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Article

Complexion of Fe^{3+} with Some Heterocyclic Ligands and Their Antibacterial Activities

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Abstract: Complexes can be used for different purposes based on their colors, solubility and chemical changes of the behavior of the central metal ions when they form ligands complex. Complex agents are often used to alleviate the water, because they like our tie Ca^{2+}, Mg^{2+}, Fe^{2+}, which causes the strength of the water. Complexes agents that bind the metal ions are used as drugs. Complexes can be quite effective in the treatment of agricultural lands, as they relate to metals mentioned, make the metal more assimilation of plants. In the galvanizing industry, it was discovered that galvanizing is higher quality and more aesthetic when used precious-metal complexes with which will be worn metal surfaces. In this paper, mixed ligands complex compounds which could provide valuable information in explaining the enzymatic processes in biological activity were synthesized and microbiological activities were studied.

Keywords: heterocyclic ligands, Antibacterial activity, Fe^{3+}

1. Introduction

Complex compounds have great importance in many branches of natural sciences such as chemistry, biology, pharmacology and pharmaceutical industry, technology, mineralogy, geology in the industry of colors (colorants etc.). Complexes can be used for different purposes based on their color, solubility and chemical changes of the behavior of central metal ions when they form ligands

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complex such as Chlorophyll, the green pigment of plants is a complex containing. More and more complex are being studied and applied in improving our life.

2. Materials and Methods

2.1. Synthesis

Synthesis of the complex products was made by means of refluxing reaction. Follow the course of the reaction, thin layer chromatography method and IR spectroscopy was used to identify the products.

For thin layer chromatography, stationary phase is silica gel, and mobile phase is a mixture of benzene: ethanol: acetone in volume ratio of 80: 10:10. It was observed that the substances traveled too much and cannot clearly separate each product with this mixture of mobile phase. Then a less polar mobile phase composed of dichlormetan and acetone in volume ratio 1:2 was used to better identify products by ligands. With this mobile phase clear substances trip can be identified on the thin layer plate. Visualization of the separation products was made in the bathroom of iodine.

- Determination of melting points was done with camera BUCHI Melting Point B-545 and Thiele-ut basin.
- Recording of IR spectra was made by Shimatzu resolution 8400S with 4 cm^{-1} at the Mathematics and Natural Sciences in the Department of Chemistry
- Microbiological activities of synthesized compounds were tested in microbiological lab at the Department of Biology.

In related compounds with coordinative (about semi polar) one or more electrons released by an atom (or a group of atoms), whereas other electrons orbital have become so common chemical bonds resulting from the combination of hetero polar connection (connection performed between ionized atoms with electric charges of opposite sign) and homo polar (the link that exists between atoms with equal polarity and in particular between the atoms of negative elements nonmetals have two chlorine atoms together and form a chlorine molecule Cl_2 creating a pair of electrons shared, by inserting one into the orbit of another one of the seven peripheral electrons.)

Reactions were developed by such mechanism; the couple of free electronic metal heteroatoms have attached the following scheme:

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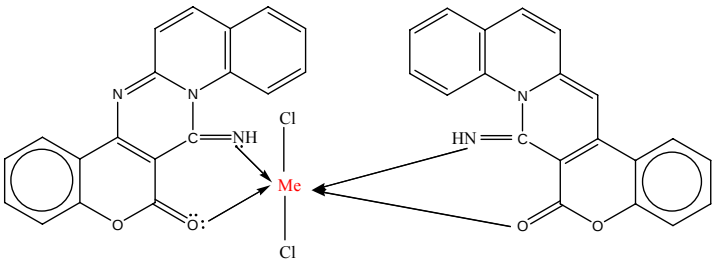
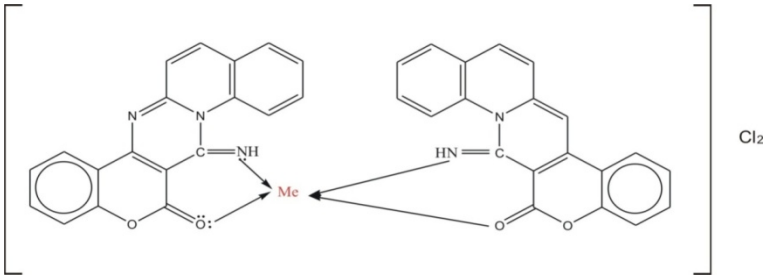


