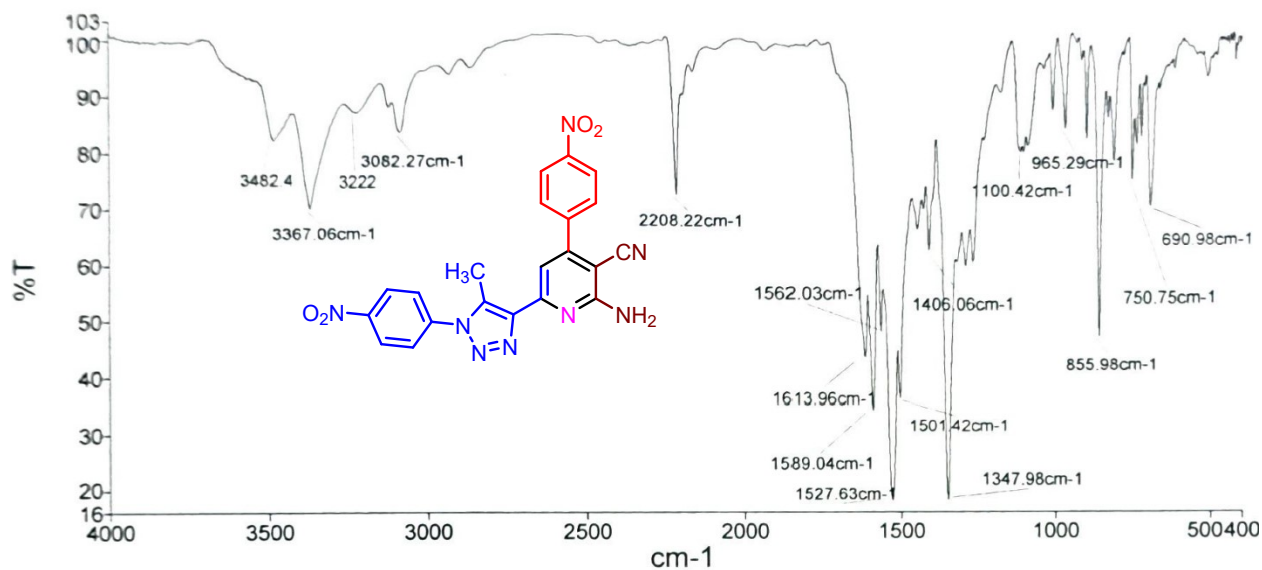
42: ^{13}C NMR spectrum of 2-amino-4-(2-fluorophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile



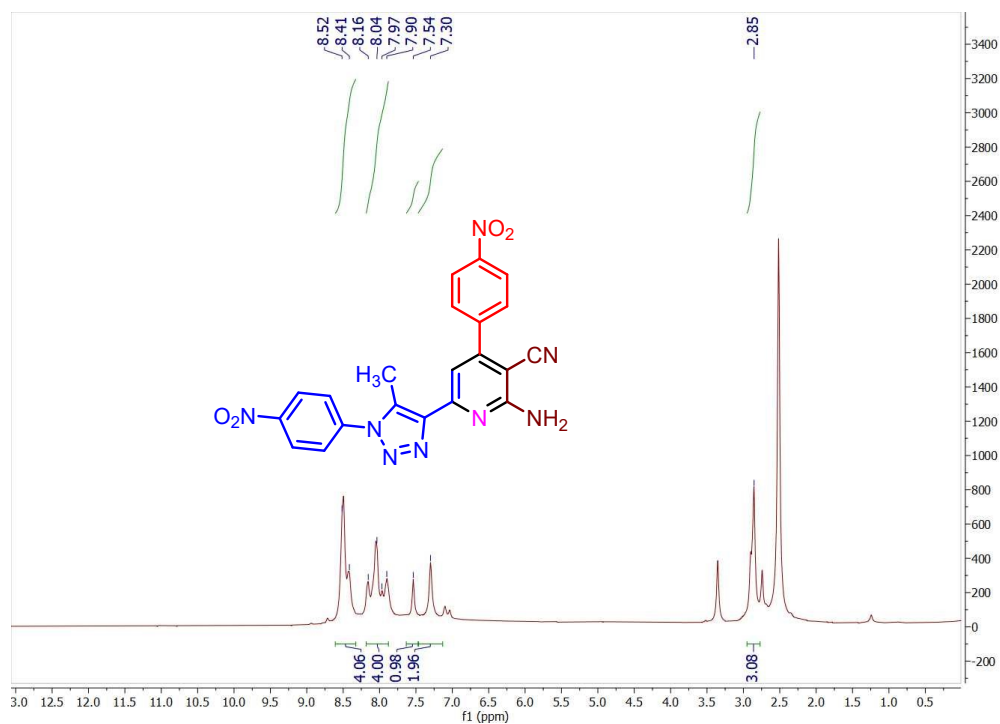
43: Mass spectrum of 2-amino-4-(2-florophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1H-1,2,3-triazol-4-yl) nicotinonitrile



