



ISSN (E): 2277-7695  
 ISSN (P): 2349-8242  
 NAAS Rating: 5.23  
 TPI 2022; 11(9): 774-777  
 © 2022 TPI  
[www.thepharmajournal.com](http://www.thepharmajournal.com)  
 Received: 12-06-2022  
 Accepted: 22-08-2022

Gajanan W Belsare  
 Department of Chemistry, Shri  
 Shivaji College of Arts,  
 Commerce and Science, Akola,  
 Maharashtra, India

## Sensitization of Pyrogallol red with Cetylpyridinium bromide for the spectrophotometric determination of $Pb^{2+}$

Gajanan W Belsare

### Abstract

The sensitization of Pyrogallol Red (PGR), a member of triphenylmethane dye in presence of Cetylpyridinium bromide (CPB), a cationic surfactant has been studied. Addition of  $Pb^{2+}$  solution in the modified reagent (PGR + CPB) formed intense coloured complexes with the shift in  $\lambda$  max and increase in molar absorptivity and sensitivity. The change in the properties of Pyrogallol Red (PGR + CPB) utilized for the Microdetermination of Lead.

**Keywords:** Sensitization, pyrogallol red, cetylpyridinium bromide, molar absorptivity etc.

### 1. Introduction

Water, soil and food products are contaminated by different sources, like mining steel industry, crop enhancers automobiles etc. These heavy metals show toxic effect on different human organs. The literature on lead toxicity is far too vast to review in this paper. Instead, this section summarizes the international scientific consensus, based on comprehensive evaluations of the literature conducted by international organizations and national governments. Lead paint is a primary source of lead exposure and the major source of lead toxicity in children. The U.S. Department of Housing and Urban Development currently estimates that 38 million homes in the United States contain lead paint. Of those, 24 million are considered to contain significant lead-based paint hazards, including deteriorating paint and/or contaminated dust or soil outside the home. As lead paint deteriorates and airborne lead settles, it contaminates dust and soil. Exposure to soil that contains particulate lead has been shown to be significantly hazardous for children, who are more commonly exposed by ingestion of house dust or soil than by paint chips. Blood lead levels are more closely related to indoor dust exposure than to outdoor soil exposure. Lead exposure can also occur during remodeling of a home built prior to 1978, when lead-based paints were still in commerce. Many workers carried out the determination of lead in many samples<sup>[1]</sup>. The analytical and complex ion chemistry of Lead have not kept pace with growing interest because the heavy metal ions have less tendency to form complexes with normally strong coordinating agents. This may be due to their large size of its cation. With the development of chelatometry in this context very few reagents have been introduced for the determination of Lead. Spectrophotometric determination of  $Pb(II)$ ,  $Bi(III)$  and  $Fe(III)$  in complexes with 1,2-diaminocyclohexane -N, N, Ni, Ni-tetraacetic acid has been carried out by Jan Krzek *et al.*<sup>[1]</sup> B. Jankiewicz carried out the determination of lead in soil sample<sup>[2]</sup>. A detail spectrophotometric determination of lead with o-hydroxy-thiobenzmorpholide has been carried out by I. Badea *et al.*<sup>[3]</sup> Spectrophotometric determination of lead with 1-(2-pyridylazo)-2-naphthol and non-ionic surfactants has been carried out by Escriche JM *et al.*<sup>[4]</sup> A detail Spectrophotometric determination of lead in foods with dibromo-p-methyl-bromosulfonazo has been carried out by Escriche. The synthesis and purification of a new chromogenic reagent dibromo-p-methyl-bromosulfonazo (DBMBSA) has been synthesized by Guozhen Fang for the spectrophotometric determination of lead in food stuff<sup>[5]</sup>. A sensitive spectrophotometric method for the determination of lead in aqueous solutions has been developed and applied for a range of concentration of 0-60  $\mu g/ml$  of lead using the complex Lead (II) Using 3-Methyl-1, 2-Cyclopentanodione Dithiosemicarbazone<sup>[6]</sup>, Thus it becomes important to sensitize these reagents by some process. The present work primarily been undertaken to explore the utility of cationic surfactant such as Cetylpyridinium bromide (CPB) in the sensitization of Pyrogallol Red (PGR) for the determination of lead.

