



# Study of Photochemical Reaction involving Nitrosyl Penta cyanoferrate (II): Thiourea and Dimethyl Thiourea Mix Ligand System

Bhoomika R. Gajjar<sup>1\*</sup>, Mayank R. Mehta<sup>2</sup> and N. R. Goyal<sup>3</sup>

<sup>1</sup>Ananya Institute of Science, Kalol-382721 (Gujarat), INDIA

<sup>2</sup>Shri P.H.G. Muni. Arts and Science College, Kalol-382721 (Gujarat), INDIA

<sup>3</sup>M.G. Science Institute, Ahmedabad-380009 (Gujarat), INDIA

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\*Corresponding Author Email: [bhoomigajjar3@gmail.com](mailto:bhoomigajjar3@gmail.com)

## Abstract

Sodium nitroprusside photochemical procedures are extremely important in photochemistry. Sodium nitroprusside's photochemical reactions have some distinctive alternative methods of replacing cyanide ligand with some mixed ligands in  $\text{Na}_2[\text{Fe}(\text{CN})_5\text{NO}]\cdot 2\text{H}_2\text{O}$ . Ligand exchange responses of  $\text{Na}_2[\text{Fe}(\text{CN})_5\text{NO}]\cdot 2\text{H}_2\text{O}$  were noted using spectral and analytical methods and elemental analysis distinguished the products. The activation parameters are recorded, and the structures of the compounds being investigated are also discussed. Tentative mechanisms have been suggested for these photochemical reactions. The antimicrobial activity for these compounds against *E-coli*, *Bacillus cereus*, *Yeast* and *Aspergillus niger* and MIC for newer complexes was also determined.

## Keywords

photochemical reactions, sodium nitroprusside, ligands, antimicrobial activity.

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## INTRODUCTION

There are few documented photoactive metal nitrosyl complexes one of them is Sodium nitroprusside. Metal complexes can carry as ligand and release them under controlled conditions<sup>1</sup>. Nonfiction survey suggests that extensive photochemical work on sodium nitroprusside will be continued because of its received negligible attention<sup>2</sup>. Loris Grossi and Sadra D'Angelo have been studied of the mechanism of interaction between sodium nitroprusside and cysteine<sup>3</sup>. V.P. Prajapati, K. N. Salvi et.al investigated the ligand exchange reactions of sodium nitroprusside with

tellurate ions and it's behaves as a unidentate ligand<sup>4</sup>.

The photochemical ligand exchange reactions of sodium nitroprusside were studied by Prajapati et al<sup>5&6</sup>. M.R. Mehta and V.P. Prajapati investigated the ligand exchange reactions are well known in the field of coordination chemistry, but these reactions ordinarily involve the substitution of a weaker ligand by a stronger ligand and their experimental work were not only provides a pathway for this unfavourable reaction, but also it will open further avenues for such photochemical ligand exchange reactions<sup>6</sup>. The effects of different parameters like

pH, intensity of light concentration of sodium nitroprusside and ligand concentration on the yield of this substitution product were studied by M.R. Mehta et.al.<sup>5&10</sup>. Determination of persulphate using photochemical exchange reaction of sodium nitroprusside has been investigated by Ajay Sharma et.al.<sup>7</sup>. A newer, faster, inexpensive and convenient quantitative method for the determination of hydroxylamine hydrochloride using photochemical ligand exchange reaction of sodium nitroprusside has been investigated by Hardik Bhatt et.al.<sup>8</sup> and they observed sodium nitroprusside is a photolabile complex and it undergoes photochemical ligand exchange reactions rapidly. Photochemistry plays a pivotal role in a number of chemical and biological processes<sup>9</sup>.

The photochemical reaction of sodium nitroprusside may be useful to have an insight in the understanding of its photochemical behaviour on one hand and it will also provide some alternate routes to prepare some newer complexes on the other<sup>10</sup>. Sodium nitroprusside contains cyanide ligands that cannot be so easily replaced chemically. An effort has been made to substitute this ligand by another ligand like dimethyl urea. The progress of this photochemical reaction has been observed spectrophotometrically and the product was isolated and characterized by M.R. Mehta et.al.<sup>11</sup>. The electronic structure of sodium nitroprusside was investigated by using soft X-ray absorption (XA) spectroscopy<sup>12</sup>.

A synthesis and reactivity of Nitrosyl complexes of metals were studied by Ashley M. Wright and Trevor W. Hayton<sup>13</sup>. M. R. Mehta, Vipul Prajapati et.al investigated the photochemical reaction of sodium nitroprusside in the presence of dimethylurea gives bidentate ligand<sup>14</sup>. Peter C. Ford suggests that the photochemistry and photophysics of transition metal compounds has blossomed from relatively a major research theme<sup>15</sup>.

