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Detection and spectrophotometric determination of paracetamol using NBS

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ABSTRACT

A simple and accurate spectrophotometric method has been developed by the authors for the detection and determination of paracetamol in pharmaceutical formulations in the form of tablets. Paracetamol, after dissolving in 4M sulphuric acid treated with 1% aqueous N-bromo succinamide (NBS) solution, exhibits a stable bluish violet colour. The coloured compound shows a $\lambda_{\max}$ at 560 nm. The method also recommended as a spot test for paracetamol. It is precise and found to be accurate for qualitative and quantitative determinations of paracetamol.

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KEYWORDS

Paracetamol;
N-bromosuccinamide;
Quantitative determination.

INTRODUCTION

In continuation to our earlier studies^[1,2], a new reagent for the spectrophotometric determination of paracetamol was developed. Paracetamol is a widely used analgesic and antipyretic pharmaceutical compound. It belongs to the class of drugs, known as aniline analgesics. It is commonly used for the relief of headaches, other minor aches, pains, inflammations and is a major ingredient in numerous cold and flu remedial combination drugs. While generally safe for use at a recommended dose, toxicity of paracetamol is the foremost cause of acute gastro intestinal problems. Many methods for its determination have been described, including chromatography, spectrophotometry and electro chemistry. In the standard method (IP and BP), paracetamol is determined titrimetrically with Ce (IV) in acidic medium, using ferroin as indicator. The titration is performed in cold conditions and the process of esti-

mation thus is time consuming. Hence a quick and accurate method is needed and developed by the authors.

During the course of experiments in search of specific colour reagents for paracetamol, it is noticed that solution of paracetamol gives a stable bluish- violet coloured product with 1% aqueous NBS. A survey of literature indicated that this specific colour reaction between paracetamol and NBS has not been reported previously. A spectrofluorimetric method was found in literature for the determination of paracetamol^[3]. In view of the toxicity of the over dosage of the drug and the quality tests, the above method of its estimation is recommended to be accurate, simple, and specific and will find a wide range of application in quick estimation.

EXPERIMENTAL

Reagents

Paracetamol pure form: This was prepared in our

laboratory by acetylation of p-amino phenol and recrystallized. The so prepared pure crystalline product of paracetamol has been standardized by the standard method^[4].

Paracetamol Tablets: Ten tablets of paracetamol of each pharmaceutical company under study are weighed and ground into a fine powder. From this, a sample of 500 mg of paracetamol is weighed, mixed with about 40 ml of 4M sulphuric acid and 50 ml of distilled water, heated at a temperature of 80°C for 90 min. After complete dissolution, the cooled solution is filtered through a Whatman No 40 filter paper, the solution is made up to the mark into a 100 ml volumetric flask and standardized^[4].

1% NBS solution: It is prepared by dissolving an adequate amount of substance Anala R MERCK sample, in double distilled water.

All the other reagents used are of Anala R grade only.

Apparatus

An ELICO- SI-177, Scanning Visible Spectrophotometer with recording unit and matched set of 1 cm. glass or quartz cuvettes is used for recording the spectra.

All the weighing measurements are made by a Shimadzu-AUX-220 model digital electronic balance.

All the pH-measurements are made by an ELICO-LI-127 pH-meter.

Recommended procedure for the determination of paracetamol with NBS

An aliquot of paracetamol solution (2.0 ml) is mixed with 0.4 ml of 1% aqueous NBS solution to a give a

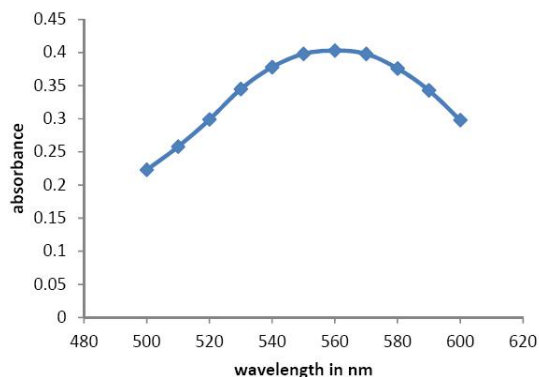


Figure 1 : Absorption spectrum of the bluishviolet coloured product obtained by reaction between paracetamol, NBS. The $\lambda_{\max}$ is 560 nm

