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 succinimide (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-bromosuccinimide; 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 electrochemistry. 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 estimation 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
Pracetamol pure form: This was prepared in our
laboratory by acetylation of p-amino phenol and re- 
crystallized. 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 com-
plete 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 stan-
dardized\(^4\).

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- Sl-177, Scanning Visible Spectropho-
tometer 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 
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 
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 \(\lambda_{\text{max}}\) at 
560 nm (Figure 1).

For the determination of paracetamol, an aliquot 
volume of paracetamol is mixed with 0.4 ml of the re-
agent 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 stan-
dard curve (Figure 2). Beer’s law is found to be obeyed 
up to 400µg 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. Concent-
ration below 1% and above 1% of the reagent is 
preserved 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

![Figure 1](image1.png)

**Figure 1**: Absorption spectrum of the bluish-violet coloured 
product obtained by reaction between paracetamol, NBS. The 
\(\lambda_{\text{max}}\) is 560 nm

![Figure 2](image2.png)

**Figure 2**: Calibration plot for estimation of paracetamol. 
Beer’s law obedience was 100-400µg mL\(^{-1}\) at \(\lambda_{\text{max}}\) 540nm
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 $\lambda_{\text{max}}$ for the bluish violet colour product is 560 nm (Figure 1), with molar absorptivity, $\varepsilon = 160.6$ M$^{-1}$ 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 100-400µg ml$^{-1}$ of paracetamol.

Results of the determination of paracetamol in selected, available samples

<table>
<thead>
<tr>
<th>S. No.</th>
<th>Drug proprietary name</th>
<th>Proposed Method</th>
<th>Standard Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Paracetamol pure</td>
<td>100.12</td>
<td>100.15</td>
</tr>
<tr>
<td>2</td>
<td>Calpol (500mg)</td>
<td>98.96</td>
<td>99.65</td>
</tr>
<tr>
<td>3</td>
<td>Parakem (500mg)</td>
<td>99.02</td>
<td>99.58</td>
</tr>
</tbody>
</table>

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 $\lambda_{\text{max}}$ for the bluish violet colour product was 560 nm, with molar absorptivity, $\varepsilon = 160.6$ M$^{-1}$ cm$^{-1}$ at 560 nm. Beer’s law is found to be obeyed over the range of 100-400µg ml$^{-1}$ of paracetamol. This determination of paracetamol is rapid and accurate.

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REFERENCES