



# Evaluation of Hexadate Ligand 1, 3-bis(2,2':6',2''-Terpyridyl-5-Ylmethylsulfany l)Propane in the Determination of Iron(II) in Solution by Spectrophotometric and Fluoremetric Methods of Analysis

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**How to cite this paper:** Maritim, P.K. (2017) Evaluation of Hexadate Ligand 1, 3-bis(2,2':6',2''-Terpyridyl-5-Ylmethylsulfany l) Propane in the Determination of Iron(II) in Solution by Spectrophotometric and Fluoremetric Methods of Analysis. *Open Access Library Journal*, 4: e3715.

<https://doi.org/10.4236/oalib.1103715>

**Received:** June 6, 2017

**Accepted:** August 28, 2017

**Published:** August 31, 2017

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## Abstract

Uv-visible and fluorescence spectra of the ligand 1,3-bis(2,2':6',2''-terpyridyl-5-ymethylsulfanyl)propane **L** and it's iron(II) complex have been investigated for analytical purposes. The two spectra of **L** and terpy are very similar which confirmed the ability of **L** to co-ordinate through the six N atoms of **L** with minimum distortion of the metal ion's octahedral geometry. The ligand-based absorption band of **L** is shifted to the longer wavelength. It was found that **L** is able to displace the two terpyridine groups in the complex to give  $[\text{FeL}]^{2+}$ . The high stability of the complex makes it good in spectrophotometry analysis of metals ions in solution. The fluorescence of **L** was progressively quenched with an increasing concentration of iron(II). This makes **L** a possible reagent for the quantitative analysis of metal by measuring fluorescence quenching.

## Subject Areas

Analytical Chemistry

## Keywords

Ligand, Fluorescence, Spectrophotometry

## 1. Introduction

Spectrophotometric and fluorescence methods are sensitive for metal ion analy-

sis. The equipment is compact and easy to use. This has made the two methods popular, and they are used in routine laboratories all over the world. Any attempt to improve on detection limits of this type of equipment is valuable.

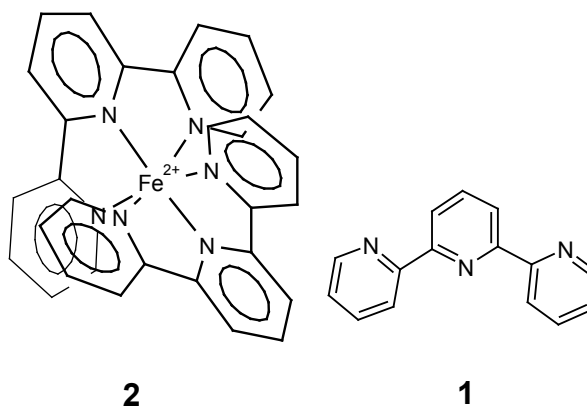
Spectroscopy analysis is based on the relationship between the degree of absorption and the concentration of the absorbing materials. Some ligands formed coloured complex with metals. The intense of the colour of the metal complexes can make spectrophotometer instruments to be able to detect very low concentration of metal, hence improving the detection limit.

Fluorescence is one of the several mechanisms by which a molecule can return to the ground state after being excited by absorption of radiation. All molecules have a potential to fluoresce but most do not, because their structure provides a radiationless pathway at a greater rate than the fluorescence emissions. Compounds that can fluoresce are often rigid molecules such as aromatics, compounds with conjugated double bonds, and heterocycles.

Inorganic fluorimetry is often based upon the reaction of an analyte with a chelating agent to form a complex that fluoresces or upon measurement of fluorescence quenching as a result of the analyte co-ordination. The fluorescence is measured at  $90^\circ$  to the excitation light. Fluorescent spectroscopy can often give high sensitivity and specificity. In favourable cases, it can measure as low as  $10^{-9}$  mg/cm<sup>3</sup> of analyte. The intense colour of the terpyridine metal complexes can make such instruments capable of detecting very low concentration of metal, hence improving the detection limit [1].