The present work describes the photochemical reaction of nitrosyl pentacyanoferrat (II), in the presence of thiourea and Dimethylthiourea.

#### Antimicrobial Activity

The development of new antimicrobial and anticancer therapeutic agents is one of the fundamental goals in medicinal chemistry.<sup>16</sup> Antimicrobial activity of thiourea derivative ligands and their metal complexes against bacteria and yeast were reported by Hakan Arslan, Nizami Duran et al.<sup>17</sup>. Jaggilal, S. K. Gupta et al. synthesized 3, 4-Dihydrpyrimidinones and evaluated their synergistic antimicrobial activity against bacteria and fungi.<sup>18</sup> Joanna Stefanska, Daniel Szulczyk et al.; synthesized series of new thiourea derivatives and obtained

compound were tested for their antibacterial and antifungal activities. From obtained eighteen compounds, five of them showed significant inhibition against Gram positive cocci.<sup>19</sup> The antibacterial and antifungal activities of all synthesized fluorinated 1-aryol-3-aryl thioureas has been explored against various bacterial and fungal strains were reported by Aamer Saeed, Uzma Shaheen and et al.<sup>20</sup>

Rajesh H. Tale, Atish H. Rodge and et al. were investigated novel 3, 4-dihydropyrimidin-2(1H)-one urea derivatives. Also, the synthesized compounds were screened against antimicrobial and anti-inflammatory activities. Some of them showed promising antimicrobial activity.<sup>21</sup> G.P. Suresha, R. Suhas, and et al. revealed that the compounds containing urea and thiourea functionalities along with fluoro group have exerted a highly potent activity. Also, they conclude that the antibacterial activity of the synthesized compounds showed that the urea and thiourea moieties play a major role in enhancing the activity.<sup>22</sup>

Our study aims at synthesizing a new compound and its exploring antimicrobial activity.

#### EXPERIMENTAL

In this research, 0.060gm of sodium nitroprusside (SNP) and 0.048gm thiourea and 0.038gm dimethylthiourea have been dissolved in 100ml of distilled water and blended in 250ml beaker. Sunlight was then subjected to this alternative (Light Intensity= 16.0 mWcm<sup>-2</sup>). A solarimeter was used to measure the light intensity. The solution's required pH was to adjust by adding earlier standardized sulphuric acid and solution solutions for hydroxide. The Photochemical Reaction progress was permitted to complete and then filtered the reaction combination. The filtrate was evaporated in a water bath and adequate washing and drying was used to obtain the residue. A digital balance was used to measure the yield.

#### Experimental work for Antimicrobial Activity

The synthesized compound was screened for their antibacterial and antifungal activity against *Escherichia coli*, *Bacillus cereus*, *Aspergillus niger* and *Saccharomyces cerevisiae*. The compound was dissolved in water at an initial concentration 10mg/ml and then was serially diluted in culture medium. Antimicrobial activity was checked by Kirby Baurer's method using Muller-Hinton agar for both bacteria and fungi. Fluconazole and Streptomycin were used as reference antifungal agent and antibacterial agent respectively. The inhibition zones

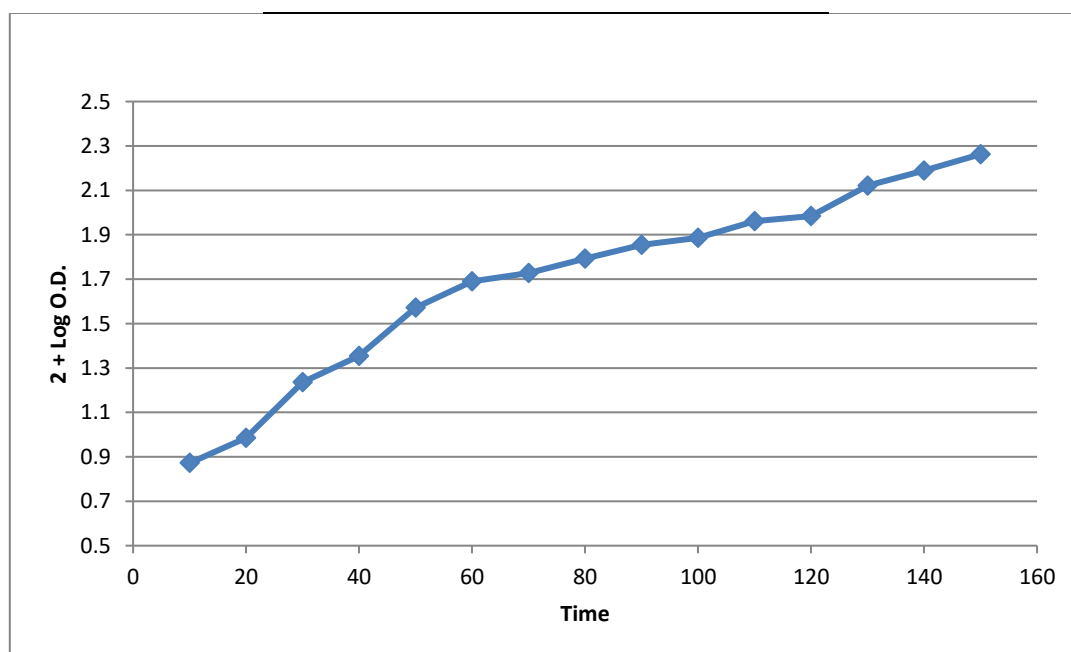
were measured in cm at the end of incubation period of hours at C for bacteria and hours at 2 C for fungi. MIC for compound was determined for *E-coli*. The minimum inhibitory concentrations of the chemical compound were recorded as the lowest concentration of chemical compound in the tubes with no growth (i.e., no turbidity) of inoculated bacteria. At the end of the incubation period, MIC values were recorded as the lowest concentrations of the substances that gave no visible turbidity.