During complexion of metal will be developed general reaction:



2.2. Microbiological Activity Methods

Microbiological activity of new synthesized compounds has been tested in microbiological laboratory in the Department of Biology. The method used for determining the susceptibility of microorganisms to our compounds is obtained with disks diffusion method according to Kirby-Bayer's (51), the recommended method, because of its technical simplicity, especially this method is suitable for routine laboratories which applied to bacteria which grow fast and requires no special conditions for the media (culture plates) or the conditions of incubation. Microorganisms used for testing are (culture) of bacteria standard reference for quality control as list below:

(1). Escherichia coli, (2). Enterococus, (3). Citobacter and (4). Aeromonas.

Procedure to determine the microbiological activity is as below:

- The tablets of Hagar substances, the drives are brought to room temperature.
- Prepare inoculums bacterial cultivation of primary plaques affecting the Anza (Eze) 3-5 edges of colonies and transferred this material into test tube.
- On top of duct capillary sterile bacteria goes through the whole surface of the sheet feeder of Hagar.
- This procedure is repeated twice so that the plates rotate at an angle of approximately 600 each time in order to enable the absorption of moisture before applying discs with substance.
- With sterile tweezers placed impregnated drives which are substances in different concentrations on the surface of the inocular Hagar.

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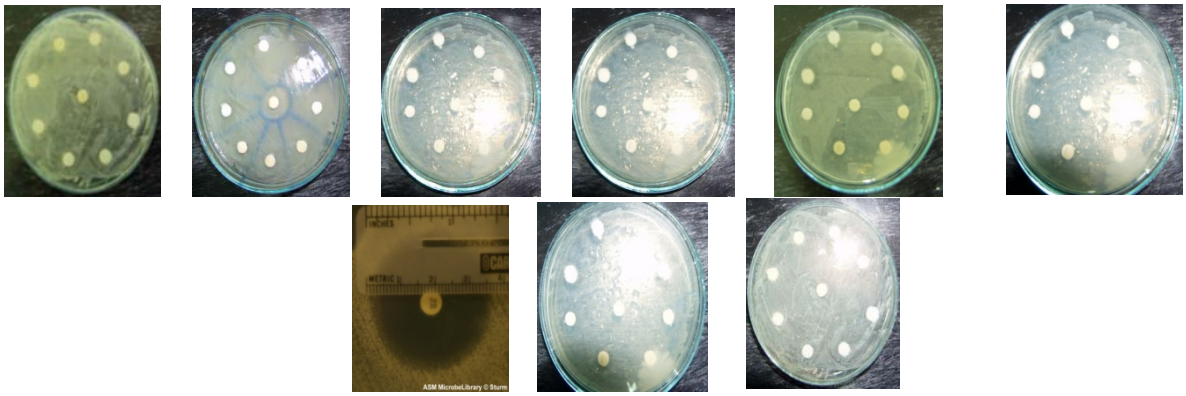
- 9 discs placed on agar in areas that have separated earlier.
- Easy disk was pressed with tongs through in order to ensure full contact with the agar disk.
- Tablets are labeled with derma graph (water-resistant pencil) with a specified number and type of bacteria, with concentration 3mg/ml, 5 mg/ml, dated and placed in the incubator 36.5^0C.
- Incubated for 48 hours.



After 48 hours incubation each plate was controlled in particular by using the compass or ruler (the spline has been set earlier) to measure the diameter and mark the area of activity, growth inhibition zone of bacterial inhibition zone of sterile or bacteria growth near the disk with the substance.

3. Results and Discussion

3.1. Synthesis Data

Heterocyclic complexes are synthesized by mixing and refluxing ethanol solution of ligands / salt $FeCl_3$ in ratio of 3:1.

Fe(2-amino-5-nitro-tiazol)$_3Cl_3$ was produced by the reaction of 2-amino-5-nitro-tiazole and $FeCl_3$, m = 1. 25g, m.p. 155-159 ^{0}C and yield was 60.29%. Identification of the product produced was made by means of infrared spectroscopy.

