ISSN 2350-1030

International Journal of Recent Research in Physics and Chemical Sciences (IJRRPCS)
Vol. 4, Issue 1, pp: (1-7), Month: April 2017 – September 2017, Available at: www.paperpublications.org

Spectrophotometric Determination of Antibiotic Drug Penicillin in Pharmaceutical Samples Using 2, 6 Dichlorophenol Indophenol, N-Bromocaprolactam and N-Chlorosuccinimide

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Abstract: **Penicillin, an antibiotic drug is quantitatively determined by converting it into penicillamine in aqueous media.**
Penicillamine is β-β[1] dimethyl cysteine having sulphydryl functionality.
Methods developed have been applied by adopting simple procedures using oxidizing reagent as NBCL, NCS, 2,6 DCPIP have been considered for determining the drug by titrimetery.

Keywords: **Antibiotic, N-Bromocaprolactam, N-Chlorosuccinimide; 2, 6 Dichlorophenol indophenol Thiols, Titrimetery.**

1. INTRODUCTION

Antibiotics are chemical substances produced by various species of microorganisms (bacteria, fungi, actinomycetes) that suppress the growth of other microorganisms and may eventually destroy them. Antibiotics commonly include synthetic antibacterial agents having selective toxicity for bacterial cells. These include sulfonamides and quinolones that are not the products of microbes[1].

Antibiotics maybe classified[2] into four major classes –

(i) Drugs that prevent the synthesis of folic acid necessary for all cells e.g. : Sulphonamides.

(ii) Drugs that attack the peptide bond during transpeptidation, thus affecting the bacterial cell wall formation e.g. penicillin.

(iii) Drugs that inhibit the protein synthesis in bacteria by binding to a protein of unknown function in the ribosomes e.g. streptomycin.

(iv) Drugs that kill bacterial cell by penetrating the cell membrane and disrupting the electrostatic bonded area and ultimately leads to cell degradation e.g. Tyrocidin A.

2. REVIEW OF METHODS OF ANALYSING ANTIBIOTIC DRUGS

Unlike other groups of natural compounds i.e. alkaloids, glycosides, antibiotics have no group reactions for their quantitative identification. Thus identification has to be based on the chemical structure of that particular antibiotic. It is therefore the functional groups, which give distinguishing reactions especially forming coloured products which can be identified by a number of spectroscopic methods.

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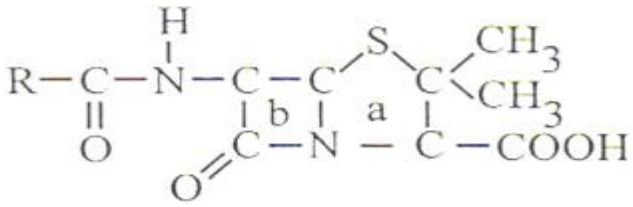
Recent chemical methods for the quantitative analysis of antibiotics preferentially includes photo colorimetry and spectro photometry[3]

Penicillin consists of a thiazolidine ring (A) connected to a beta – lactam ring (B) to which is attached a side chain (R).

The penicillin nucleus is the chief structural requirement for biological activity, metabolic transformation or chemical alteration of this portion of the molecule causes loss of anti bacterial activity. The side chain determines many of the antibacterial and pharmacological characteristics of a particular type of penicillin s