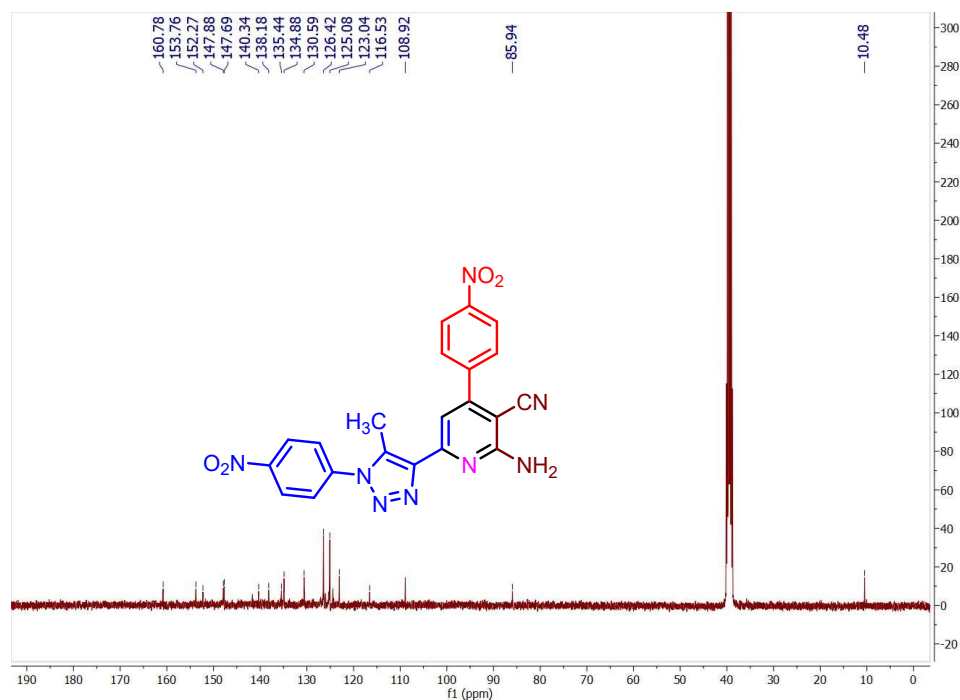
44: FT-IR spectrum of 2-amino-6-(5-methyl-1-(4-nitrophenyl)-1H-1,2,3-triazol-4-yl)-4-(4-nitrophenyl) nicotinonitrile



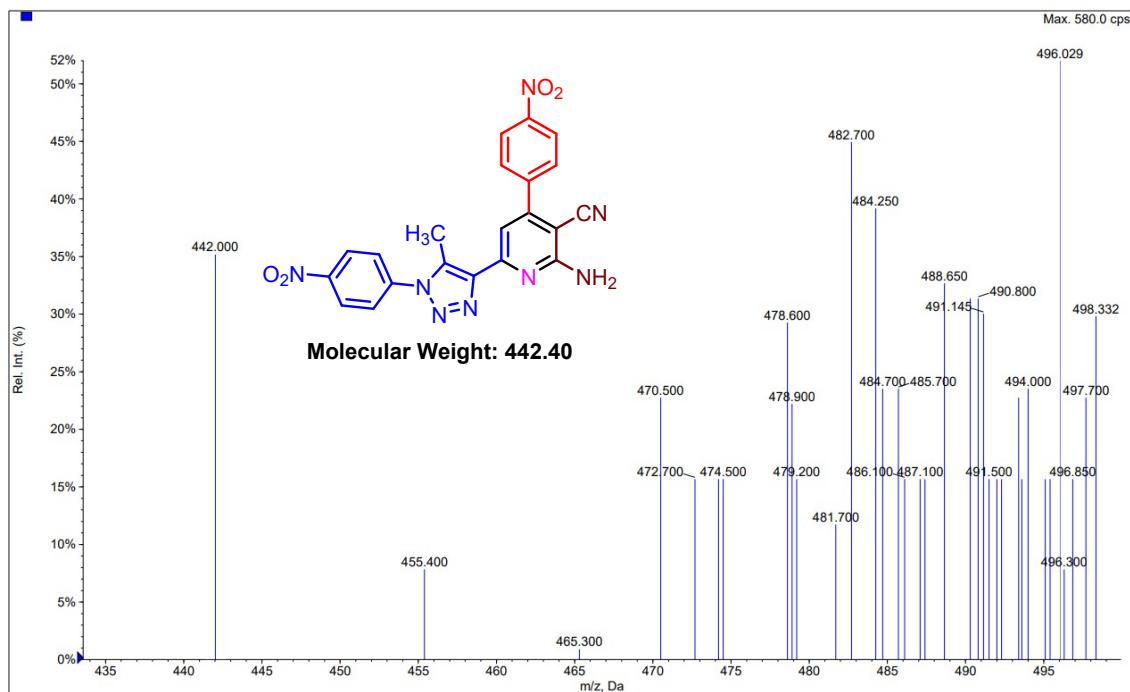
45: ^1H NMR spectrum of 2-amino-6-(5-methyl-1-(4-nitrophenyl)-1H-1,2,3-triazol-4-yl)-4-(4-nitrophenyl) nicotinonitrile