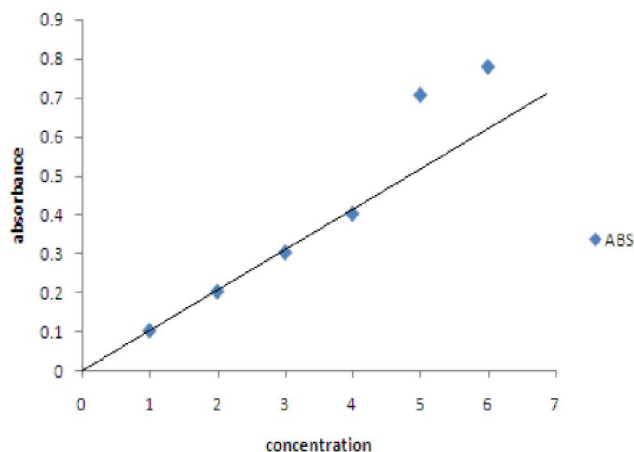


Figure 2 : Calibration plot for estimation of paracetamol. Beer's law obedience was 100-400 $\mu\text{g mL}^{-1}$ at $\lambda_{\max}$ 540nm

stable bluish- violet coloured product. The mixture is made up to 25 ml in a volumetric flask and the spectra are taken for an aliquot of the solution showed a $\lambda_{\max}$ at 560 nm (Figure 1).

For the determination of paracetamol, an aliquot volume of paracetamol is mixed with 0.4 ml of the reagent to a give a stable bluish- violet coloured product and the mixture is made up to the mark. The solution is taken in an optically matched cuvette of the ELICO spectrophotometer and the absorbances are measured at 560 nm. The absorbance is compared with the standard curve (Figure 2). Beer's law is found to be obeyed up to 400 $\mu\text{g mL}^{-1}$ of paracetamol (Figure 2).

RESULTS AND DISCUSSION

The specific colour reaction between paracetamol and NBS is studied in various concentration ranges of the reagent and in different media such as hydrochloric acid, acetic acid, sulphuric acid, phosphoric acid and in alkali. It was found that characteristic colour reaction between drug sample and NBS in the acid and alkaline medium stated above was not observed. It was found that the specific colour reaction is independent on the pH as well as medium. The concentration of the reagent also has an appreciable effect on the colour produced. Concentration below 1% and above 1% of the reagent is prepared and the absorbance measurements are made. The bluish-violet colour produced then is not found to be stable as performed with 1% reagent solution. The colour produced with the reagent with higher concentration than 1% is observed to fade rapidly again and is found to

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have an appreciable change in the absorbance measurements with respect to time. Hence the concentration of the reagent is prescribed at 1%. The volume of the reagent added to the sample was found to have an appreciable effect on the absorbance measurements and the stability of the colour produced. The colour reaction between the drug sample solution and the reagent was studied by varying the volume of the reagent. And it was found that the colour produced and the absorbance measurements are stable with the addition of 0.4ml of the reagent. And hence the volume of the reagent was fixed as 0.4ml. The λ_{max} for the bluish violet colour product is 560 nm (Figure 1), with molar absorptivity, $\epsilon = 160.6 \text{ M}^{-1} \text{ cm}^{-1}$ at 560 nm. There is no overlapping of the spectra of the bluish-violet coloured product of NBS and other species present in the solution. There are no interferences. Beer's law is found to be obeyed over the range of 100-400 $\mu\text{g ml}^{-1}$ of paracetamol.

Results of the determination of paracetamol in selected, available samples

S. No.	Drug proprietary name	Proposed Method	Standard Method
1	Paracetamol pure	100.12	100.15
2	Calapol (500mg)	98.96	99.65
3	Parakem (500mg)	99.02	99.58

From the above said data it is clear that the proposed method for the determination of paracetamol in pharmaceutical formulations is comparable and recommended due to the advantages mentioned earlier.

CONCLUSIONS

Paracetamol solution gives a stable bluish-violet coloured product with 1% aqueous solution of NBS. The λ_{max} for the bluish violet colour product was 560 nm, with molar absorptivity, $\epsilon = 160.6 \text{ M}^{-1} \text{ cm}^{-1}$ at 560 nm. Beer's law is found to be obeyed over the range of 100-400 $\mu\text{g ml}^{-1}$ of paracetamol. This determination of paracetamol is rapid and accurate.

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