Structurally penicillin is

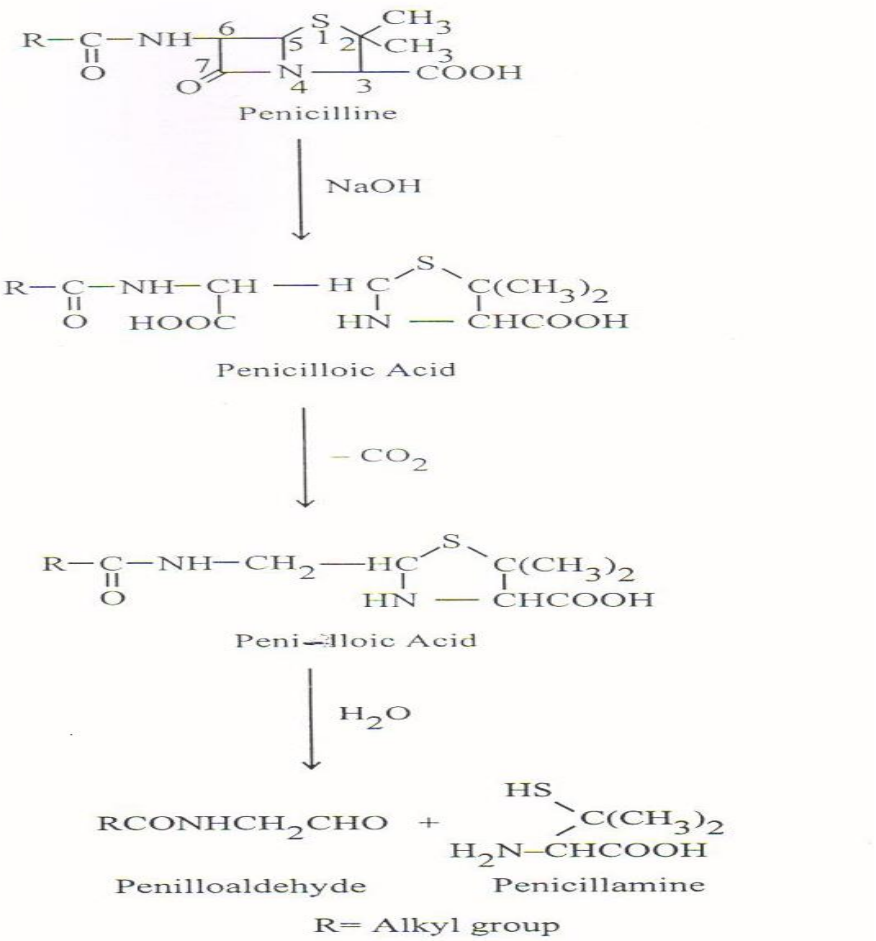


a = Thiazolidine

b = β - Lactam

Various methods have been proposed for the quantitative analysis of penicillin drug Iodimetric determination of penicillin involves its alkaline hydrolysis and then determining at a P^H near 4.5

Although penicillin cannot be easily oxidised by various oxidizing agents, but the products of its alkaline hydrolysis are easily oxidized one carbon atom is eliminated as carbon dioxide and two products are obtained in equimolar amounts one being amine- Penicillamine and other an aldehyde,- Penillo aldehyde. All the Penicillins give the same amine, but different aldehydes. The hydrolysis occurring is as-



