

Efficient synthesis and characterization of new ligand and their transition metal complexes derived from 4-methyl-1,2,3-thiadiazoles-5-carboxylic acid hydrazide

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¹H NMR Spectra of ligand and their metal complexes

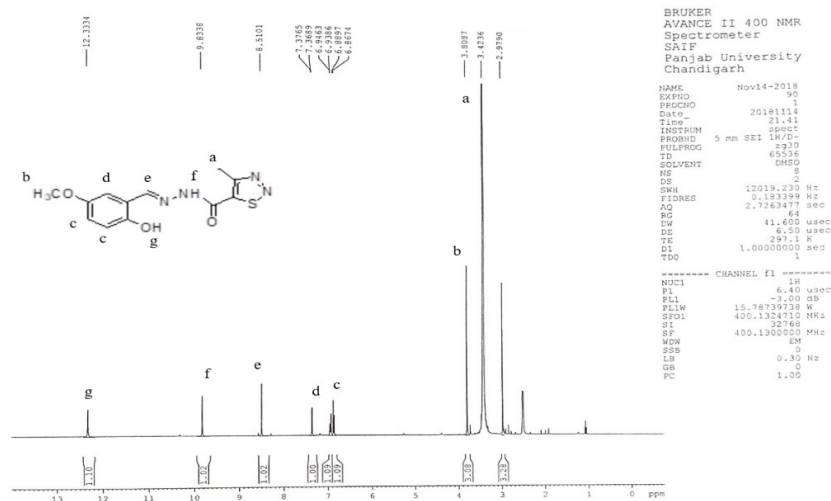


Fig.1. ¹H NMR spectra of the ligand HL (3)

¹H NMR (400 MHz, DMSO-*d*₆) = δ 2.97 (3H^a, CH₃), δ 3.80 (3H^b, s, -OCH₃), δ 6.86-6.94 (2H, dd, Ar-H^c), δ 7.37 (1H, s, Ar-H^d), δ 8.51 (1H^e, s, -CH=N), δ 9.83 (1H^f, s, -CONH), δ 12.33 (1H^g, s, Phenolic -OH).

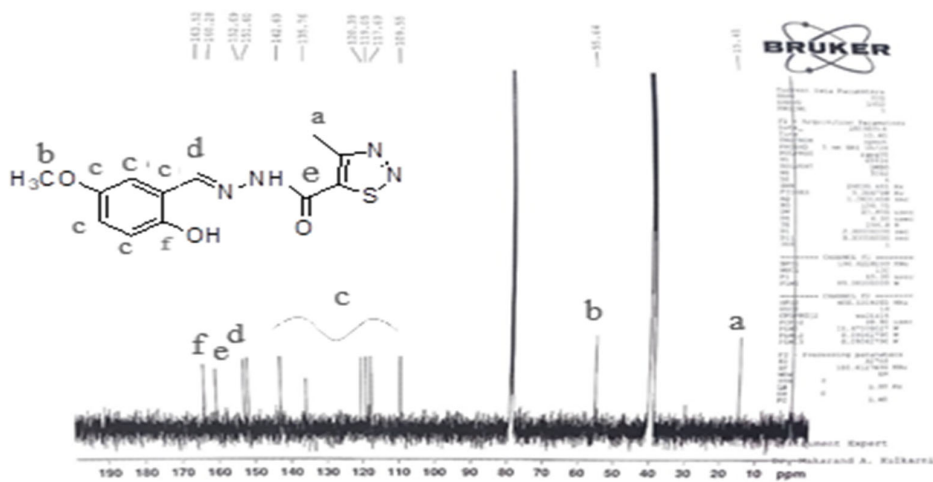


Fig.2. ¹³C NMR spectra of the ligand HL (3)

¹³C- NMR (400 MHz, DMSO, ppm) 163.52 (Phenolic carbon)^f, 160.28 (amide carbon)^e, 152.69 (azomethine carbon)^d, 151.60, 142.69, 135.76, 120.39, 119.05, 117.69, 109.55, (aromatic carbon)^c, 55.64 (methoxy carbon)^b 15.41(methyl carbon)^a