## RESULTS AND DISCUSSION

A 5.0 ml aliquot was removed from the reaction mixture and with time of exposure the change in absorbance was observed calorimetrically at  $\lambda_{\text{max}} = 620 \text{ nm}$ . If a log (absorbance) v/s time plot was observed to be linear and pseudo-first order kinetics followed. The rate constant of the reaction has been calculated by the expression  $K = 2.303 \times \text{slope}$ . The results are summarised in Table A-1 and graphically shown in Figure A-1.

**Table-A-1: Typical Run**

Time	2 + Log O.D.	O.D.
10	0.8721	0.0745
20	0.9858	0.0968
30	1.2367	0.1725
40	1.3543	0.2261
50	1.5721	0.3734
60	1.6899	0.4870
70	1.7279	0.5345
80	1.7931	0.6211
90	1.8545	0.7154
100	1.8864	0.7710
110	1.9617	0.9156
120	1.9849	0.9660
130	2.121	1.3214
140	2.1884	1.5432
150	2.2626	1.8310



**Figure-A-1: Typical Run**

### Effect of pH

The pH values may affect the photochemical reaction of sodium nitroprusside in the presence of thiourea and dimethylthiourea ligand and the effect of pH on this reaction has been studied. Table A-2 shows the results.

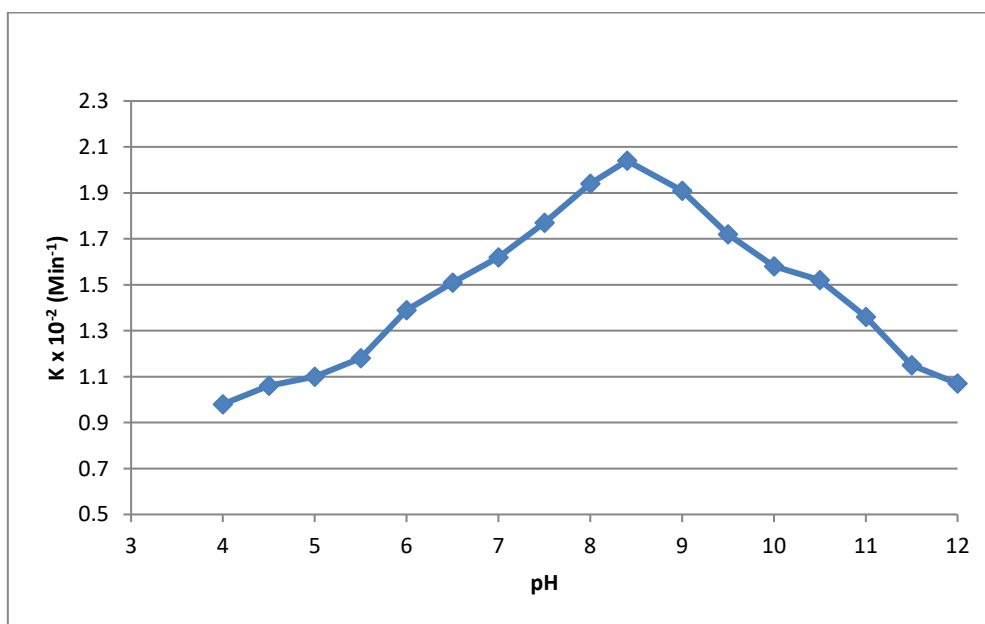
It has been observed that this ligands exchange reaction rate increases as the reaction medium increases its pH. At pH=8.4, it reaches a maximum.

Increasing pH further, a decrease in the product was observed. It can be explained on the basis that, as the pH has been increased, the availability of the ligand ions increases in their ionic form of thiourea and dimethylthiourea, thus increasing the reaction rate. But there is a possibility of a competitive reaction of ligand ions, thiourea and dimethylthiourea to that of OH<sup>-</sup> ions by increasing the pH above 8.4. Therefore, there was a corresponding decrease in yield.