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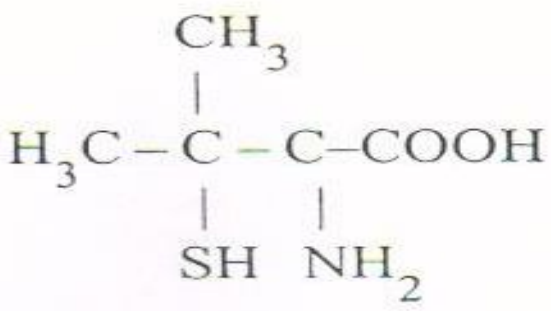
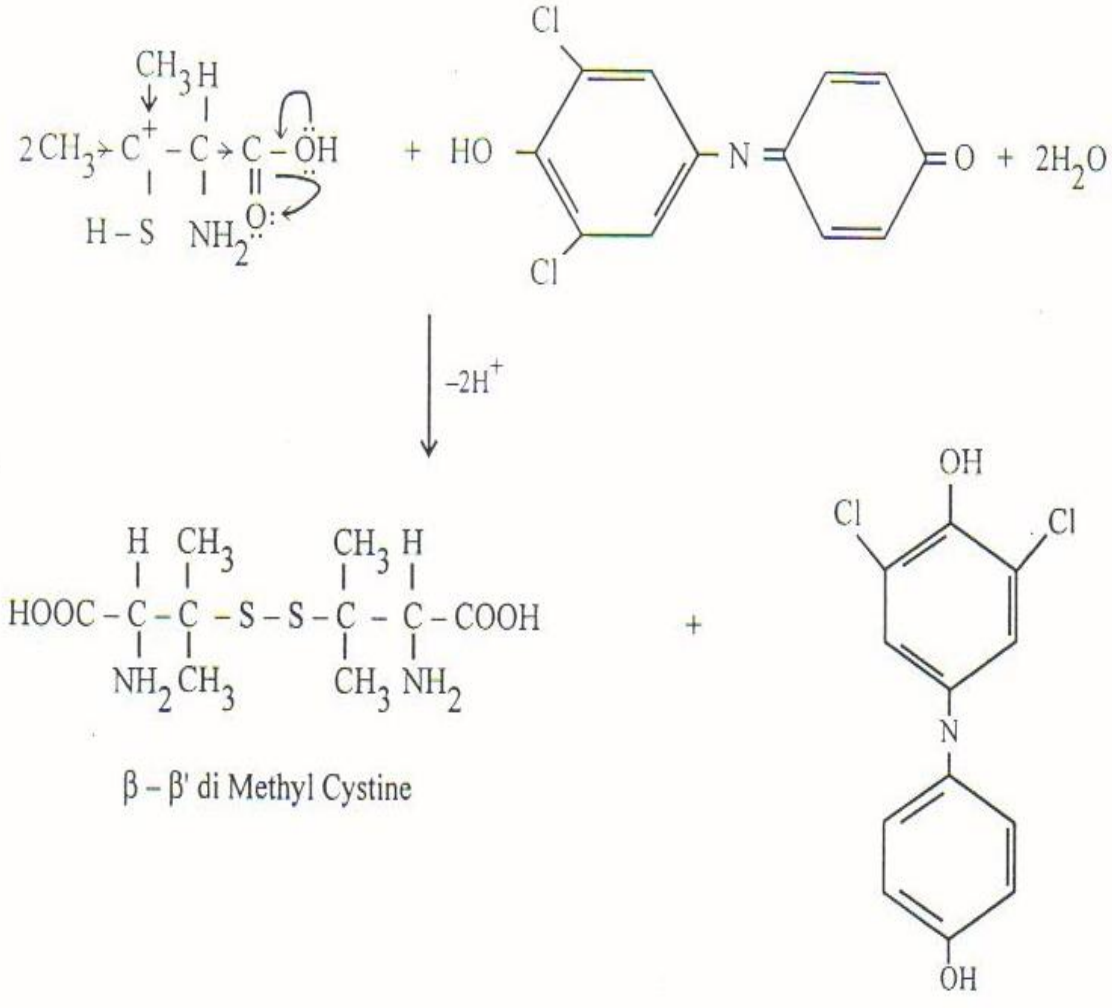
Thus we considered the hydrolysis product penicillamine as the basis for the quantitative determination and spectroscopic characterization of its products. Penicillamine is D- β,β[1] – dimethyl cysteine having the following structure –



The D – isomer is used clinically, although the L- isomer also forms chelation complexes. The present work proposes a simple method of determination of penicillamine sample by oxidizing its sulphydryl group into corresponding disulphide according to the reaction given below. The oxidising reagent undertaken is 2-6, dichlorophenol indophenol.



The method is simple, accurate and free from shortcomings of earlier reported methods Halogenating reactions like N-Bromo caprolactam and N-Chloro succinimide has also been considered for monitoring and for the analysis of penicillamine. In presence of potassium iodide and starch, there reagents yielded the stoichiometry 1:1 and the reactions taking place are as –