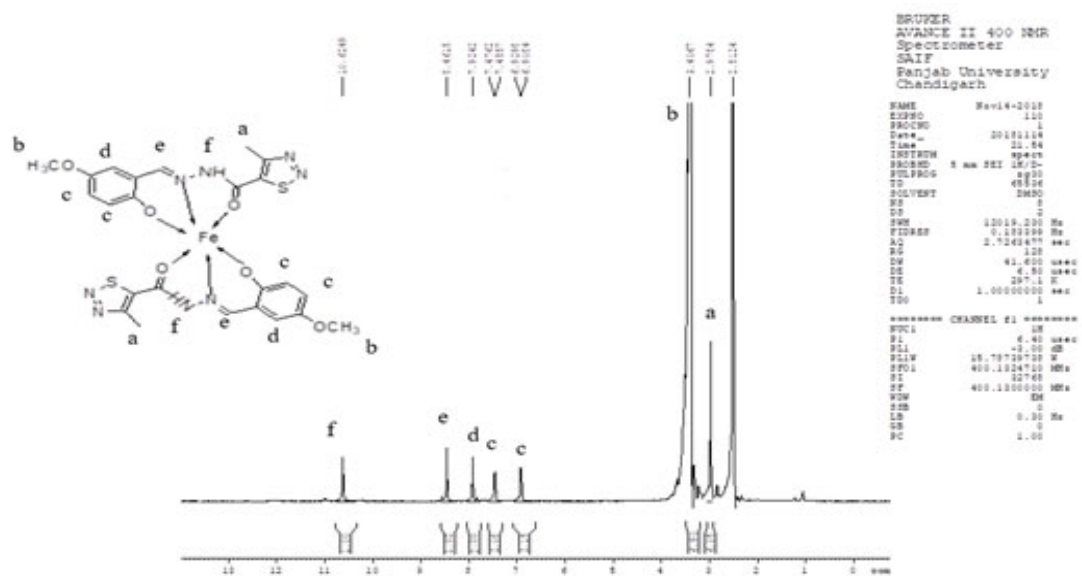


Fig.3. ^1H NMR spectra of the Fe(II) (**4a**), M : L ratio (1 : 2)

^1H NMR (400 MHz, DMSO-*d*₆) δ (ppm) 2.97 this is due to the proton of methyl group, (6H^a, s, (CH₃)₂), 3.40 attributed to methoxy proton (6H^b, s, (OCH₃)₂), 6.90-7.92 revealed presence of aromatic proton (6H^{c,d}, m, Ar-H), 8.46 attributed to azomethine proton (CH=N)^e, 10.62 this chemical shift indicates presence of amide proton (CONH)^f.