**Table-A-2: Effect of pH**

pH	K x 10 <sup>-2</sup> (Min <sup>-1</sup> )
4	0.98
4.5	1.06
5	1.1
5.5	1.18
6	1.39
6.5	1.51
7	1.62
7.5	1.77
8	1.94
<b>8.4</b>	<b>2.04</b>
9	1.91
9.5	1.72
10	1.58
10.5	1.52
11	1.36
11.5	1.15
12	1.07

[SNP] = 2.0137 x 10<sup>-3</sup> M; [Dimethylthiourea] = 3.64 x 10<sup>-3</sup> M  
[Thiourea] = 6.305 x 10<sup>-3</sup> M; λ max = 620 nm Light Intensity = 16.0 mWcm<sup>-2</sup>



**Figure-A-2: Effect of pH**

### Effect of sodium nitroprusside concentration

Effect of sodium nitroprusside on the photochemical reaction was observed and the results are reported in Table-A-3.

It was observed that the rate of sodium nitroprusside photochemical reaction with thiourea and dimethylthiourea ions increases in sodium nitroprusside concentration. This may be due to the fact that as the sodium nitroprusside concentration has been increased; the number of excited species is also increasing, resulting in a corresponding increase

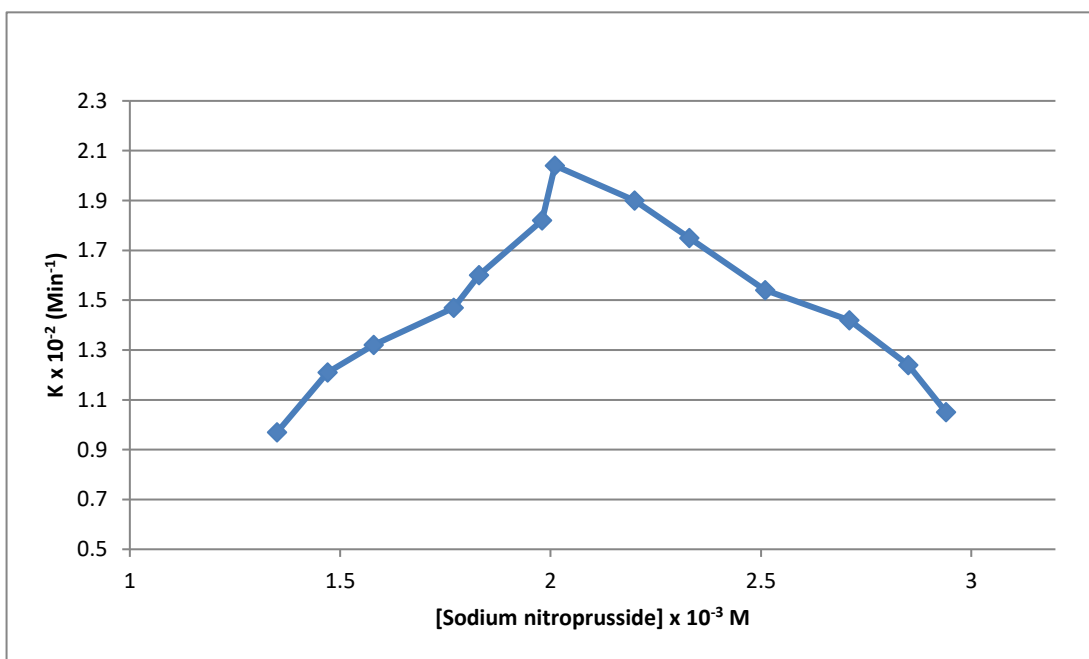
in the reaction rate. After a certain limit, if the sodium nitroprusside concentration was further increased, the reaction rate decreased.

This decrease can be explained on the basis that the substrate is dark red colored, which will absorb a significant portion of incident radiation traveling through the mixture of reactions. In other words, the nitroprusside solution will begin to act as a filter and as a result, the reaction rate has been found to decrease.

**Table-A-3: Effect of SNP**

[Sodium nitroprusside] x 10 <sup>-3</sup> M	K x 10 <sup>-2</sup> (Min <sup>-1</sup> )
1.35	0.97
1.47	1.21
1.58	1.32
1.77	1.47
1.83	1.60
1.98	1.82
<b>2.01</b>	<b>2.04</b>
2.20	1.90
2.33	1.75
2.51	1.54
2.71	1.42
2.85	1.24
2.94	1.05

[Thiourea] = 6.305 x 10<sup>-3</sup> M; [Dimethylthiourea] = 3.64 x 10<sup>-3</sup> M  
Light Intensity = 16.0 mWcm<sup>-2</sup>; λ<sub>max</sub> = 620 nm pH = 8.4



**Figure-A-3: Effect of SNP**

### Effect of Thiourea concentration

The effect of thiourea concentration on the rate of sodium nitroprusside's photochemical reaction was also observed by taking various thiourea concentrations. The results are reported in Table-A-4.