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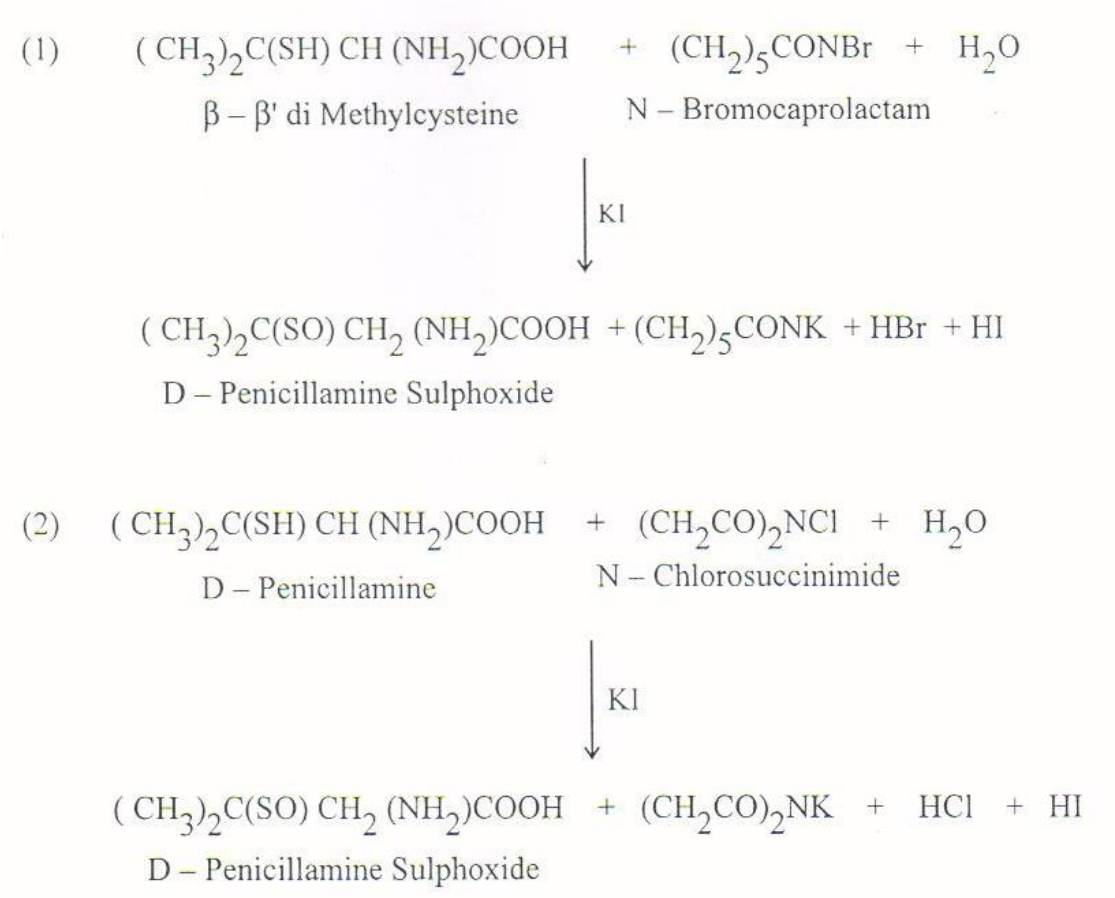

The stability of the system is apparently good and was monitored in aqueous media at pH = 7.0 and can have wider applicability for the determination of a variety of sulphydryl groups as such or in mixtures.

3. EXPERIMENTAL

INSTRUMENT:

- UV – visible spectrophotometer – UV -2000 chemito single beam spectro photometer.

REAGENTS:

- **Leuco – 2,6 Dichlorophenol Indophenol –**

0.005 M solution was prepared by standard reported methods and was standardized by freshly prepared ascorbic acid

- **N- Bromocaprolactam and N-Chloro succinimide –**

0.0025 M solution of hologenating reagents were prepared by standard methods and standardized iodometrically.

- **Penicillin solution –**

0.00635 M solution of Penicillin was prepared by dissolving 200 mg of procaine tablet in 100 ml of double distilled water. The solution was filtered and was then hydrolysed by standardised NaOH to get penicillamine. Penicillamine thus formed was titrated Iodimetrically. One mole of penicillamine reacts with three moles of standard Iodene. Thus all the reactions of penicillamine it self.

- **Penicillamine solution –**

0.005 M solution of penicillamine was prepared by dissolving 150 mgs of Artamin capsule in 500 ml of double distilled water the solution was filtered in order to get rid of impurities and was standardized iodo metrically. The method of standardization shows only 0.02% error during determination.

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4. PROCEDURES

(A)**SPECTROPHOTOMETRIC METHOD:**

An aliquot containing 1.0-3.0 ppm of the drug sample is pipetted to which 10ml of 2,6 DCPIP solution was added and the volume was made upto the mark by adding distilled water. The absorbance value was recorded at 610 nm.

The concentration of an unknown solution was calculated from already drawn Beer Lambert Law graph by drawing a perpendicular on the base line from the point where absorbance value intercepts the graph.

(B)**VOLUMETRIC METHOD:**

Aliquots containing 0.3 ppm to 3.0 ppm of drug sample was taken in a 150 ml Erlenmeyer flask. The contents were titratedagainst standardised solution at neutral pH by

N-Bromocaprolactam using methyl red indicator or

N-chlorosuccinimide using starch Iodide as indicator

5. RESULTS AND DISCUSSION

The results of spectrophotometric and volumetric determination of penicillamine with 2,6 DCPIP are summarized in Table I, II, III, IV.