**Corresponding Author:**  
 Gajanan W Belsare  
 Department of Chemistry, Shri  
 Shivaji College of Arts,  
 Commerce and Science, Akola,  
 Maharashtra, India

In this context new and sensitive method is proposed which will helpful to determine percentage toxicity of lead in micrograms. The proposed method will be applicable to control water, air pollution causes because of these toxic metals in future.

## 2. Material and Methods

### 2.1 Instrumentation and Reagent solutions

All the spectral measurements have been carried out on Chemline model CL 133 microcontroller based spectrophotometer with glass cuvettes of light paths 10mm. distilled water has been used as a reference solution. Chemline model CL 180 pH meters with combine electrode has been used for the adjustment of pH. The scale has been standardizing every day before making the pH measurement with buffer solutions of pH 4.0, 7.0 & 9.0. The pH of the each solution has been adjusted with HCl and NaOH solution of suitable concentration.

The Pyrogallol Red and Cetylpyridinium bromide were used in this work were of analytical grade purity and supplied by Sigma-Aldrich chemical company, USA. The Lead acetate of analytical grade purity was supplied by Merck chemical company. The solution of both the reagents have been prepared by using distilled water using ethanol. The stock solution of all reagents have been prepared in the concentration  $1.0 \times 10^{-2} M$ . The HCl and NaOH used was supplied by SD fine chemical laboratories.

### 2.2 General Procedure

All experiments are carried out at room temperature  $30 \pm 2$  °C.

The CPB solution was first added to the PGR solution and kept for equilibration for half an hour. The metal solution was then added in the dye-surfactant solution and kept for half an hour for complete equilibration. This order of mixing of solution was maintained throughout the investigation.

## 3. Result and Discussion

It has been considered necessary to have prior information on the nature of interaction between PGR and CPB before evaluating the PGR as sensitive reagent for the estimation of Lead in the presence of CPB. Therefore, absorption spectra of PGR in absence and presence of CPB, composition of dye-surfactant complex, absorption spectra of lead in absence and presence of CPB, effect of pH, composition of the chelates in absence and presence of CPB, have been studied.

### 3.1.1 Absorption spectra of PGR in the absence and presence of CPB

The color of PGR has been found to be different at different pH values. The addition of CPB brings about a slight change in color of PGR at the same pH value. The absorption spectra of PGR, has been therefore, studied at different pH values (3.0 to 10.0) in the absence and presence of CPB. The wavelength of maximum absorbance of PGR in the absence and presence of CPB are summarized in table 1.

Absorption spectra of alkaline PGR solution at pH 8.0 show a characteristics maximum at 580 nm in presence of CPB with the increase in the absorbance value. This may be due to the formation of dye-detergent complex.

**Table 1:** Wavelengths of maximum absorbance of PGR in the presence and absence of CPB.

PGR		PGR + CPB	
pH	$\lambda_{max}$ (nm)	pH	$\lambda_{max}$ (nm)
1.0-3.0	480	1.0-3.0	480
4.0-4.5	520	4.0-4.5	520
5.0-12.0	560	5.0-12.0	600

### 3.1.2 Composition of PGR-CPB Complex

The effect of varying CPB concentration on the absorbance of PGR has been studied in basic medium at pH 8.0, at  $\lambda_{max}$ , 440 nm where the maximum discoloration takes place. The absorbance of different concentrations of PGR is plotted against the variable concentration CPB. It has been observed that the two times higher concentration of CPB required for complete decolorization of PGR. Thus, the ratio of PGR: CPB will be 1:2 the modified reagent species thus formed, May therefore, be written as  $[PGR (CPB)_2]$ .