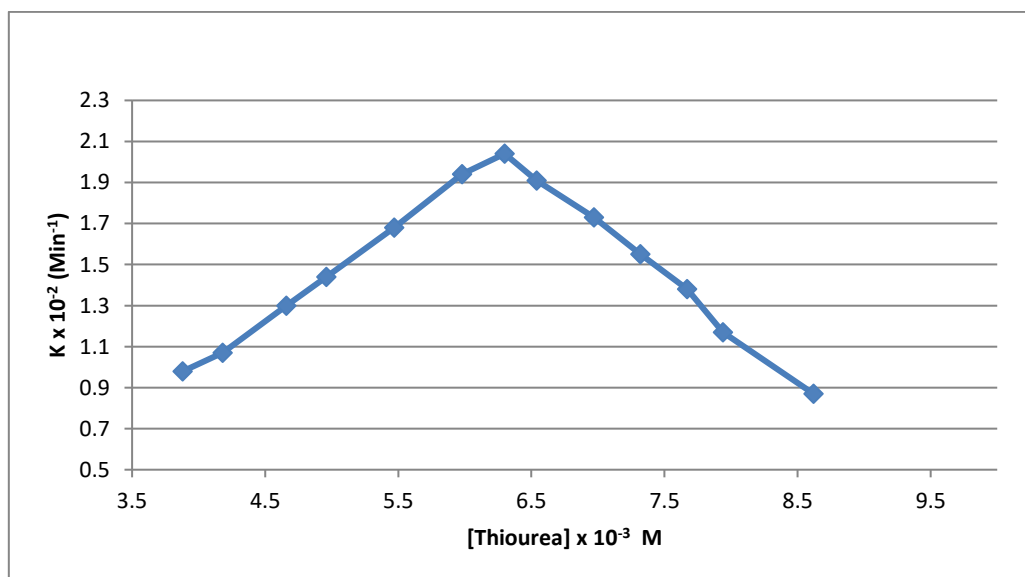
It was observed that there was a corresponding increase in the reaction rate as the concentration of thiourea increased, reaching a maximum of  $[\text{thiourea}] = 6.30 \times 10^{-3} \text{ M}$ . It can be explained on the

basis that, in the concentration of participating species, the reaction rate increases. On the other hand, on increasing the thiourea concentration above  $6.30 \times 10^{-3} \text{ M}$ , a decrease in the reaction rate was observed. It can be attributed to the fact that higher thiourea concentration may hinder its own movement within a desired time limit to reach the excited species of sodium nitroprusside. Thus, for higher concentrations of thiourea ions, a decrease in product yield was observed.

**Table-A-4: Effect of Thiourea**

<b>[Thiourea] x 10<sup>-3</sup> M</b>	<b>K x 10<sup>-2</sup> (Min<sup>-1</sup>)</b>
3.88	0.98
4.18	1.07
4.66	1.30
4.96	1.44
5.47	1.68
5.98	1.94
<b>6.30</b>	<b>2.04</b>
6.54	1.91
6.97	1.73
7.32	1.55
7.67	1.38
7.94	1.17
8.62	0.87

[SNP] =  $2.013 \times 10^{-3} \text{ M}$  [Dimethylthiourea] =  $3.64 \times 10^{-3} \text{ M}$   
 Light Intensity =  $16.0 \text{ mWcm}^{-2}$   $\lambda_{\text{max}} = 620 \text{ nm}$  pH = 8.4



**Figure-A-4: Effect of Thiourea**

### Effect of Dimethylthiourea concentration

The effect of dimethylthiourea concentration on the rate of sodium nitroprusside photochemical reaction was also observed by taking different dimethylthiourea concentrations. The results are reported in Table-A-5.

