

# Mössbauer Study of Iron Complexes of Medicinal Interest

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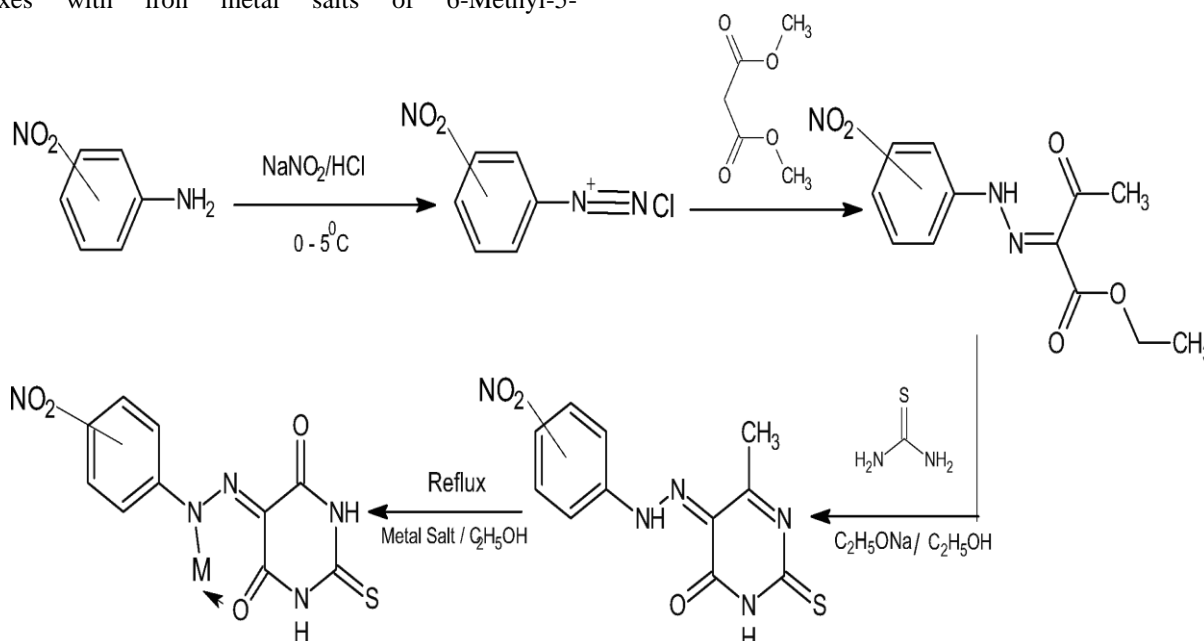
**Abstract-** The article deals with a study to synthesize transition metal complexes of iron with the ligand 6-methyl-5-arylhydrazono-2-thio-4-oxo-pyrimidine (MATOPyr). The synthesized metal complexes were characterized by X-ray diffraction (XRD), lattice parameters, particle size and hkl values of Fe complexes has been observed by the calculations of XRD data. Mössbauer measurements of O-Nitro-Fe and M-Nitro-Fe complexes has been taken to find out the oxidation state of Iron after complexation, it has been observed that it's been changed and the percentage of Fe of different oxidation state has been found by fitting the Mössbauer data.

**Key Words:** Iron Complex, X-Ray Diffraction, Mössbauer Spectroscopy.

**Introduction:** Pyrimidine and heterocyclic annulated pyrimidine systems undoubtedly belong to the most ubiquitous heterocycles in nature [1]. They have attracted considerable attention of both synthetic and medicinal chemists by virtue of their interesting biological activities [2] and immense synthetic potential for the constructions of many pharmacologically important novel heterocycles [3]. In particular, this type of pyrimidine derivative, an important class of nitrogen-containing fused heterocycles [4–5] has received extensive interest in the recent past owing to its wide spectrum of biological activity and clinical applications [6]. Pyrimidine derivatives constitute a very important class of compounds because they are components of nucleic acids and synthesized as a modulator of antitumor drug activity, antioxidants, antiplatelet, antiviral, antifungal, and hepatoprotective agents. In this article, we have synthesized complexes with iron metal salts of 6-Methyl-5-