It has been recognised that metals sometimes react with ligands to form intensely coloured compounds. These ligands have been used for the determination of metal ions in solution at very low concentration. There are a number of ligands that are available for analysis of metals. Those for determining low concentration of iron(II) include 2,2'-bipyridyl, 4,7-diphenyl-10-phenanthroline, and 2,4,6-tri(2-pyridyl)-1,3,5-triazine; they react with iron(II) to form Fe(II) complexes with their molar extinction coefficient ( $\epsilon_{\text{max}}$ ) of 800, 2240, 2260 dm<sup>3</sup> mol<sup>-1</sup>·cm<sup>-1</sup> respectively as demonstrated by R. C. Denny *et al.* [2]. Another known ligand which is used for the determination of iron(II) by spectrophotometry is 2,2':6',2''-terpyridine. 2,2':6',2''-terpyridine was first isolated by Morgan and Burstall [3] in 1937 in low yield. Since then, varied and exciting co-ordination chemistry of 2,2':6',2''-terpyridine has been established as demonstrated by E. C. Constable *et al.* [4]. This has led to the development of synthetic strategies enabling a wide range of its substituted analogues to be prepared in good yield. Today the parent ligand **1** is commercially available [5].

This ligand **1** has been recognised as a useful ligand in the fields of organic, organometallic and co-ordination chemistry. It is a good ligand for analytical chemistry since it forms bis(terpyridine) metal complexes with many metal ions e.g. the iron(II) complex **2** which has blood red colour. 2,2':6',2''-Terpyridine co-ordinates strongly through the three nitrogen atoms, and remains planar in a meridional mode of co-ordination.



Terpyridine ligands (terpy), and their metal complexes, can have very intense colours which arise from electronic transitions  $\pi \rightarrow \pi^*$  and metal to ligand charge transfer. The terpyridine ligand possess low-lying  $\pi^*$  orbital which are able to accept electrons density from the metal as earlier explain by G. T. Morgan *et al.* [3]. The strong colour which results from this metal to ligand charge transfer band is useful for trace metal analysis because even in small quantities metal ions can produce compounds with high absorbance.

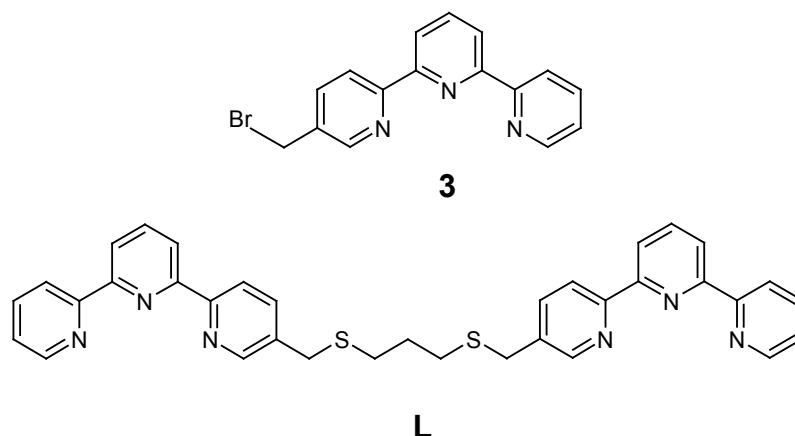
It has been reported that 2,2':6'2''-terpyridine (terpy) and its substituted derivative form stable complexes with transition metals such as iron(II) and nickel (II) [6] [7] [11].

For example bis(2,2':6',2''-terpyridine) iron(II) complex which is stable over a wide range of pH, and has a high molar extinction coefficient ( $\epsilon_{552}$  12 500  $\text{dm}^3 \cdot \text{mol}^{-1} \cdot \text{cm}^{-1}$ ) as earlier reported by R. L. Morris [8]. Therefore, pyridine is a highly sensitive reagent for  $\text{Fe}^{2+}$  determination. It has been used to determine  $\text{Fe}^{2+}$  in water in the present of heavy metals [9].

The detection limit in the use of these ligands depends on the stability of the complexes that are formed, and on the magnitude of the molar extinction coefficients of the complexes.