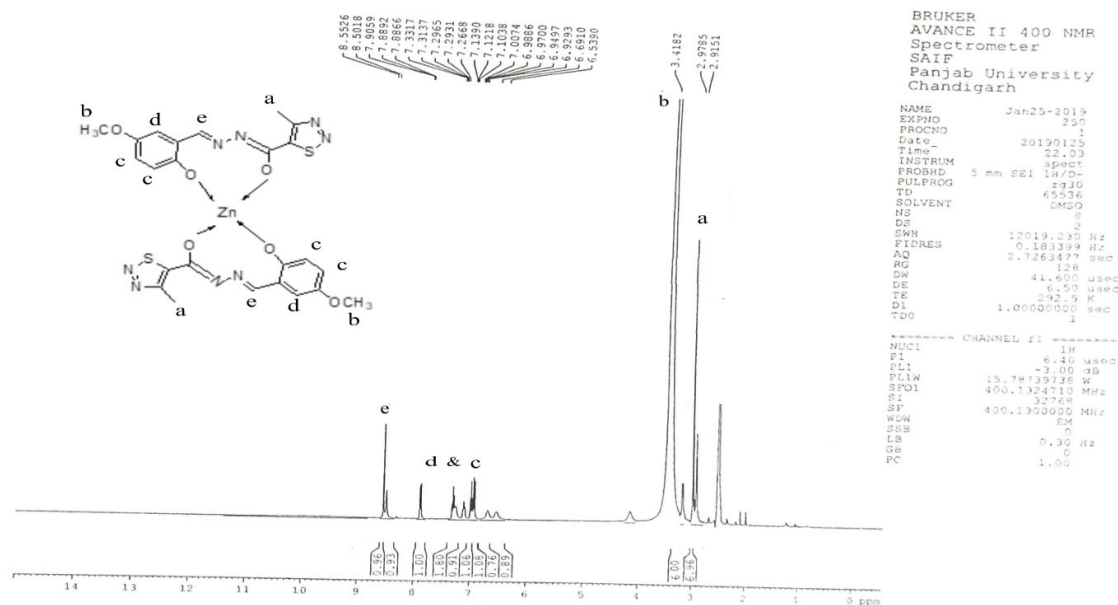


Fig.4. ^1H NMR spectra of the Zn(II) (**4b**), M : L ratio (1 : 2)

^1H NMR (400 MHz, DMSO-*d*₆) δ (ppm) = 2.97 this is due to the proton of methyl group, (6H^a, s, (CH₃)₂), 3.41 attributed to methoxy proton (6H^b, s, (OCH₃)₂), 6.53-7.90 revealed presence of aromatic proton (6H^{c,d}, m, Ar-H), 8.55 attributed to azomethine proton (CH=N)^e

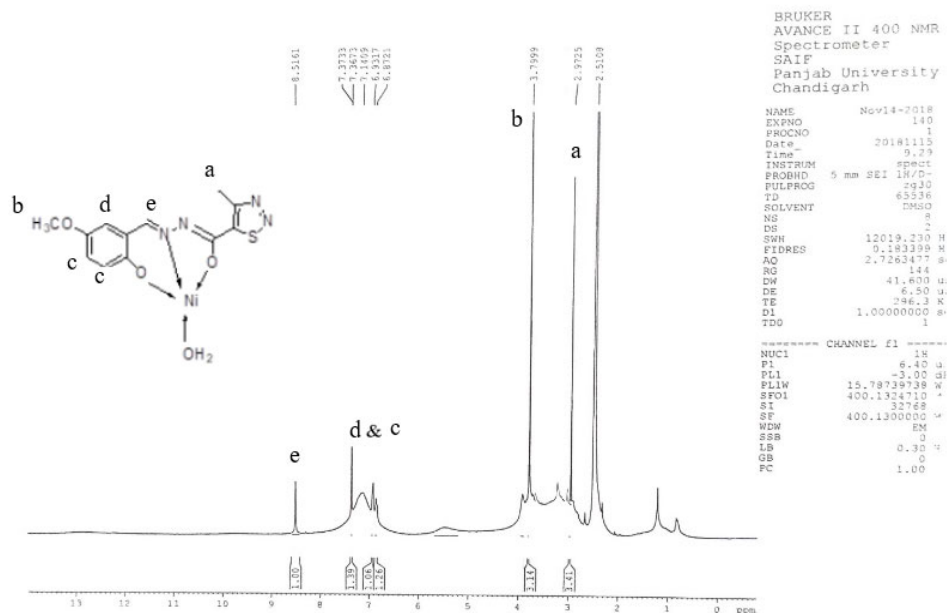


Fig.5. ^1H NMR spectra of the Ni(II) (**5b**), (M:L ratio 1:1)

^1H NMR (400 MHz, DMSO- d_6) δ (ppm) = 2.97 this is due to the proton of methyl group, (3H^a , s, CH_3), 3.41 attributed to methoxy proton (3H^b , s, OCH_3), 6.53-7.90 revealed presence of aromatic proton (3H^c , d, m, Ar-H), 8.55 attributed to azomethine proton ($\text{CH}=\text{N}$)^e.