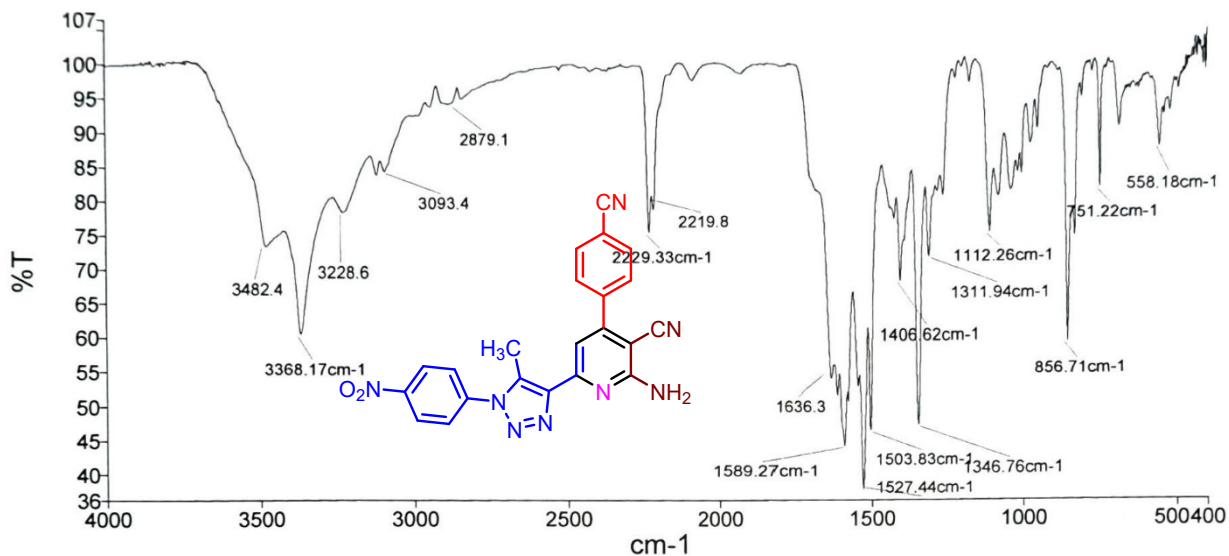
46: ^{13}C NMR spectrum of 2-amino-6-(5-methyl-1-(4-nitrophenyl)-1H-1,2,3-triazol-4-yl)-4-(4-nitrophenyl) nicotinonitrile