### 3.1.3 Absorption Spectra of Lead-PGR Chelates in Presence and Absence of CDMEAB

A series of solutions were prepared keeping the ratio of  $Pb^{2+}$ : PGR:CPB as 1:1:5 and 4:1:5. A number of sets were prepared for each ratio and pH was adjusted to 3.0 to 10.0. The absorption Spectra were recorded in the entire visible region from 400 nm to 700 nm. Absorbance maxima of PGR and its complexes with Lead in the absence and presence of CPB have been summarized at different pH values in table 2.

**Table 2:** Absorbance Maxima (nm) of PGR and its chelates in the absence and presence of CPB at different pH. Absorption Spectra of Pb-PGR Chelates in Absence and Presence of CPB

System/pH	4.0	5.0	6.0	7.0
PGR	520	560	560	560
PGR + CPB	520	600	600	600
PGR + $Pb^{2+}$	520	520	620	620
PGR + CPB + $Pb^{2+}$	540	540	660	660

The absorption spectra of PGR shows peak at 440 nm in the presence of CPB in the pH range 3.0 to 5.0 but shows peak at 580 nm in the pH 8.0 to 10.0. In the pH range 3.0 to 7.0, in absence and presence of CPB, the wavelength maxima of PGR shows small no change in  $\lambda_{max}$  and absorbance values; indicating poor complexation. But, change in  $\lambda_{max}$  and

increase in the absorbance value in pH range 8.0 to 10.0 show complex formation in absence and presence of CPB. By comparing the absorption spectra and the absorbance values of the reagent and complex in presence of CPB, it has been observed that the maximum complexation takes place at pH 8.0. Thus bath chromic shift of 140 nm in absence and 20nm

in the presence of CPB have been observed for Lead. The absorption spectra of lead chelate in absence and presence of CPB at pH 8.0 has been recorded in Fig 3

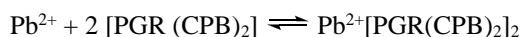
### 3.2 Effect of pH

Effect of pH on  $\lambda_{\max}$  and on the absorbance, of the Lead complexes of PGR in the absence and presence of tenfold excess of CPB have been studied. It is found that the  $\lambda_{\max}$  of the complexes remain constant in the pH range 8.0-10.0 indicating pH range of stability of complex formation in presence and absence of CPB.

### 3.3 Composition of Chelates

The composition of the chelates has been studied by the Mole ratio method. solutions of Lead and PGR have been taken in two equimolar concentrations of  $2.0 \times 10^{-5}$  M and,  $4.0 \times 10^{-5}$  M; Five times excess of CPB has been then added for studying the composition in the presence of surfactant.

The stoichiometric composition between the  $Pb^{2+}$  and PGR in the presence and absence of CPB has been found to be 1:2. It has been observed that PGR reagent at pH 8.0 exists as  $[PGR(CPB)_2]$  and therefore, the composition of complexes in the presence of CPB may be written as  $Pb^{2+}[PGR(CPB)_2]_2$  for lead.



### 3.4 Analytical Applications of lead chelates with PGR in absence and in presence of CPB

#### 3.4.1 Order of Addition of Reactants

The sequence of addition of reactants must be followed

strictly. In all the experiments, CPB was first added to PGR solution. This solution was kept for at least 30 minutes for equilibration. To this solution of modified PGR,  $Pb^{2+}$  solution was then added which again kept for 30 minutes for complete formation of the ternary complex.

#### 3.4.2 Rate of Color Formation and Stability of Color at Room Temperature

The color formation does not depend on reaction time and is almost instantaneous. However, the mixtures were kept for 30 minutes for equilibration. The temperature was found to have no effect on color intensity of ternary complexes from 20 °C to 60 °C.