The reaction of 4-benzaldehidë dimethilamino and $FeCl_3$ produced Fe(4dimetilamino-benzaldehid)$_3Cl_3$ with m = 1.84g, m.p 40-45 ^{0}C, yield of 90.19%. Identification of the product was made by infrared spectroscopy. Peak in 2900 cm^{-1} belongs to the vibrations of links υ (NH) and aromatic ring stretching vibrations, whereas the signal at 2700 cm^{-1} are str.υ vibrations (CH) of aliphatic stretching.

The product of 2-amino-piridinë and $FeCl_3$ was Fe(2-amino-piridina)$_3Cl_3$, with m = 2.20g, m.p. 40-45 ^{0}C and yield 92.82%. In IR spectra, signals at 3350 cm^{-1} belongs to the vibrations of links υ (NH) ring aromatic, while the signal at 3290 cm^{-1} are str.υ vibrations (CH) of stretching fragrant, shown at length vibrations 1620 cm^{-1} band belongs NH 1570 cm^{-1} for connecting the signal is absorbed

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υ (C = C) stretching fragrant. Absorbs signal at 1650cm^{-1} was generated by the double bond C = N stretching fragrant, the 1280 cm^{-1} frequencies responses signal is formed by CN bond, 1450 cm^{-1} is the connection signal δ (CC) of aryl, whereas frequencies 720 cm^{-1} is the bending of CH of aromatic ring.

The product of winning Pyrazol and $FeCl_3$ was Fe(Pyrazol)$_3$Cl$_3$ with m = 2. 56g, m.p. 155-159 ^{0}C and yield of 94.46%.

The product of 2-amino-4-chlorobenzothiazole and $FeCl_3$ was Fe(2-amino-4-chlorobenzotiazole)$_3$Cl$_3$ with m = 1. 42g, m.p. 110-150 ^{0}C and yield 72.82%.

3.2. Interpretation of Results

The complexes which completely destroy the bacteria (bactericidal effect) or inhibit their growth (bacterium static effect) is required not to harm patients who use these substances for the treatment or prevention of diseases. Inhibitory effect of the compounds analyzed in inhibition the development of microorganisms tested was usually distinguished by increasing the concentration of compounds that is added to the zone of inhibition, but in some cases the opposite happened. Two compounds, Fe(Pyrazol)$_3$Cl$_3$ and Fe(2amino-4-chlorobenzothiazole)$_3$Cl$_3$ were analyzed in this study.

Based the data in table 1, the antibacterial activities of Fe(Pyrazol)$_3$Cl$_3$ are:

- To Escher Coli, 9 mm zone of inhibition zone was obtained with concentration 1 mg / ml, same value was obtained with concentration 5 mg / ml, while at concentration 3 mg / ml no activity has been shown.
- In term of antifungal activity to Enterococus, three tested concentrations 1 mg / ml, 3 mg / ml and 5 mg / ml, all of them displayed an 8 mm of inhibition area.
- Fe(Pyrazol)$_3$Cl$_3$ showed high antibacterial activity to Citobacter with inhibition area of 10 mm for 3 mg / ml of concentration, 7 mm for concentration of 5 mg / ml, however at concentration of 1 mg / ml no any activity has been observed.

For Fe(2-amino-4-chlorobenzotiazolë)$_3$Cl$_3$, the antibacterial activities are:

- High average activity against Escher coli was shown. When concentration is 3 mg / ml, a 13 mm width of inhibition area was observed and 8 mm of that with concentration at 5 mg / ml. But with concentration of 1 mg / ml, no activity has been shown.
- To Enterococus, antibacterial activity was shown only at high concentration; at concentration 1 mg / ml and 3mg/ml, no activity has been shown. When concentration reached to 5 mg / ml, an inhibition area with 8 mm width was observed.
- Fe(2-amino-4-chlorobenzotiazole)$_3$Cl$_3$ shown no antibacterial activity to Citobacter at concentrations of 1 mg / ml, but an inhibition zone of 8 mm width was reached when concentration are 3 mg / ml and 5 mg / ml.

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- To Aeromonas, when concentration of Fe (2-amino-4-chlorobenzotiazole)$_3$Cl$_3$ is 5mg/ml, an 8mm of width of inhibition area was observed, no activity was shown with other studied concentrations.