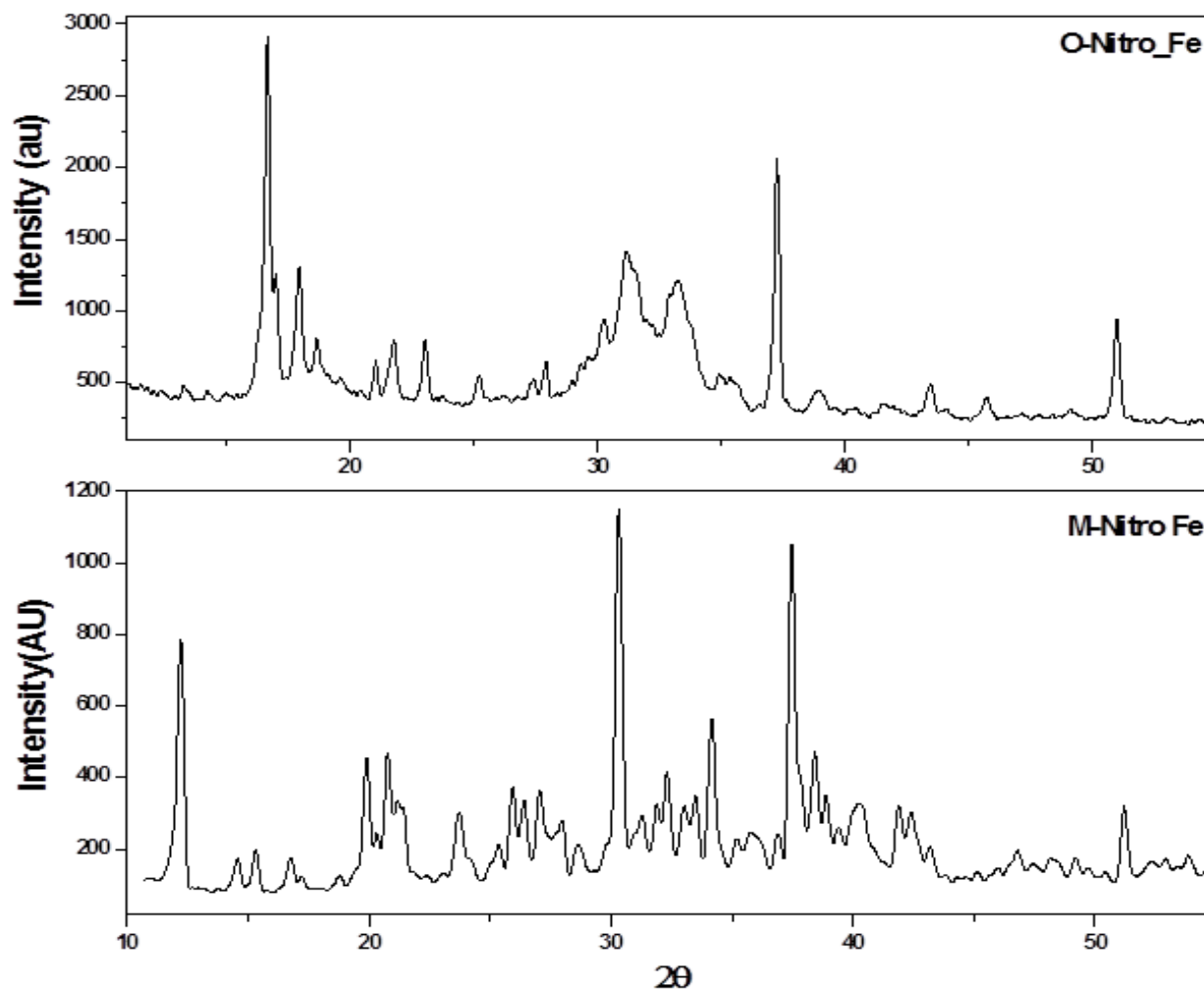
arylhydrazono-2-thio-4-oxo-pyrimidine Ligand. The prepared molecules possess hydrazono, carbonyl, and thionyl functional groups as potential binding sites for metal ions. This article reports the synthesis and structural elucidation of Fe (III) metal complexes with this ligand.