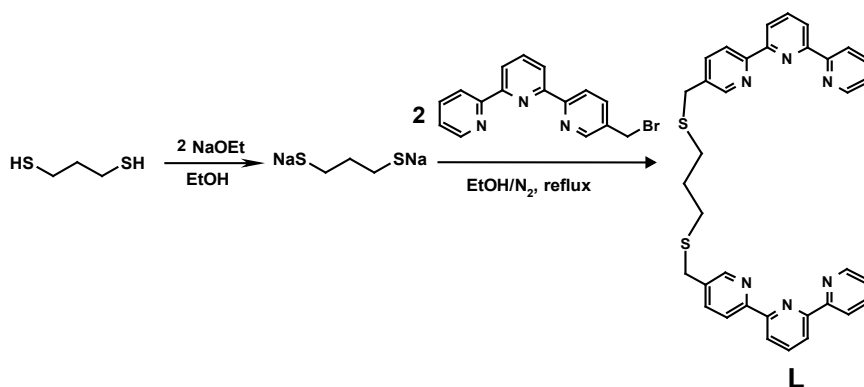
In search for even more ligand that forms more stable complexes with metals we synthesis and characterise a new ligand (1,3-bis(2,2':6',2''-terpyridyl-5-yl-methylsulfanyl) propane **L** as reported by Gleb *et al.* [12]. In the study, two terpyridines derivatives (**3**) were linked together through the reaction of a dithiol with sython **3**. The 5-substituted bromomethyl derivative **3** is ideal for the proposed complex in order to avoid the sulphurs at the "bridge" from taking part in co-ordination with the metal ions. The aim here was to sythesis a complex of a single metal by co-ordination with the two bridged terpyridines to form octahedral complex. The use of 1,3-propanedithiol has advantages in this case because sulphur atoms of the product **L**, unlike nitrogen atoms, do not undergo protonation in aqueous solutions. Therefore, not pH dependent like nitrogen atoms. The ligand was synthesised from 5-bromomethy-2,2':6'2''-terpyridine **3** obtained by method of P. Sheldon [10]. The ligand was found to form stable complexes nickel(II).

Therefore the objective of this study is to react ligand **L** with iron(II) and evaluate whether can be used in determining iron in solution by UV/Vis spectroscopy and Fluorescence method of analysis.



## 2. Results and Discussion

The ligand **L** was synthesis using the methods reported by Gleb *et al.* 2000 [12] from Bromo-terpy **3** as shown on the scheme provided in Scheme 1.



### 2.1. Spectrophotometric Analysis

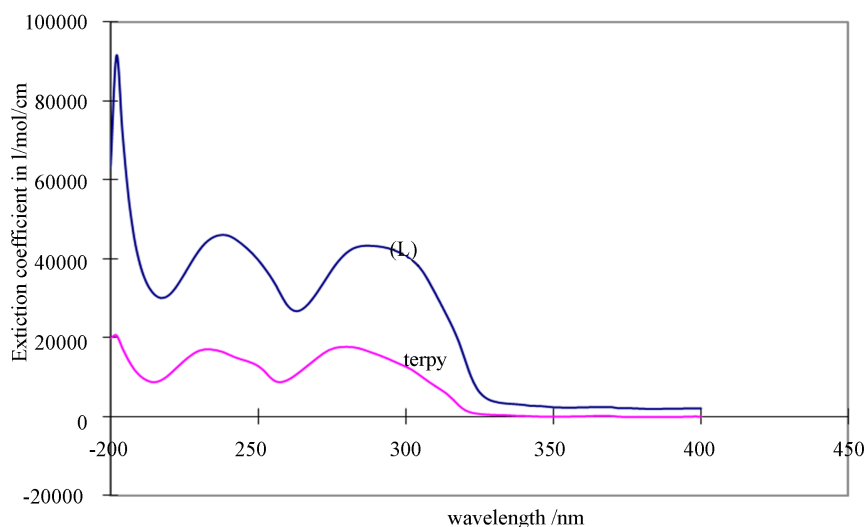
#### 2.1.1. Comparison of uv Spectra of **L** and Terpy

The electronic absorption spectra of **L** and terpy (**3**) are shown in **Figure 1**.

**L** and terpy show  $\lambda_{\max}$  at 238 nm and 280 nm respectively. Their molar extinction coefficients ( $\epsilon_{\max}$ ) are  $46,000 \text{ dm}^3 \cdot \text{mol}^{-1} \cdot \text{cm}^{-1}$  and  $18,000 \text{ dm}^3 \cdot \text{mol}^{-1} \cdot \text{cm}^{-1}$  respectively. It was noted that both terpy and **L** exhibit similar electronic transitions. Curve (**L**) indicates that one linked terpyridine has a higher absorbance than terpyridine alone, and the longest wavelength peak is shifted to a longer wavelength.