Table 1**.** Diameter of Zones Inhibition (mm) of Disks Impinged to Escheria Coli, Enterococus, Citobacter and Aeromonas with Different Concentrations of Synthesized Compounds

Compound	**Concentration**	**Escheria Coli**	**Enterococus**	**Citobacter**	**Aeromonas**
Fe (pyrazole)$_3$Cl$_3$	1 mg/ml	9	8	0	11
Fe (pyrazole)$_3$Cl$_3$	3 mg/ml	0	8	10	8
Fe (pyrazole)$_3$Cl$_3$	5 mg/ml	9	8	7	0
Fe(2amino-4-chlorobenzothiazole)$_3$Cl$_3$	1 mg/ml	0	0	0	0
Fe (2amino-4-chlorobenzothiazole)$_3$Cl$_3$	3 mg/ml	13	0	8	0
Fe (2amino-4-chlorobenzothiazole)$_3$Cl$_3$	5 mg/ml	8	8	8	8

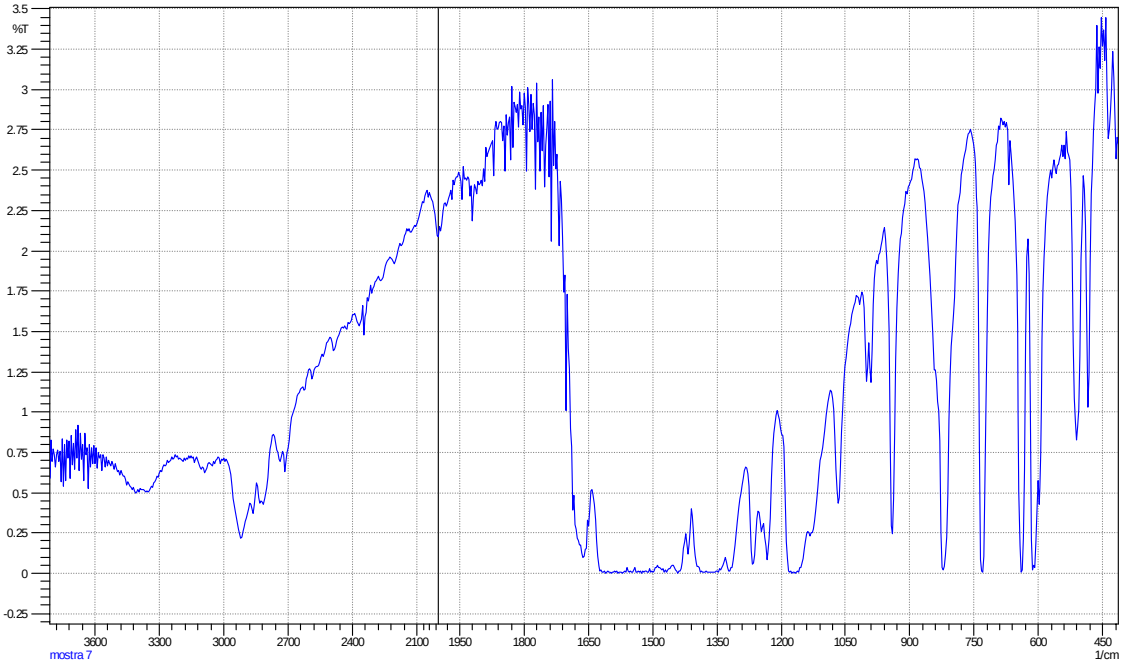

Fig. 1: IR of Fe (4-dimetilamino-benzaldehid)$_3$Cl$_3$

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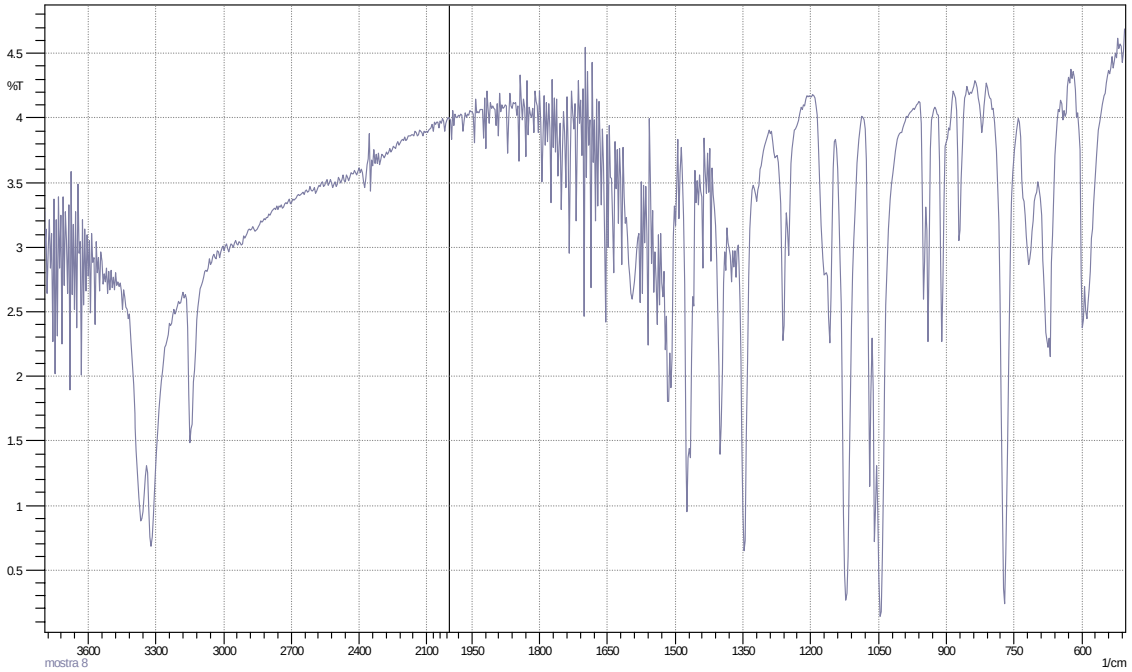