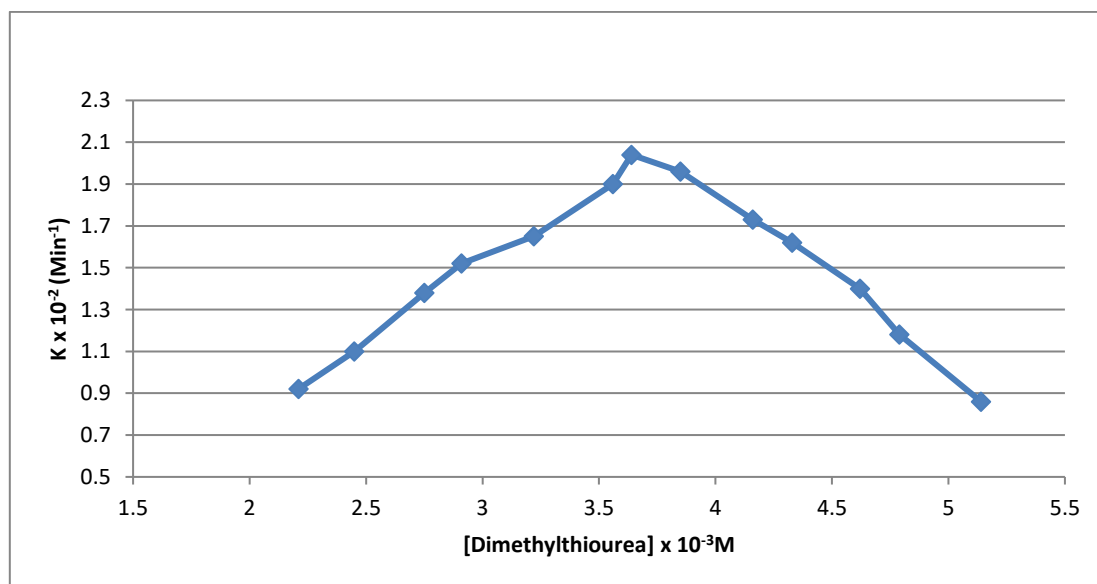
It was observed that there was a corresponding increase in the reaction rate as the concentration of Dimethylthiourea was increased, reaching a maximum at  $[\text{Dimethylthiourea}] = 3.64 \times 10^{-3} \text{ M}$ . It can be explained because the reaction rate increases due

to an increase in participating species concentration. On the other hand, on increasing the concentration of Dimethylthiourea above  $3.64 \times 10^{-3} \text{ M}$ , a decrease in the reaction rate was observed. It can be attributed to the fact that in a desired time limit, higher concentration of Dimethylthiourea may impede its own movement to reach the excited species of sodium nitroprusside. As a result, a decrease in product yield was observed for higher Dimethylthiourea ion concentrations.

**Table-A-5: Effect of Dimethylthiourea**

<b>[Dimethylthiourea] x 10<sup>-3</sup>M</b>	<b>K x 10<sup>-2</sup> (Min<sup>-1</sup>)</b>
2.21	0.92
2.45	1.10
2.75	1.38
2.91	1.52
3.22	1.65
3.56	1.90
<b>3.64</b>	<b>2.04</b>
3.85	1.96
4.16	1.73
4.33	1.62
4.62	1.40
4.79	1.18
5.14	0.86

$[\text{SNP}] = 2.013 \times 10^{-3} \text{ M}$      $[\text{Thiourea}] = 6.30 \times 10^{-3} \text{ M}$   
 Light Intensity =  $16.0 \text{ mWcm}^{-2}$      $\lambda_{\text{max}} = 620 \text{ nm}$  pH = 8.4



**Figure-A-5: Effect of Dimethylthiourea**

### Effect of light intensity

The effect of light intensity on sodium nitroprusside's photochemical reaction rate was observed by varying the distance between the reaction mixture's exposed surface and the light source. The results are reported in Table-A-6.

The above results indicate that the rate of reaction is accelerated as the light intensity has been increased. This may be because any increase in light intensity increases the number of striking photons per unit area of the reaction mixture. No reasonable increase in the reaction rate was observed after a certain limit (i.e.  $I = 16.0 \text{ mWcm}^{-2}$ ) when using higher light intensities, instead it reaches a saturation point at a

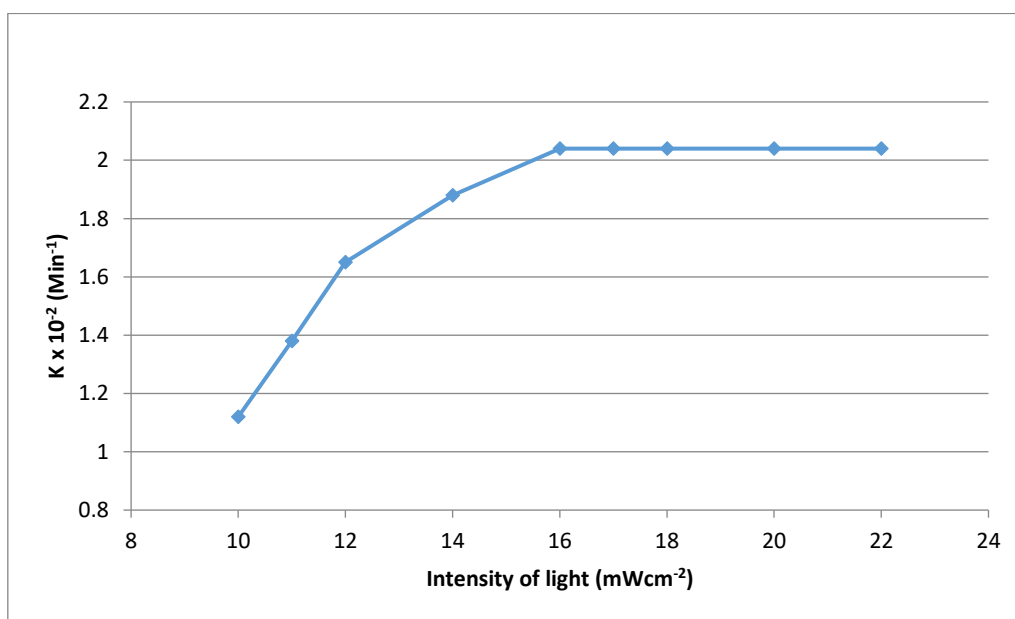
particular intensity. It suggests that further light intensity increases will no longer increase this photochemical reaction rate as the reaction mixture surface remains fixed. When vessels of smaller dimensions were used, this point was moved to reduce value. An inverse trend has been achieved with vessels of larger dimensions.