#### 2.1.2. Comparison of uv-Visible Spectra of $[\text{FeL}]^{2+}$ and $[\text{Fe(Terpy)}_2]^{2+}$

A uv/vis spectra of methanolic solutions of  $[\text{Fe(terpy)}_2]^{2+}$  and  $[\text{FeL}]^{2+}$  was obtained at concentrations of  $1.43 \times 10^{-5} \text{ mol} \cdot \text{dm}^{-3}$  and  $1.19 \times 10^{-5} \text{ mol} \cdot \text{dm}^{-3}$  respec-



**Figure 1.** Comparison of UV spectra of (L) and (terpy).

tively and then the normalised. The two spectra were very similar which confirmed the ability of L to co-ordinate through the six N atoms of L with minimum distortion of the metal ion's octahedral geometry. The ligand-based absorption band of L is shifted to the longer wavelength. They both exhibit a metal to ligand charge-transfer band at 552 nm of molar extinction coefficients.

( $\epsilon_{552 \text{ nm}}$ ) of  $11,500 \text{ dm}^3 \cdot \text{mol}^{-1} \cdot \text{cm}^{-1}$   $[\text{FeL}]^{2+}$  and  $9050 \text{ dm}^3 \cdot \text{mol}^{-1} \cdot \text{cm}^{-1}$   $[\text{Fe}(\text{terpy})_2]^{2+}$ .

The two complexes show two ligand-based absorption bands  $[\text{FeL}]^{2+}$  at 276 and 328 nm, and  $[\text{Fe}(\text{terpy})_2]^{2+}$  at 273 and 319 nm. The maximum molar extinction coefficients for each of the complexes are ( $\epsilon_{328 \text{ nm}}$ )  $41,800 \text{ dm}^3 \cdot \text{mol}^{-1} \cdot \text{cm}^{-1}$   $[\text{FeL}]^{2+}$  and ( $\epsilon_{319 \text{ nm}}$ )  $41,200 \text{ dm}^3 \cdot \text{mol}^{-1} \cdot \text{cm}^{-1}$   $[\text{Fe}(\text{terpy})_2]^{2+}$ .

## 2.2. Fluorescence Measurements

A solution of L ( $4.92 \times 10^{-6} \text{ mol} \cdot \text{dm}^{-3}$ ) was reacted with increasing concentrations of standardised  $\text{Fe}^{2+}$  ions of concentration between ( $2 \times 10^{-6} - 2 \times 10^{-5} \text{ mol} \cdot \text{dm}^{-3}$ ) to form  $[\text{FeL}]^{2+}$ . The fluorescence of these solutions was scanned in a 1 cm fluorescence cuvette from 350 - 600 nm with an excitation at 276 nm.

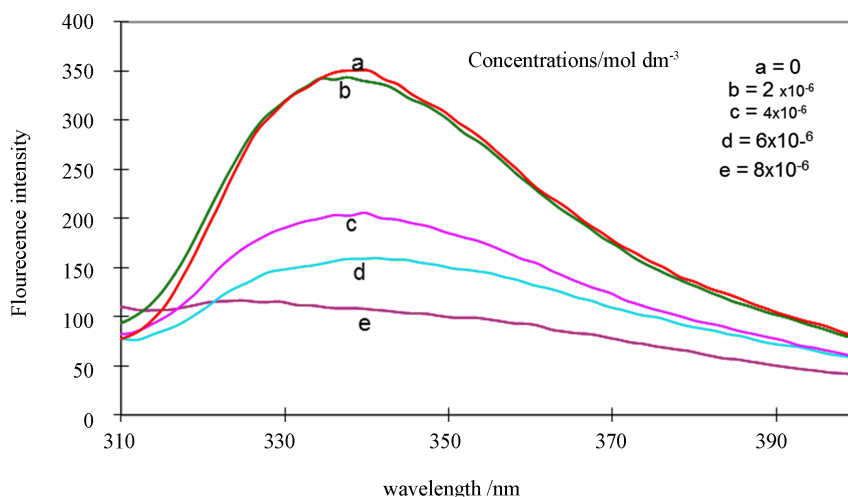
L is luminescent with a maximum in the emission spectrum at 350 nm.