#### 3.4.3 Beer's Law and Effective Photometric Ranges

The linearity between the absorbance of the chelates and concentration of metal ion has been tested by taking the different volumes of metal ion solution ( $1.0 \times 10^{-3}$  M in absence in presence of CPB). The final concentration of PGR taken was  $2.0 \times 10^{-5}$  M, of CPB was  $1.0 \times 10^{-4}$  M. Total volume was kept constant at 25ml at pH 8.0. The absorbance values were measured in the absence of CPB at 580 nm. However, in the presence of CPB, all the spectral measurement was made at 600 nm. The range of Beer's law is given in table 3 in absence and presence of CPB. The effective range for photometric determination was also calculated from this data by Ringbom (23) plot of log of metal ion concentration versus percentage transmittance. Thus, the range as derived by the slope of the curve is selected to be range for the effective photometric determination as given in table 3.

**Table 4:** Photometric Determination of Lead with PGR in the Absence and Presence of CPB. Photometric Determination of Pb-PGR Chelates in presence and absence of CPB

PGR Chelates	pH of Study	Wavelength of Study (nm)	Beers Law Range (ppm)	Effective Photometric range (ppm)	Molar Absorptivity	Sensitivity ( $\mu\text{g}/\text{cm}^2$ )
PGR + $Pb^{2+}$	6.5	600	0.517-3.619	1.551-2.585	3600	1.40
PGR + CPB + $Pb^{2+}$	6.5	660	0.517-4.136	1.034-3.619	5400	1.03

### 4. Conclusion

The spectrophotometric determination of lead with Pyrogallol Red in the presence and absence of Cetylpyridinium Bromide has been studied. Following are the merits of modified method. The sensitization of PGR by the addition of CPB is clear from the fact that the formation of stable ternary complex with Lead occurs at pH range 6.0-7.0 with bath chromic shift in the  $\lambda_{\max}$  of Pb-PGR complexes in the presence of CPB. This change  $\lambda_{\max}$  and high absorbance value is attributed due to the formation of ternary complex system in the presence of CPB compared to the binary system in the absence of CPB. Due to the shifted  $\lambda_{\max}$  towards higher wavelength (From 620 nm to 660 nm) a large difference in the absorbance between the reagents blank (PGR + CPB) and its ternary complex results in enhancement of the sensitivities and molar absorptivities again indicate the great sensitivity of colour reaction. Further, the modified method requires smaller molar concentration of PGR over the metal ion concentration for full colour development and is instantaneous in the presence of CPB, again indicates the stability of the colour reaction. The modified reagent i.e.  $[PGR(CPB)_2]$  has also been found to be extremely useful in the Complexometric titration of the Lead. This modified reagent act as sensitive metallochrome indicator giving a very

sharp colour change at the end of complex metric titration. The increase in the sensitivity and absorptivity facilitate the determination of lead in the given photometric range.

### 5. Acknowledgments

Authors are thankful to Dr. A. L. Kulat, Principal, Shri Shivaji College of Arts, Commerce and Science, Akola for providing necessary facilities

### 6. References

- Jan Krzek, Anna Apola, Mariusz Stolarczyk, Weodzimierz Rzeszueko. Medyczna Str. 30-688 Kraków, Poland; c2000.
- Jankiewicz B, Ptaszyński B, Wiczorek M. Żwirki 36, 90-924 Łódź, Poland; c2000.
- Badea, Luminita Vladescu, Cornelia Cercasov; c2001.
- Escrache JM, Estelles ML, Cabeza AS; c2004.
- Guozhen Fang, Shuangming Meng, Guizhi Zhang, Jiaomai Pan. Yanbei; Normal Institute, Datong 037000.
- Guozhen Fang, Yongwen Liu, Shuangming Meng, Yong Guo; Talanta. 2002;57:1155-1160.
- Anil Zade B, Pawan Kalbande P, Mayuri Umekar S, Gajanan Belsare W. E-Journal of Chemistry. 2012;9(4):2394-2406.

8. Belsare GW, Zade AB, Kalbande PP, Belsare PU. Der Pharma Chemica. 2012;4(3):1226-1238.
9. Upase AB, Zade AB, Kalbende PP. E-J Chem. 2011;8(3):1132-1141.
10. Upase AB, Zade AB, Kalbende PP, Belsare GW. J Indian Chem. Soc. 89.