Mass Spectra of ligand, HL (3)

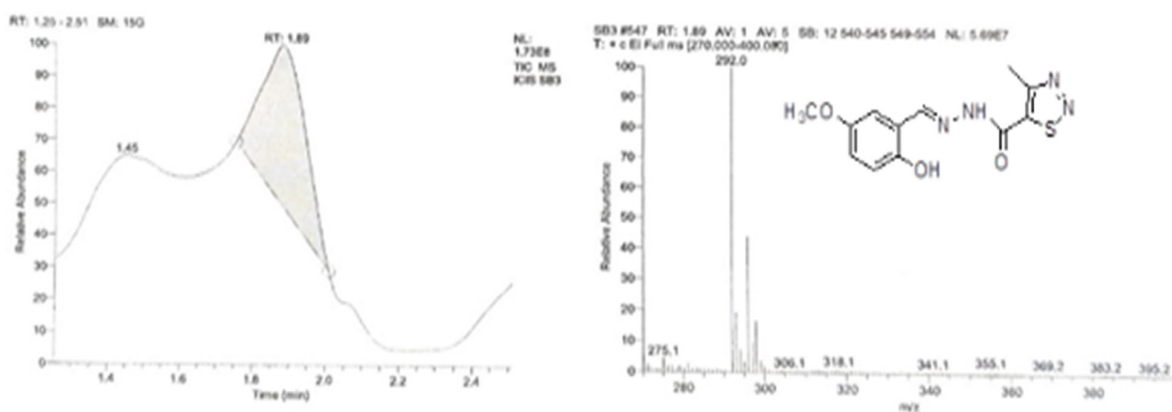


Fig.6. Mass spectra of ligand HL (**3**) MS (m/z): 292 (M^+)

IR Spectra of ligand and their metal complexes

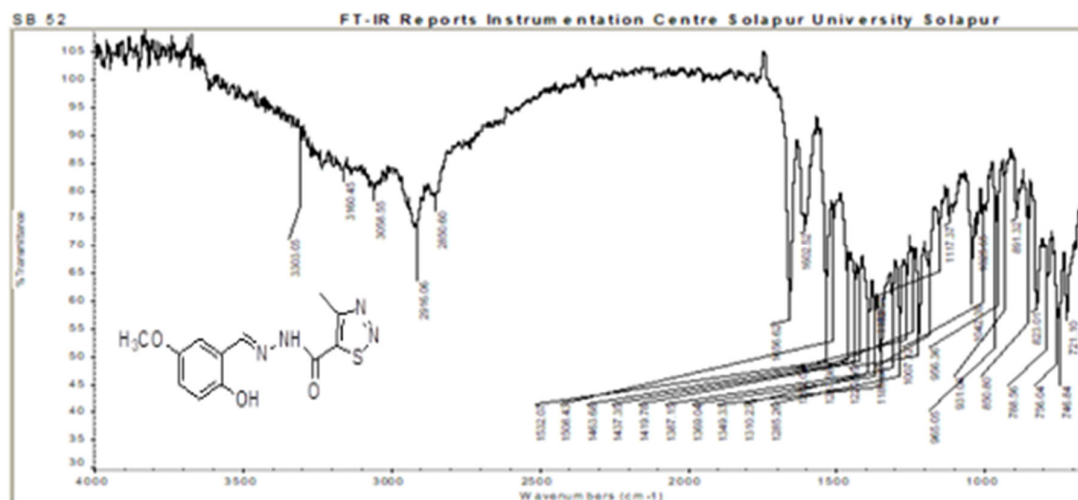


Fig.7. FT-IR spectra of the ligand, HL (3)

FT-IR (KBr, cm^{-1}): 3303 ν (Ph-OH), 3160 ν (N-H), 1656 ν (C=O), 1602 ν (C=N) 1285 ν (C-O).