**Figure 2** shows the effect on the luminescence of L with added increasing amount of  $\text{Fe}^{2+}$ . The luminescence of L is quenched progressively with increase in the concentration of iron(II). It can be noticed that at concentration e fluorescence intensity as reached the lowest hence its detection limits. This makes L a good reagent for the qualitative analysis of metal ions by measuring the fluorescence quenching.

## 3. Experimental

### 3.1 Synthesis of $[\text{FeL}](\text{PF}_6)_2$

A solution of iron(II) tetrafluoroborate (28.8 mg, 0.0835 mmol) in methanol (30



**Figure 2.** Change of Fluorecence spectra of methanolic solutions of L ( $4.92 \times 10^6$  mol-dm<sup>3</sup>) with increasing [Fe<sup>2+</sup>] (excitation at 276 nm).

cm<sup>3</sup>) was added dropwise to a solution of L (50 mg, 0.0835 mmol) in methanol (refluxed to dissolve), and the resulting deep purple solution was stirred for 30 minutes. Excess methanolic ammonium hexafluorophosphate was added, and the volume reduced with a rotary evaporator to precipitate [FeL](PF<sub>6</sub>)<sub>2</sub>. The precipitate was filtered and washed with ether to give the complex as fine purple solid, (yield 41 mg, 53%).

<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>): δ/p.p.m 161.26, 160.86, 158.92, 156.44, 155.45, 153.61, 141.82, 139.54, 139.47, 138.94, 128.14, 124.4, 124.04, 123.74, 118.14, 33.06, 31.05, 29.08. Elemental Analysis; observed C 42.1, H 3.85, N 8.06 and calculated for C<sub>35</sub>H<sub>30</sub>N<sub>6</sub>S<sub>2</sub>P<sub>2</sub>F<sub>12</sub>Fe 2.5H<sub>2</sub>O is C 42.5, H 3.56, N 8.49% FAB mass spectrum; calculated for C<sub>35</sub>H<sub>30</sub>N<sub>6</sub>S<sub>2</sub>P<sub>2</sub>F<sub>12</sub>Fe *i.e.* [FeL][PF<sub>6</sub>]<sub>2</sub> m/z 944.6.

Found no molecular ion but daughter ion at m/z 799, 673, and 654.

### 3.2. Standardisation of FeSO<sub>4</sub>·(NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>·6H<sub>2</sub>O with Standard 0.1 mol·dm<sup>-3</sup> Cerium(IV) Sulphate

Ferriin indicator was first prepared by dissolving FeSO<sub>4</sub>·(NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>·6H<sub>2</sub>O (784 mg, 0.2 mmol) in deionised water (10 cm<sup>3</sup>). To this solution, 1,10-phenanthroline monohydrate (120 mg, 0.6 mmol) was added to form [Fe(phen)<sub>3</sub>]<sup>2+</sup> (ferriin) which has a red blood colour [13]. FeSO<sub>4</sub>·(NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>·6H<sub>2</sub>O 1.961 gm, (5.0 mmol) was dissolved in N<sub>2</sub> scrubbed deionised water and made to 50 cm<sup>3</sup> in a volumetric flask. 5.0 ± 0.1 cm<sup>3</sup> of this solution was transferred into a conical flask, 0.5 cm<sup>3</sup> of 0.5 mol·dm<sup>-3</sup> sulphuric acid and a drop of ferriin was added. This solution was titrated with standard 0.1 mol·dm<sup>-3</sup> cerium(IV) sulphate solution to the end point red to green. Titration was repeated four times and an average of the volume of Ce<sup>4+</sup> taken. The four titre values obtained were 5.04, 5.03, 5.29 and 5.03 cm<sup>3</sup>. The average is 5.03 ± 0.01. The concentration of the Fe<sup>2+</sup> was found to be 0.101 ± 0.002 mol·dm<sup>3</sup>.

