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Research Article

Indirect Novel Masterful eco-friendly maintainable Spectrophotometric Determination of Propranolol Hydrochloride in Environmental Wastewater Samples and Pharmaceutical Preparations

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Abstract

A simple, rapid, accurate and sensitive spectrophotometric method for determination of Propranolol Hydrochloride has been developed. The proposed method is based on the reaction between chloride ion and mercuric thiocyanate, formation of a colored complex by the reaction between released thiocyanate and ferric ions to form red soluble product with maximum absorption at 454 nm. Beer's law is obeyed over the concentration range of 2.5-35µg/ml, with molar absorptivity of 0.88444×104 l/mol.cm. The present method is considered to be simple because it does not need either heating or hydrolysis or solvent extraction steps. The method has been successfully applied for the determination of Propranolol Hydrochloride in environmental wastewater sample and in pure form, pharmaceutical preparations (Tablets).

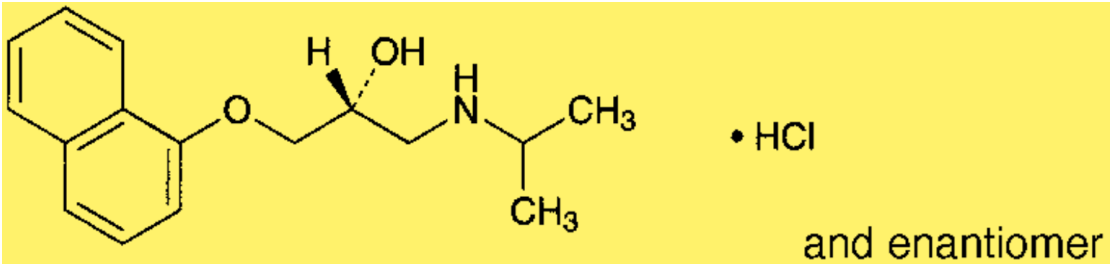
Key words: propranolol hydrochloride; mercuric thiocyanate; pharmaceutical preparation; environmental wastewater sample

Introduction:

Propranolol Hydrochloride **is** Beta-adrenoceptor antagonist., White or almost white powder, Soluble in water and in ethanol (96 per cent), with M.P about 164°.

Propranolol Hydrochloride (ATNL), chemically identified as 2-[4-[(2RS)-2-hydroxy-3-[(1-methylethyl) amino] proxyphenyl] acetamide (Figur1).

Propranolol Hydrochloride (ATEN), a beta-blocker is used to treat angina and high blood pressure. Hydrochlorothiazide (HCTZ) is a thiazide diuretic that increases the urine flow and prevents the retention of fluid in the body. It is used to treat high blood pressure. [1-4]



C16H21NO2.HCl: 295.80

(2RS)-1-(1-Methylethyl) amino-3-(naphthalen-1-yloxy) propan-2-ol mono hydrochloride

Figure 1: Propranolol Hydrochloride Chemical structure

Literature survey reveals that numerous methods have been published for quantitative analysis of Propranolol Hydrochloride alone and in combination with other drugs such as.Titrimetric method [5]. Spectrofluorometric methods [6] Spectrophotometric methods [7-12], RP-HPLC methods [13-15], Square-wave voltammetry [16] Flow-injection chemiluminescence analysis method [17], Capillary electrophoresis [18] continuous flow injection analysis via turbid metric method [17]., second derivative spectroscopy method [18], atomic absorption spectrophotometric method [19] and colorimetric [20]. In the view of the need in the industry for routine analysis of Propranolol Hydrochloride, attempts are being made to develop simple and accurate instrumental methods for quantitative estimation of Propranolol Hydrochloride. Thus, there is need for the development of new, simple, sensitive and accurate analytical method for the quantitative estimation of Propranolol Hydrochloride as an active pharmaceutical ingredient. The present work describes simple and accurate Spectrophotometric methods for the estimation of Propranolol Hydrochloride in bulk, dosage form and environmental wastewater samples: Application to content uniformity testing and in industrial wastewater samples. The method is based on reaction between chloride ion and mercuric thiocyanate, formation of a colored complex by the reaction between released thiocyanate and ferric ion.