The range of beer's law followed is 1.05 – 3.00 ppm with 2:1 stoichiometry and $\pm$ 0.48% accuracy, 4.472×10^{-4}, by observing absorbance values of 610 nm.

The β - β^1 – dimethyl cysteine penicillamine gets oxidised to its corresponding disulphide with 2-6 dichlorophenol indophenol whereas NBCL and NCS forms sulphoxides with 1:1 stoichiometry thus vitiating the non stoichiometric conditions reported in other functional moieties.

TABLE – I SPECTROPHOTOMETRIC DETERMINATION OF PENICILLAMINE WITH 2,6 DICHLOROPHENOL INDOPHENOL

S.No.	Range of Sample in ppm	Weight in ppm		% Error	Standard deviation	Coefficient of variance (%)
		Taken	Found			
1.	1.05 – 3.0	1.05	1.05	0.00	4.472×10^{-4}	0.0425
2.	1.200-3.0	1.200	1.200	0.00	4.472×10^{-4}	0.0399
3.	1.350-3.0	1.350	1.350	0.00	4.472×10^{-4}	0.0331
4.	1.500-3.0	1.500	1.500	0.00	4.472×10^{-4}	0.0298
5.	1.650-3.0	1.65	1.658	0.48	4.472×10^{-4}	0.0269
6.	1.80 – 3.0	1.80	1.800	0.00	4.472×10^{-4}	0.0248
7.	1.95-3.0	1.95	1.950	0.00	4.472×10^{-4}	0.0229
8.	2.10 – 3.0	2.10	2.10	0.00	4.472×10^{-4}	0.0213
9.	2.25 – 3.0	2.25	2.252	0.02	4.472×10^{-4}	0.0198
10.	2.40 – 3.0	2.40	2.406	0.25	4.472×10^{-4}	0.0185
11.	2.55 – 3.0	2.55	2.550	0.20	4.472×10^{-4}	0.0175
12.	2.70 – 3.0	2.70	2.704	0.15	4.472×10^{-4}	0.0165
13.	2.85 – 3.0	2.85	2.850	0.00	4.472×10^{-4}	0.0157
14.	3.0 – 3.0	3.0	3.010	0.33	4.472×10^{-4}	0.0148

* Data are an average of eight determinations.

Maximum range of sample determination – 3.0 ppm

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TABLE – II VOLUMETRIC DETERMINATION OF PENICILLAMINE WITH N-BROMOCAPROLACTAM

S.No.	Range of Sample in ppm	Weight in ppm		% Error	Standard deviation	Coefficient of variance (%)
		Taken	Found			
1.	0.3 – 3.0	0.3	0.288	- 4.16	0.10	34.72
2.	0.6 – 3.0	0.6	0.597	- 0.50	0.10	16.75
3.	0.9 – 3.0	0.9	0.90	0.00	0.10	11.11
4.	1.2 – 3.0	1.2	1.194	-0.50	0.102	8.54
5.	1.5 – 3.0	1.5	1.494	-0.40	0.102	6.83
6.	1.8 – 3.0	1.8	1.788	-0.67	0.102	5.70
7.	2.1 – 3.0	2.1	2.008	-0.57	0.10	4.79
8.	2.4 – 3.0	2.4	2.406	0.25	0.10	4.16
9.	2.7 – 3.0	2.7	2.682	-0.67	0.10	3.73
10.	3.0	3.0	2.976	-0.80	0.10	3.36

* Data are an average of eight determinations.

Maximum range of sample determination – 4.5 ppm

TABLE – III VOLUMETRIC DETERMINATION OF PENICILLAMINE WITH N-CHLOROSUCCINIMIDE

S.No.	Range of Sample in ppm	Weight in ppm		% Error	Standard deviation	Coefficient of variance (%)
		Taken	Found			
1.	0.3 – 3.0	0.3	0.3024	0.79	0.089	29.43
2.	0.6 – 3.0	0.6	0.6060	0.99	0.089	14.68
3.	0.9 – 3.0	0.9	0.9003	0.03	0.089	9.88
4.	1.2 – 3.0	1.2	1.201	0.08	0.089	7.10
5.	1.5 – 3.0	1.5	1.501	0.08	0.089	5.92
6.	1.8 – 3.0	1.8	1.801	0.05	0.089	4.94
7.	2.1 – 3.0	2.1	2.101	0.05	0.089	4.24
8.	2.4 – 3.0	2.4	2.402	0.08	0.089	3.70
9.	2.7 – 3.0	2.7	2.702	0.08	0.089	3.29
10.	3.0	3.0	3.001	0.04	0.089	2.96

* Data are an average of eight determinations.