### 3.3. Fluorescence Analysis

**Table 1** demonstrates how  $\text{Fe}^{2+}$  solution of original concentration of  $2 \times 10^{-4} \text{ mol}\cdot\text{dm}^{-3}$  were diluted. A volume of 49.5 microliters of the standardised solution of  $0.101 \text{ mol}\cdot\text{dm}^{-3} \text{ FeSO}_4\cdot\text{NH}_4)_2\text{SO}_4\cdot 6\text{H}_2\text{O}$  was taken and diluted to  $25 \text{ cm}^3$  in a  $25 \text{ cm}^3$  volumetric flask with spectrophotometric methanol to give a solution of concentration  $2 \times 10^{-4} \text{ mol}\cdot\text{dm}^{-3}$ . A solution of **L** (4.915 mg,  $8.1 \times 10^{-6}$  moles) was prepared with methanol in a  $50 \text{ cm}^3$  volumetric flask to give a solution of concentration  $1.62 \times 10^{-4} \text{ mol}\cdot\text{dm}^{-3}$ . This solution was diluted and mixed as shown in the table below. The concentration of **L** was kept constant at  $4.92 \times 10^{-6} \text{ mol}\cdot\text{dm}^{-3}$  whilst that of  $\text{Fe}^{2+}$  was varied.

Each solution was transferred in turn to a quartz fluorescence cuvette and fluorescence emission spectrum was scanned between 200 - 450 nm with a Perkin Elmer LS50 luminescence spectrometer. The excitation wavelength was 276 nm (exit slit 5 nm and em. slit 10 nm). The spectrum was processed using a spread sheet.

### 4. Conclusions

The electronic absorption spectra of **L** and terpy (**3**) are shown in **Figure 1**.

**L** and terpy show  $\lambda_{\text{max}}$  at 238 nm and 280 nm respectively. Their molar extinction coefficients ( $\epsilon_{\text{max}}$ ) are  $46,000 \text{ dm}^3\cdot\text{mol}^{-1}\cdot\text{cm}^{-1}$  and  $18,000 \text{ dm}^3\cdot\text{mol}^{-1}\cdot\text{cm}^{-1}$  respectively. It was noted that both terpy and **L** exhibit similar electronic transitions. The UV/Vis spectra of both ligand **L** and Terpy are very similar which confirmed the ability of **L** to co-ordinate through the six N atoms of **L** with minimum distortion of the metal ion's octahedral geometry. The ligand-based absorption band of **L** is shifted to the longer wavelength. They both exhibit a metal to ligand charge-transfer band at 552 nm of molar extinction coefficients ( $\epsilon_{552 \text{ nm}}$ ) of  $11,500 \text{ dm}^3\cdot\text{mol}^{-1}\cdot\text{cm}^{-1}$  and  $9050 \text{ dm}^3\cdot\text{mol}^{-1}\cdot\text{cm}^{-1}$  respectively.

The stability of  $[\text{FeL}]^{2+}$  is demonstrated by the ability of **L** to displace both terpy ligands in  $[\text{Fe}(\text{terpy})_2]^{2+}$  which was as earlier discussed by Gleb *et al.* [12]. The equilibrium formation constant  $\beta_2$  of  $[\text{Fe}(\text{terpy})_2]^{2+}$  is  $10^{20.9}$  [11] [12] and from the above observation,  $\beta_1$  of  $[\text{FeL}]^{2+}$  is more than this. The exact value of  $\beta_1$  for  $[\text{FeL}]^{2+}$  could be established by competition experiments with other multidentate ligands of known donor strength.

**Table 1.** Preparation of **L** and  $\text{Fe}^{2+}$  solutions for fluorescence analysis.

Volume of <b>L</b> /cm <sup>3</sup>	Volume of $\text{Fe}^{2+}$ /cm <sup>3</sup>	Volume of solvent (metha-nol)/cm <sup>3</sup>	Final concentration of $\text{Fe}^{2+}$ /mol·dm <sup>-3</sup>
0.3	0.40	9.30	$8 \times 10^{-6}$
0.3	0.30	9.40	$6 \times 10^{-6}$
0.3	0.20	9.50	$4 \times 10^{-6}$
0.3	0.10	9.60	$2 \times 10^{-6}$
0.3	0.00	9.70	0

Uv spectra of both **L** and terpy are very similar.

Iron(II) is low spin when complex with **L**, which contributes to the high stability of the complex, therefore, making **L** a good reagent for the determination of iron(II) by spectrophotometric method of analysis.

The ligand **L** is fluorescent. From the results in **Figure 2**, the fluorescence is progressively quenched by increased concentration of the iron(II). Therefore, **L** can be used for the determination of some metal ions by measuring the fluorescence quenching.

The use of **L** for the analysis of other Fe(II) ions in the presence of several other metal ions, e.g. in industrial waste water, needs further investigation.

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