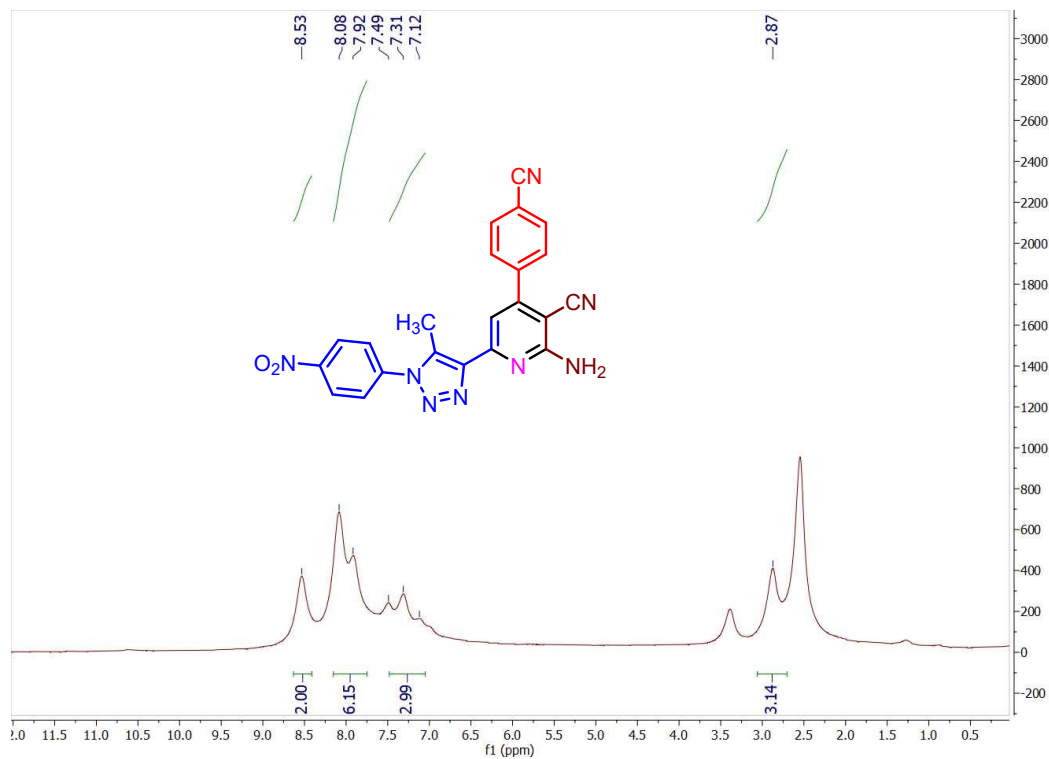
47: Mass spectrum of 2-amino-6-(5-methyl-1-(4-nitrophenyl)-1H-1,2,3-triazol-4-yl)-4-(4-nitrophenyl) nicotinonitrile



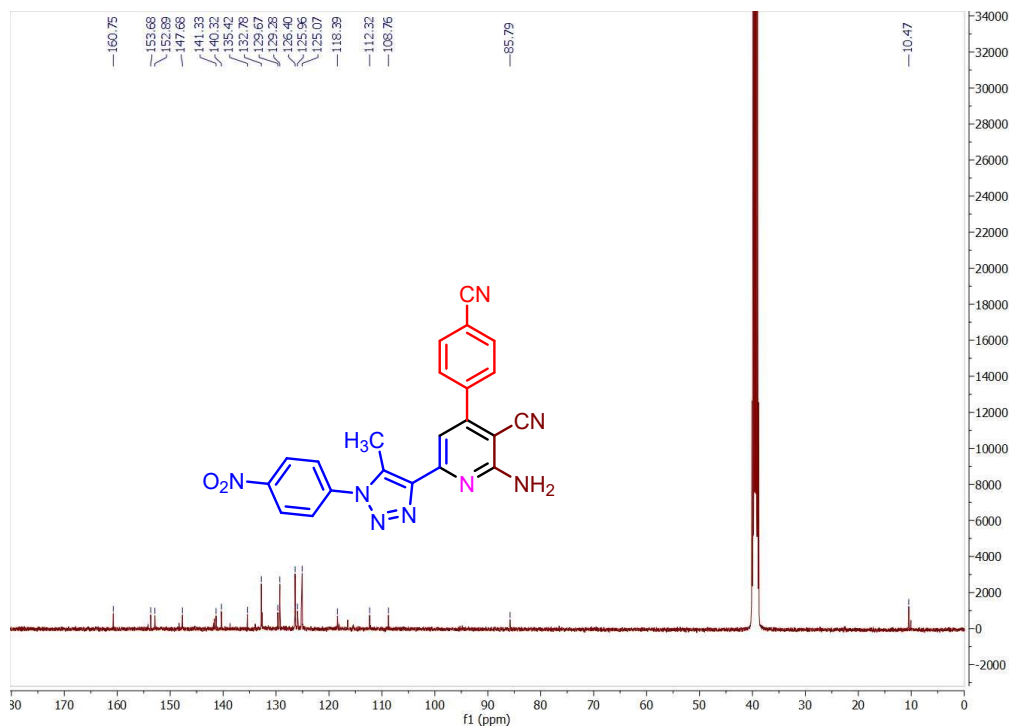
48: FT-IR spectrum of 2-amino-4-(4-cyanophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1H-1,2,3-triazol-4-yl) nicotinonitrile