Fig. 2: IR of Fe(2-amino-piridin)$_3$Cl$_3$

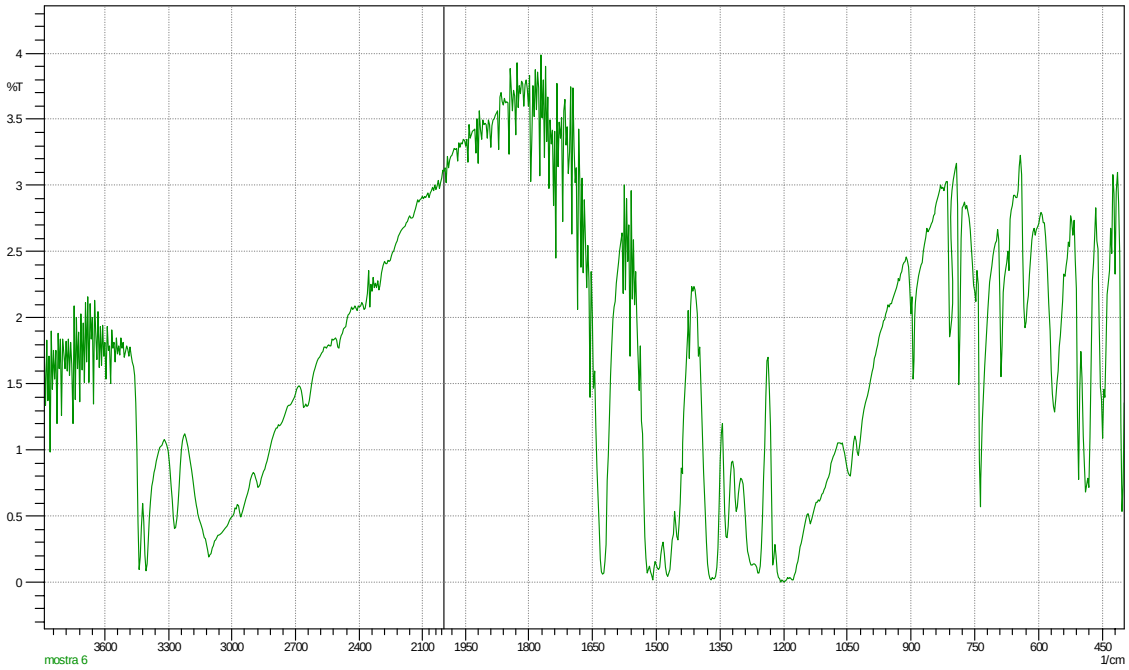

Fig. 3: IR of Fe(2-amino-5-nitro-tiazol)$_3$Cl$_3$

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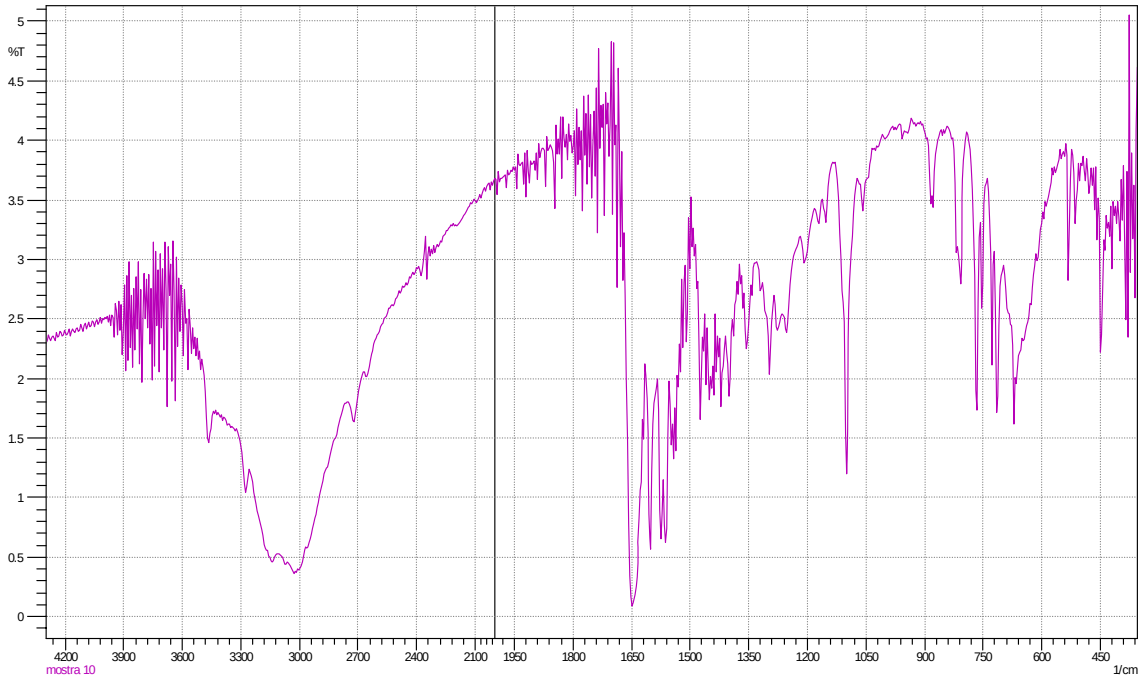

Fig.5: IR of Fe(2-amino-4-chlorobenzotiazol)$_3$Cl$_3$

4. Conclusion

Heterocyclic complexes are synthesized by mixing ethanol solution of heterocyclic ligands / salt $FeCl_3$ in ratio of 3:1.

By reaction of 2-Amino-5-nitro-tiazole and $FeCl_3$, Fe(2-Amino-5-nitro-tiazol)$_3$Cl$_3$ was obtained with m = 1. 25g, m.p. 155-159 ^{0}C and yield of 60.29%.

By reaction of 4-benzaldehide dimethilamino and $FeCl_3$, Fe(4dimetilamino-benzaldehide)$_3$Cl$_3$ was obtained with m = 1. 84g, m.p. 40-45 ^{0}C and yield of 90.19%.

By reaction of 2-Amino-piridine and $FeCl_3$, Fe(2-Amino-piridine)$_3$Cl$_3$ was obtained with m = 2.20g, m.p. 40-45 ^{0}C and yield of 92.82%.

By reaction of winning Pyrazol and $FeCl_3$, Fe(Pyrazol)$_3$Cl$_3$ was obtained with m = 2. 56g, m.p. 155-159 ^{0}C and yield of 94.46%. Fe(Pyrazol)$_3$Cl$_3$ showed high activity against bacteria Citobacter, Escher Coli and Enterococus.

By reaction of 2-amino-4-clorobenzothiazole and $FeCl_3$, Fe(2-amino-4-chlorobenzotiazole)$_3$Cl$_3$ with m=1.42g, m. p. 110-150 ^{0}C and yield of 72.82%. This compound showed reactivity to Escheria coli, but no reactivity to Aeromonas, Enterococus and Citobacter.

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