In various steps of this response, an attempt was produced to understand the need for light. It was noted that when the light source was cut off, the response stops at a specific step. The response only comes to an end when the light exposure continued throughout the reaction advancement.

**Table-A-6: Effect of light intensity**

Intensity of light ( $\text{mWcm}^{-2}$ )	$K \times 10^{-2} (\text{Min}^{-1})$
10	1.12
11	1.38
12	1.65
14	1.88
<b>16</b>	<b>2.04</b>
17	2.04
18	2.04
20	2.04
22	2.04

[SNP] =  $2.013 \times 10^{-3} \text{ M}$  [Thiourea] =  $6.30 \times 10^{-3} \text{ M}$   
 [Dimethylthiourea] =  $3.64 \times 10^{-3} \text{ M}$   $\lambda_{\text{max}} = 620 \text{ nm}$   
 pH = 8.4



**Figure-A-6: Effect of light intensity**

### Analysis of the Product

The photochemical reaction with thiourea and dimethylthiourea between sodium nitroprusside was performed. The original ligand color of the reaction blend has been noted to alter to peacock blue. It

permitted the response to proceed to completion and then it was filtered. Evaporated on a water bath, the filtrate was recrystallized with methanol giving the product red crystals, which was analysed as follows.

**Table-A-7: Elemental Analysis**

Element	Found (%)	Calculated (%)
Na	6.32	6.19
Fe	15.21	15.09
C	22.60	22.64
N	30.38	30.18
O	4.05	4.31
H	4.11	4.31
S	17.07	17.25

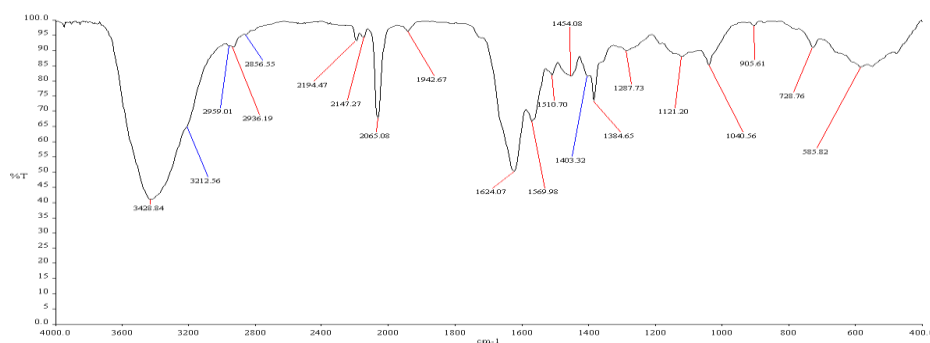
### IR spectral studies

While studying I.R. Spectrum of complex, -OH stretching band was found absent in the product. When comparing -NH<sub>2</sub> stretching band were found 3373 – 3276 cm<sup>-1</sup> for thiourea and 3237 cm<sup>-1</sup> for DMTU also at 3212 and 3428 cm<sup>-1</sup> in the product. The -C≡N Stretching vibration of sodium nitroprusside dehydrated shows at 2144.25 cm<sup>-1</sup> and 2065.08 cm<sup>-1</sup> in the spectra of complex. -N=O stretching and -OH bending vibrations were found absent in the newer complex.

The -C=S stretching band were found at 1620 cm<sup>-1</sup> thiourea and 1666-1626 cm<sup>-1</sup> for DMTU, also at 1569.8 cm<sup>-1</sup> in the product. >C=O stretching and -C-N bending vibrations were found at 1642.07 cm<sup>-1</sup> and 1384.65 cm<sup>-1</sup> in the product. While -CH and -NH bending vibrations were found at 1040.56 and 905.61 cm<sup>-1</sup>. The infrared spectra of the complex confirm that the thiourea and dimethylthiourea ligand are coordinated to the metal ions via two exocyclic sulphur and one- oxygen atom.