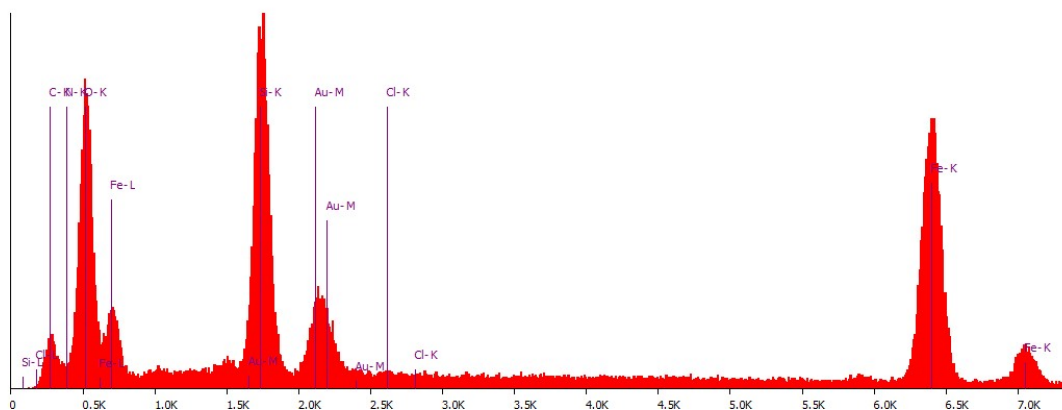
49: ¹H NMR spectrum of 2-amino-4-(4-cyanophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile



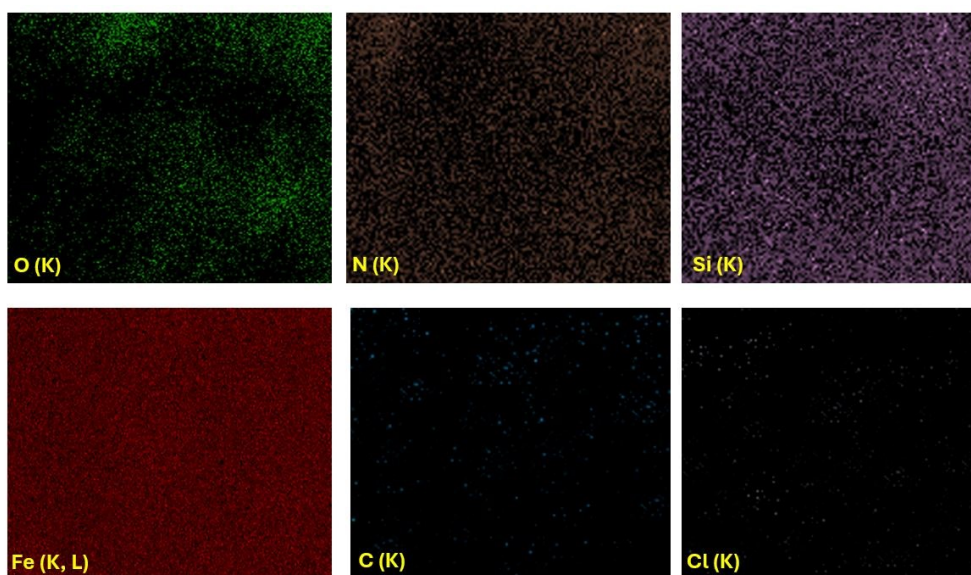
50: ¹³C NMR spectrum of 2-amino-4-(4-cyanophenyl)-6-(5-methyl-1-(4-nitrophenyl)-1*H*-1,2,3-triazol-4-yl) nicotinonitrile