**Experimental:** The preparation of metal complexes has been performed via chemical route. The XRD pattern was obtained using a Philips PW3040/60 X-ray powder diffractometer with Ni-filtered Cu K $\alpha$  radiation ( $\lambda = 1.54\text{Å}$ ) having an operating voltage of 40 kV and current of 100 mA. The Mössbauer measurements were carried out using a standard PC-based spectrometer equipped with a Weissel velocity drive operating in the constant acceleration mode. The data was fitted with the NORMOS-SITE program, and the obtained parameters are with respect to natural iron.



**SCHEME 1** Schematic overview of synthetic strategy for ligand and metal complexes; M= Fe.

**X-ray Diffraction:** The X-ray powder diffraction patterns of the complexes are indicative of their crystalline nature. The diffraction patterns have been successfully indexed. The X-ray analysis reveals that all the samples are in single phase. The XRD peaks of the single phase of both samples were indexed and grain size were calculated and presented in TABLE1. The  $2\theta$  values obtained from the positions of Bragg peaks have been used to obtain interplaner spacing expressed in terms of (hkl).



**FIGURE1.** XRD plot of O-Nitro-Fe and M-Nitro-Fe

**TABLE 1.** Lattice parameter and hkl value of complexes.

M-Nitro-Fe			O-Nitro-Fe		
$2\theta$	a	hkl	$2\theta$	a	hkl
12.34	10.14	110	16.73	11.85	210
25.96	10.28	300	17.83	12.17	211
30.35	9.10	222	22.88	11.64	300
34.08	10.51	400	37.15	11.84	422
37.38	10.19	411	50.77	11.77	533

**Mössbauer Studies:** The Mössbauer spectrum of O-Nitro-Fe and M-Nitro-Fe is shown in FIGURE2. This spectrum was

least-square fit with a superposition of two doublets and one singlet, all with Lorentzian line shape. The fitted parameters are given in the TABLE2. According to the collected data presented in table, the percentage of Fe (II) is 88.9%. The isomer shift of  $1.217 \pm 0.005$ mm/s (35.6%),  $1.213 \pm 0.006$  (53.3%) corresponds to the expected value for octahedral Fe (II) complex, as previously described complex which exhibits distorted octahedral symmetry with an isomer shift of 1.212mm/s and quadrupole splitting of 3.01mm/s. In the present case Fe (II) exist in two chemical environments. Fe<sup>2+</sup><sup>(a)</sup> with an isomer shift of 1.217mm/s and Fe<sup>2+</sup><sup>(b)</sup> with an isomer shift of 1.213mm/s is predominant in the sample (35.526%, 53.271% respectively). The presence of Fe (III) (11.1%) is well characterise by the isomer shift of  $0.341 \pm 0.032$  mm/s.