Maximum range of sample determination – 3.9 ppm

TABLE – IV VOLUMETRIC DETERMINATION OF PENICILLAMINE WITH 2,6 DICHLOROPHENOL INDOPHENOL

S.No.	Range of Sample in ppm	Weight in ppm		% Error	Standard deviation	Coefficient of variance (%)
		Taken	Found			
1.	0.3 – 3.0	0.30	0.301	0.33	0.089	29.5
2.	0.6 – 3.0	0.60	0.602	0.33	0.089	14.7
3.	0.9 – 3.0	0.90	0.9004	0.04	0.089	9.88
4.	1.2 – 3.0	1.20	1.201	0.12	0.089	7.41
5.	1.5 – 3.0	1.50	1.50	0.00	0.089	5.93
6.	1.8 – 3.0	1.80	1.802	0.11	0.089	4.94
7.	2.1 – 3.0	2.10	2.106	0.28	0.089	4.23
8.	2.4 – 3.0	2.40	2.401	0.04	0.089	3.71
9.	2.7 – 3.0	2.70	2.70	0.00	0.089	3.29
10.	3.0	3.00	3.004	0.13	0.089	2.96

* Data are an average of eight determinations.

Maximum range of sample determination – 4.2 ppm

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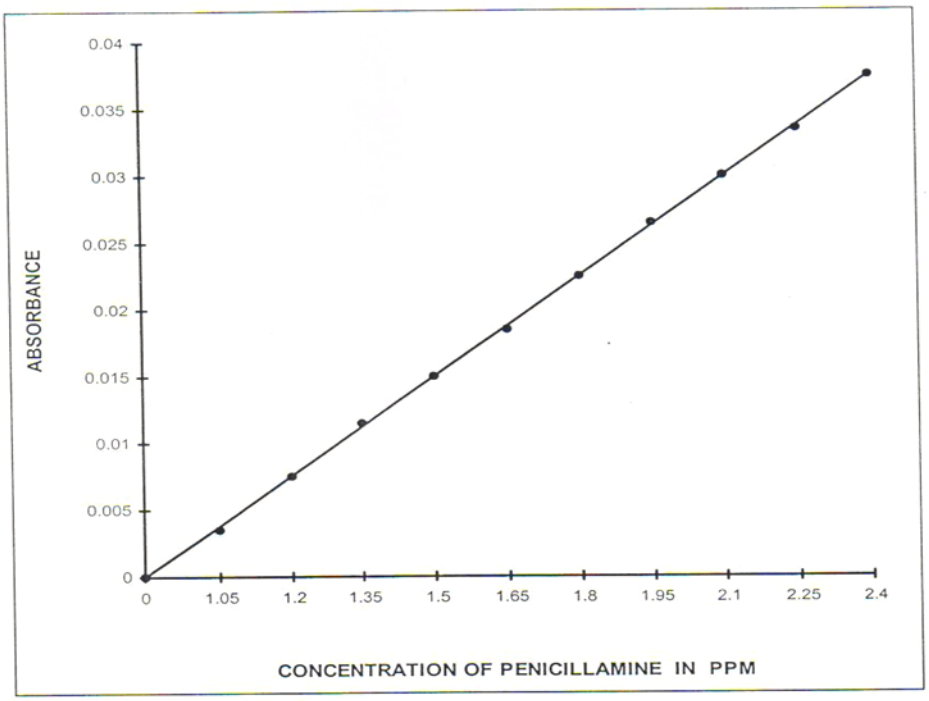

FIGURE 1 BEER LAMBERTS LAW GRAPH OF PENICILLAMINE USING 2,6 DCPIP

6. CONCLUSION

Penicillamine was quantitatively determined in aqueous media having sulphydryl functionality. Spectrophotometric and volumetric assay of drug determination using 2,6 DCPIP has been found to be precise with + 0.481 to + 0.33% accuracy. Volumetric assay of drug with hologenating reagents NBCL and NCS confirms 1:1 stoichiometry indicating the formation of sulphoxides.

Thus the methods are simple, rapid and precise and can be easily adopted for drug determination in pharmaceutical samples.

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