Experimental

Apparatus

Shimadzu UV- 1700 pharmaspec (double beam) spectrophotometer with 1.0 cm quartz cells was used for absorption measurements.

Reagents

All chemical used were of analytical or pharmaceutical grade and Propranolol Hydrochloride standard material was provided from the state company of drug industries and medical appliance (NDI) Nineveh – Iraq.

Propranolol Hydrochloride standard solution :0.01% (100µg/ml)

This solution was prepared by dissolve 0.01 gm. of Propranolol Hydrochloride in 100 mL of distilled water in volumetric flask.

Ferric ammonium sulfate solution: 5%

5g of ferric ammonium sulfate [$FeNH_4(SO_4)_2.12H_2O$] was dissolved in 50 ml double distilled water and 20ml of concentrated nitric acid was added and diluted with double distilled water to 100ml.

Mercuric thiocyanate solution: 0.5%

0.5g of mercuric thiocyanate was dissolved and diluted to 100 ml with ethanol. Mixed and filtered through filter paper.

General procedure:

Different aliquots of standard Propranolol Hydrochloride solution equivalent 25-350 µg (0.25-3.5 mL) were transferred into a series of 25ml volumetric flasks, and 2mL of ferric ammonium sulfate solution were added and 2ml of saturated solution of mercuric thiocyanate were added to each flask and mixed well with occasional shaking. This was diluted to 25ml with double distilled water. and mixed well. Let stand for 10 min, the absorbance of each solution was measured at 454 nm against a reagent blank. Procedures for pharmaceutical preparations (Tablets40 mg):

To minimize a possible variation in the composition of the tables (containing 40mg of Propranolol Hydrochloride /tablet were provide from the state company of drug industries and medical appliance (NDI) Nineveh – Iraq. was transferred into 100mL volumetric flask and diluted up to the mark with distilled water, The determination of Propranolol Hydrochloride was treated as described above under general procedure. and the concentration was calculated by using the calibration curve of this method.

Procedure for industrial wastewater samples

To demonstrate the practical applicability of the proposed method, real industrial wastewater samples from the state company of drug industries and medical appliance (NDI) Mosul-Iraq were analyzed by spiked with the concentrations ranging from 5-25 µg /ml of Propranolol Hydrochloride and aliquot of this solution was treated as described above under general procedure and the concentration was calculated by using the calibration curve of this method.

Results and Discussion

The method depends upon the displacement of thiocyanate ion from mercury(I1) thiocyanate by chloride ion in the Propranolol hydrochloride.The released thiocyanate was found to react with Fe III at room temperature resulting in formation of red colored complex which absorbed at 454nm (Figure. 2).and the intensity of its color is proportional to the original chloride ion [21-23]

$$2Cl^- + Hg(SCN)_2 + 2Fe^{3+} \longrightarrow HgCl_2 + 2[Fe(SCN)]_2^+$$

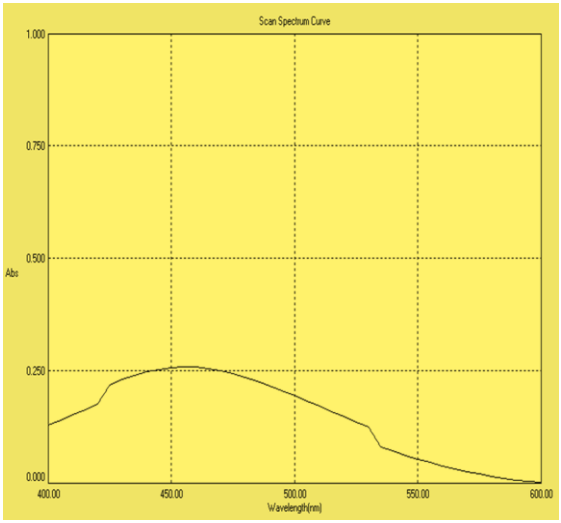


Figure 2: Absorption spectra of 10µg /ml of Propranolol Hydrochloride f

The various experimental affecting the development and stability of the reaction product was optimized by changing each variable in turn while keeping all other variables constant.