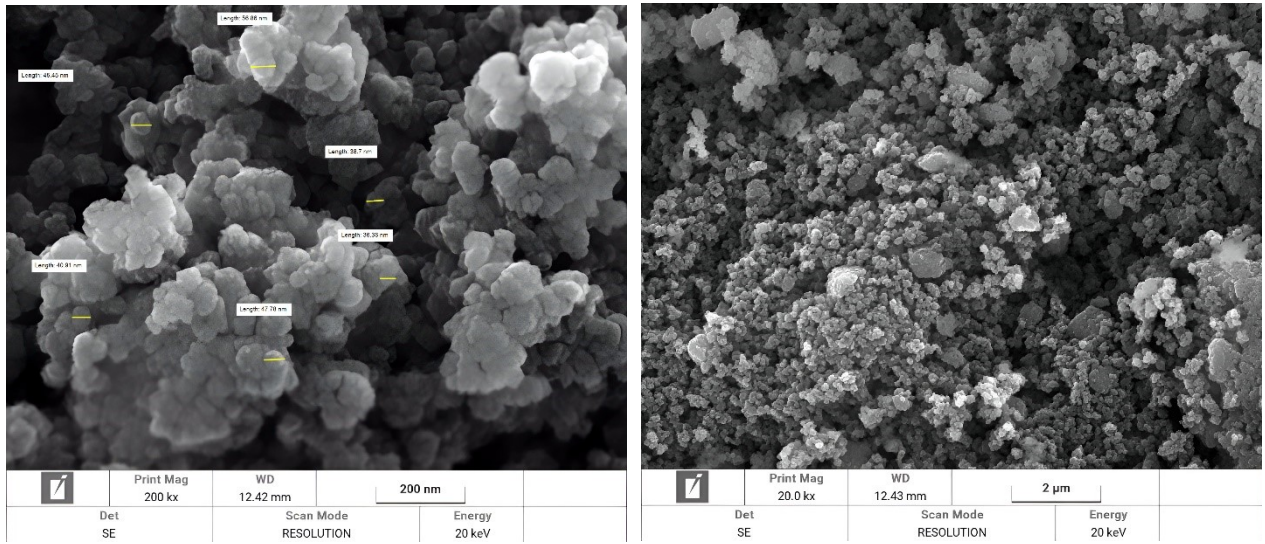
51: EDS signals of recovered $\text{Fe}_3\text{O}_4@\text{SiO}_2@\text{DES1}$



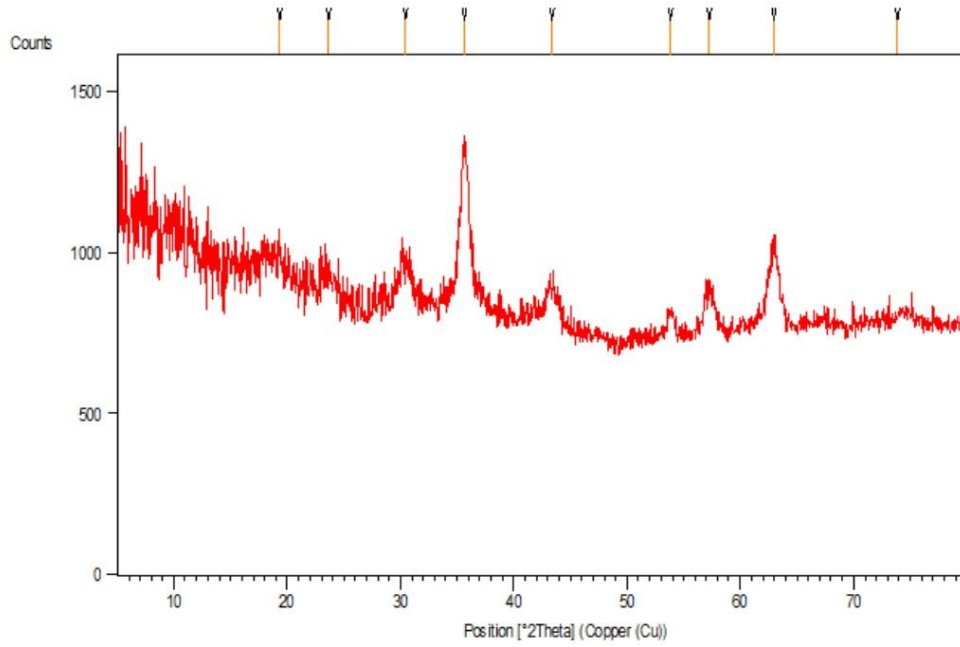
52: Mapping analysis of recovered $\text{Fe}_3\text{O}_4@\text{SiO}_2@\text{DES1}$



53: FESEM images of recovered $\text{Fe}_3\text{O}_4@\text{SiO}_2@\text{DES1}$



54: XRD pattern of recovered $\text{Fe}_3\text{O}_4@\text{SiO}_2@\text{DES1}$



55: The N₂ adsorption-desorption isotherms of Fe₃O₄@SiO₂@DES1 (a) and recovered Fe₃O₄@SiO₂@DES1 (b)