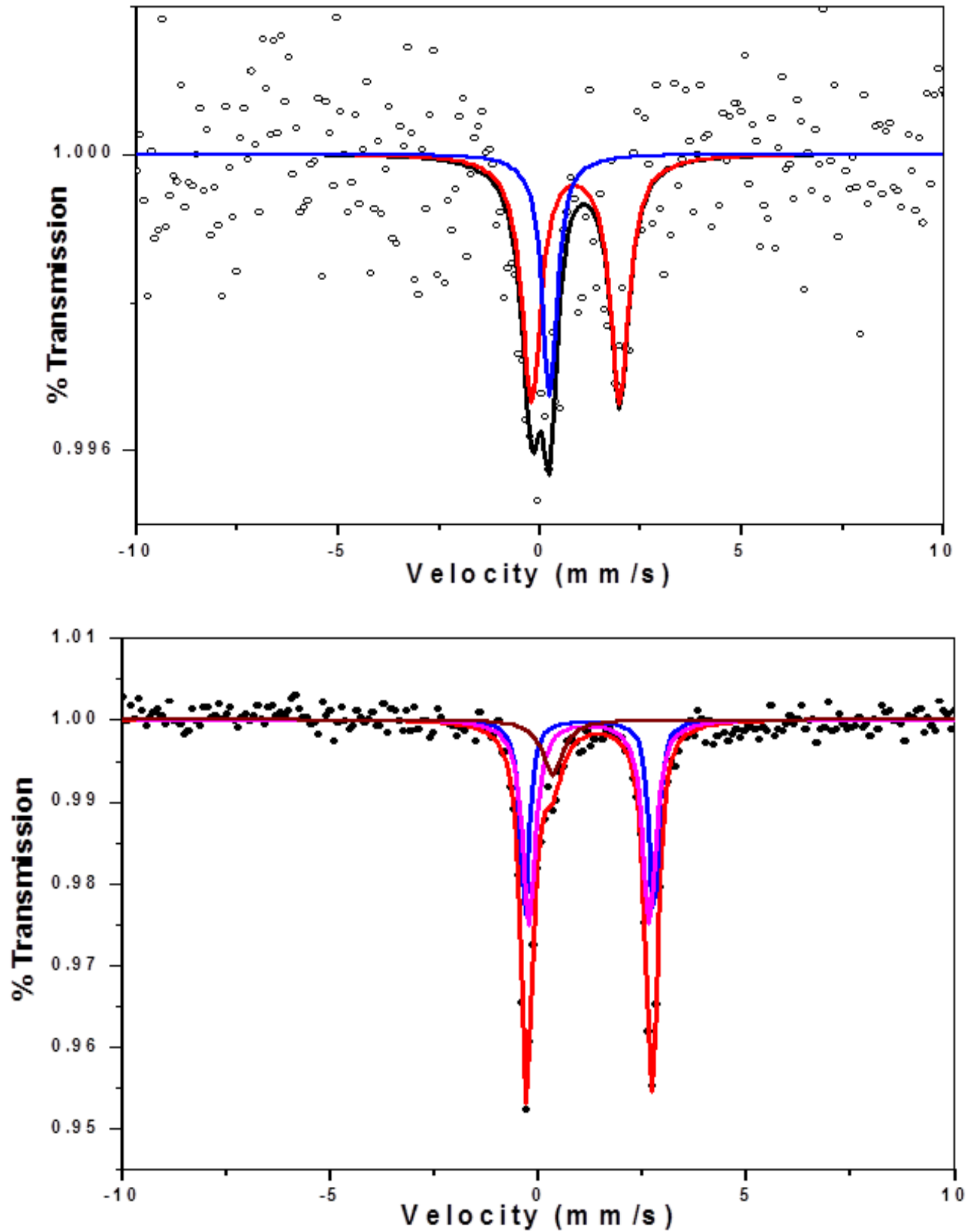


FIGURE2. Mössbauer spectra O-Nitro-Fe and M-Nitro-Fe.

**TABLE 2.** % Area of Fe<sup>2+</sup> and Fe<sup>3+</sup> present in Iron complexes

Sample	FWHM (mm/s)	Isomer shift (mm/s)	Q.S (mm/s)	% Area
[M-Nitro-Fe] Fe <sup>2+</sup>	0.568 ± 0.085	0.876 ± 0.040	2.18 ± 0.086	71.9
[M-Nitro-Fe] Fe <sup>3+</sup>	0.450	0.238 ± 0.047	2.18 ± 0.086	28.1
[O-Nitro-Fe] Fe <sup>2+(a)</sup>	0.254 ± 0.074	1.217 ± 0.005	3.09 ± 0.050	35.6
[O-Nitro-Fe] Fe <sup>2+(b)</sup>	0.373 ± 0.029	1.213 ± 0.006	2.89 ± 0.112	53.3
[O-Nitro-Fe] Fe <sup>3+</sup>	0.588 ± 0.089	0.341 ± 0.032	2.89 ± 0.112	11.1

## CONCLUSION

Complexation of Fe with 6-Methyl-5-arylhydrazono-2-thio-4-oxo-pyrimidine ligand has been successfully done via chemical route method. Samples are crystalline and in single phase. Lattice parameter of both sample obtained by XRD is 9.95 Å and 11.86 Å respectively for M-Nitro-Fe and O-Nitro-Fe. Particle size determined by XRD is 38.59 Å and 33.89 Å respectively for M-Nitro-Fe and O-Nitro-Fe. Mössbauer fitting reveals the value of isomer shift and quadrupole splitting of the complexes. It exhibits that the samples possess distorted octahedral symmetry.

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