**Table-A-8**

Sr. No.	Functional Group	Sodiumnitroprusside Dihydrate (cm <sup>-1</sup> )	Thiourea (cm <sup>-1</sup> )	DMTU (cm <sup>-1</sup> )	Compound IV (cm <sup>-1</sup> )
1	-OH (H <sub>2</sub> O) (Stretching)	3828.77 & 3547.94	-	-	-
2	-NH <sub>2</sub> (Stretching)	-	3473 & 3276	3237	3212, 3428
3	-C≡N (Stretching)	2144.25	-	-	2065.08
4	-N=O (Stretching)	1951.18	-	-	-
5	-OH (H <sub>2</sub> O) (Bending)	1618.79	-	-	-
6	-C=S (Stretching)	-	1620	1666 & 1626	1569.98
7	>C=O (Stretching)	-	-	-	1642.07
8	-C-N (Bending)	1364.27	1414	1359	1384.65
9	-CH <sub>3</sub> (Bending)	-	-	1040	1040.56
10	-NH <sub>2</sub> (Bending)	-	1086	-	905.61



**Figure-A-8**

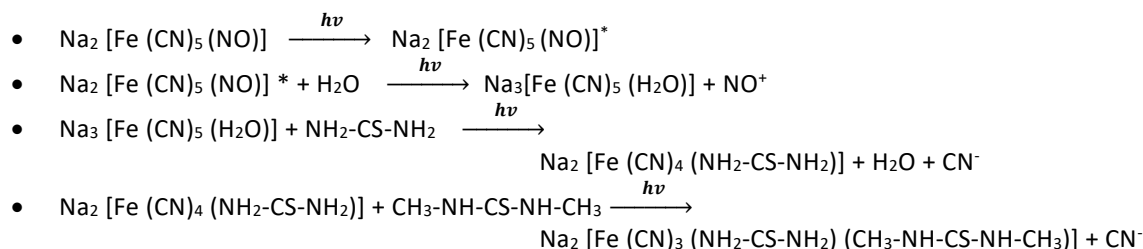
The following tentative structure for the compound was suggested on the grounds of spectral and analytical information.



Thiourea and Dimethylthiourea ion act as a bidentant ligand in this situation.

## MECHANISM

A tentative mechanism for the photochemical ligand exchange reaction of sodium nitroprusside was suggested as below based on the experimental findings.



## ANTIMICROBIAL ACTIVITY

The tests were performed in triplicate and the results are reported as its mean as summarized in table A- 9 and A-10.

Table-A-9: MIC for *E-coli*

T.T. No.	Amount of suspension of <i>E-coli</i> (ml)	Concentration of compound(mg/ml)	O.D.
1	0.2	-	0.36
2	0.2	0.1	0.79
3	0.2	0.2	0.69
4	0.2	0.3	0.55
5	0.2	0.4	0.50
6	0.2	0.5	0.42
7	0.2	0.6	0.36
8	0.2	0.7	0.30
9	0.2	0.8	0.29
10	0.2	0.9	0.23
11	0.2	1.0	0.17
12	-	1.0	0.16

Growth was found to be inhibited at 10 mg /ml concentration. (At 540 nm)

1<sup>st</sup> tube was positive control and 12<sup>th</sup> tube was negative control.

Table-A-10: MIC for *Saccharomyces cerevisiae*

T.T. No.	Amount of suspension of <i>Saccharomyces cerevisiae</i> (ml)	Concentration of compound(mg/ml)	O.D.
1	0.2	-	0.91
2	0.2	0.1	0.45
3	0.2	0.2	0.37
4	0.2	0.3	0.33
5	0.2	0.4	0.31
6	0.2	0.5	0.24
7	0.2	0.6	0.22
8	0.2	0.7	0.15
9	0.2	0.8	0.14
10	0.2	0.9	0.13
11	0.2	1.0	0.11
12	-	1.0	0.10

The zones of inhibition formed for the compound against bacteria and fungi are summarized in table A-10. Compound exhibited significant activities against various strains of microorganisms.

This compound was found to be more effective against *Bacillus* and *Aspergillus niger*. It showed no antimicrobial activity against *E-coli* and *Saccharomyces cerevisiae* though the activity was found to be slightly less than the reference drug.

**Table-A-11: Antimicrobial activity**

No.	Organisms	Zone Size (cm.)		
		Compound	Streptomycin	Fluconazole
1	<i>E-coli</i>	-	2.7	-
2	<i>Bacillus</i>	1.6	2.3	-
3	<i>Saccharomyces cerevisiae</i>	-	-	1.65
4	<i>Aspergillus niger</i>	1.45	-	2.0

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