Effect of ferric ammonium sulfate solution

The amount of ferric ammonium sulfate solution (5%) for maximal color intensity was examined the maximum constant intensity was reached at 1 ml of reagent solution and remained constant up to 5ml. However, 2 ml of the reagent solution was selected for the subsequent work.

Effect of mercuric thiocyanate solution

The amount of **mercuric** thiocyanate solution (0.5%) for maximal color intensity was examined the maximum constant intensity was reached at 1 ml of reagent solution and remained constant up to 5ml. However, 2 ml of the reagent solution was selected for the subsequent work.

Effect of temperature and time:

The results obtained indicated that complete color formation occurred immediately and not effected by temperature therefore, room temperature was selected as suitable temperature. The absorbance remained constant for 6 hours at least, and 5 min was selected as a suitable time.

Effect of order of addition

To test the effect of order of the addition of the reagents on the absorbance of the product, different order were tested. The selected order was sample solution, ferric ammonium sulfate followed by mercuric thiocyanate solution which was gave high absorbance value.

Calibration graph

Employing the conditions described in the general procedure a linear calibration graph of Propranolol Hydrochloride which obeys Beer's law in the concentration range of 2.5-35 µg/ml (Figure.3). Linear regression equation: Y= 0.0299X- 0.001 (r=0.9995) Where Y is the absorbance and X is concentration in µg/ml. The apparent molar absorptivity was 0.88444×10^4 $l.mol^{-1}.cm^{-1}$ and sand ell's sensitivity were 0.03345 $\mu g.cm^{-2}$. The limit of detection and quantification were evaluated as[24] LOD = $3.3\frac{S_0}{b}$ LOQ = 3LOD

Where b is the stop and S_0 is the standard deviation of the regression line. The limit of detection was 0.456 µg $.ml^{-1}$ and the limit of quantification as the lowest standard concentration which could be determine with acceptable accuracy, and precision was 1.5 $\mu g.ml^{-1}$. The applied method can be used routinely for the estimation of pure drug salts through their chloride concentration.

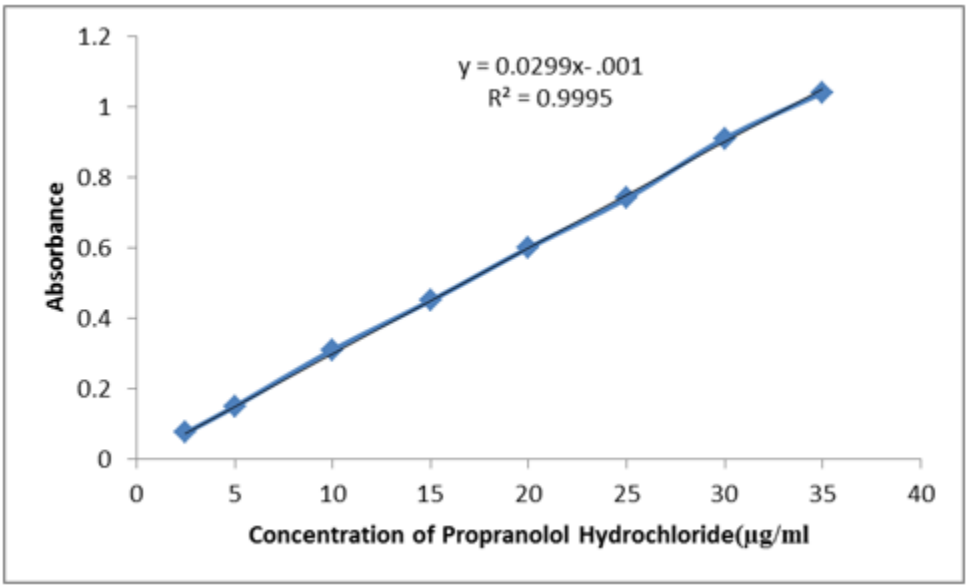


Figure 3: Calibration graph of Propranolol Hydrochloride

Accuracy and precision:

The accuracy and precision of the method were established by analyzing the pure drug solution at three different levels. The average recovery which is a measure of accuracy is 100 ±0.69% revealing high accuracy of the method. The relative standard deviation (RSD), which is an indicator of precision, is less than 1.5%, the result is compiled in (Table .1).

Parameters	Value
λ max (nm)	45460
Beer's law limits ($\mu g.ml^{-1}$)	2.5-35
Molar absorptivity ($L.mol^{-1}.cm^{-1}$)	0.88444×10^4
Limit of detection ($\mu g.ml^{-1}$)	0.456
Limit of quantification ($\mu g.ml^{-1}$)	1.5
Sandal's sensitivity ($\mu g.cm^{-2}$)	0.03345
Correlation coefficient (r)	0.9995
Regression equation (Y=C+ bX)	
Intercept (C)	0.001
Slope (b)	0.0299X
Recovery	100±0.69
Relative standard deviation (%)	< 1.5

Table 1: optical characteristics and statistical data for regression equation of the proposed method

Application of the proposed method

The proposed method was successfully applied to the analysis of Propranolol Hydrochloride in tables, Tablets and industrial waste water sample. The result of analysis for pharmaceutical formulations revels that there is close agreement between the results obtained by the proposed method and the label claim (Table. 2), And the results of water samples (Table.3) show that the recovery values obtained were close to 100%.

Pharmaceutical formulation supplied by NDI	Amount of Propranolol hydrochloride * Proposed method	Label% claim	%Recovery
	40.02mg	**40mg**	**100.05**

Table 2: Assay of Propranolol Hydrochloride in Tablets formulations

*Mean of ten determinations.

Water samples	Propranolol Hydrochloride (µg/ml) * Taken	Found	Recovery%
Industrial wastewater	**5.0**	**5.01**	**. 2100**
	10	**10.02**	**100. 2**
	25	**25 .03**	**100.12**

Table 3: Determination of Propranolol Hydrochloride in spiked industrial wastewater samples

*Mean of ten determinations.

Application of the method to content uniformity [25-3

The proposed method proved to be suitable for the content uniformity test, where a great number of assays on individual tablets are required. Data presented in Table [4] indicate that the proposed method can accurately and precisely quantitate Propranolol Hydrochloride in its commercially available tablets. The mean percentage (with RSD) of the labeled claim found in ten tablets was (0.518%) which fall within the content uniformity limits specified by the USP 33 [29].

Parameter	% of the label claim
Tablet NO. 1	100. 18
Tablet NO. 2	100. 21
Tablet NO. 3	99. 86
Tablet NO. 4	100.41
Tablet NO. 5	99.38
Tablet NO. 6	99. 55
Tablet NO. 7	99.72
Tablet NO. 8	100. 35
Tablet NO. 9	100.46
Tablet NO. 10	99.96
Mean ($\bar{x}$)	100.008
% RSD	0.518
Max. allowed unit [29]	±15%

Table 4: Content uniformity testing of Propranolol Hydrochloride tablets using the proposed method

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Conclusions

The applied method was simple, rapid, accurate, precise, sensitive and low economical cost. Furthermore, the proposed method doesn't require elaboration of procedures, which are usually associated with chromatographic methods. The proposed method could be applied successfully for determination of Propranolol Hydrochloride in environmental water samples, pure form as well as in dosage forms.

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