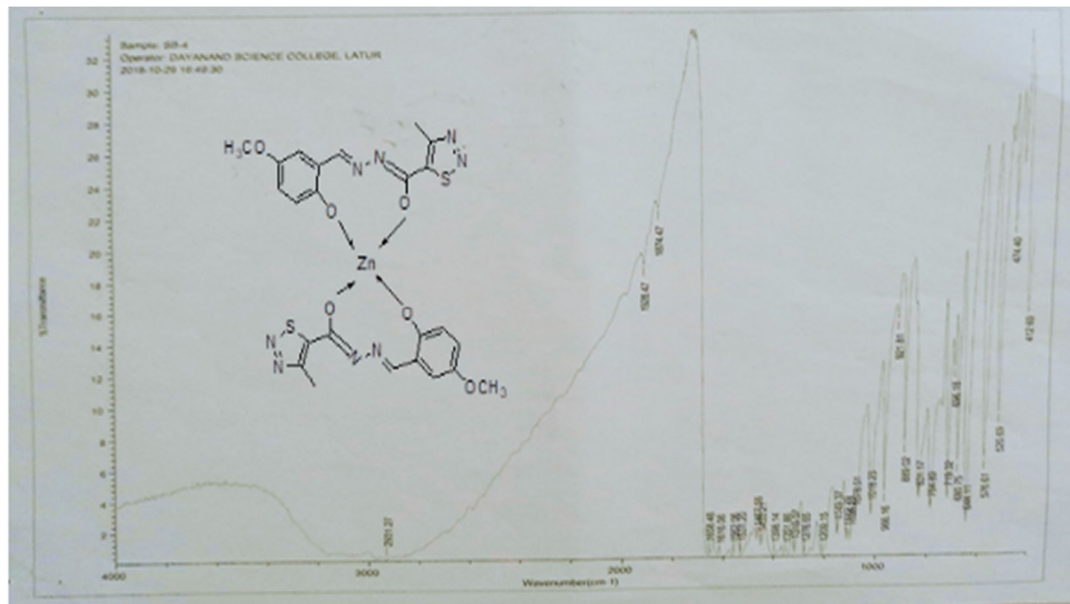


Fig.8. FT-IR spectra of the Zn(II),(4b)

FT-IR (KBr, cm^{-1}): 1562 attributed to ν (C=N), 1276 arising new band from phenolic ν (C-O), lower frequency new bands 520 and 474 attributed to ν (M-O) and ν (M-N).

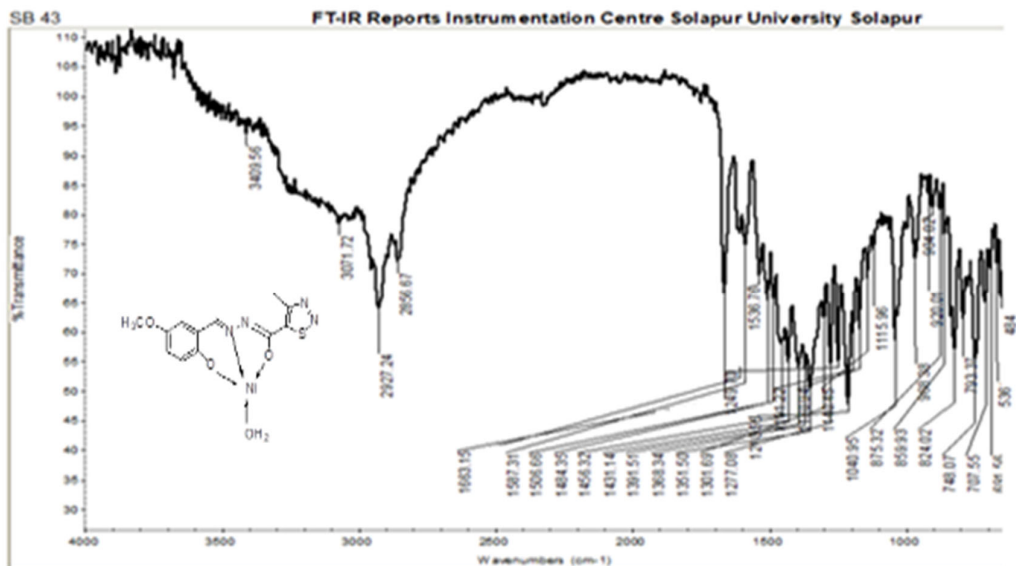


Fig.9. FT-IR spectra of the Ni(II),(5a)

FT-IR (KBr, cm^{-1}): 3409 vibrational frequency due to the coordinated water, 1536 attributed to $\nu(\text{C}=\text{N})$, 1277 arising new band from phenolic $\nu(\text{C}-\text{O})$, lower frequency new bands 536 and 484 attributed to $\nu(\text{M}-\text{O})$ and $\nu(\text{M}-\text{N})$.