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QUANTITATIVE DETERMINATION OF HYOSCINE BUTYLBROMIDE (TABLET) IN COMMERCIAL DOSAGE FORM MARKETED AN USED IN MAIDUGURI METROPOLIS, NIGERIA

ALI AUDU SANI [1]*, FARIDA IBRAHIM [1] AND MOHAMMED ILYAS [2]

1. Department of Pharmaceutical Chemistry, Faculty of Pharmacy, University of Maiduguri, Nigeria
2. Department of Pharmaceutical and Medicinal Chemistry, Faculty of Pharmaceutical Sciences, Ahmadu Bello University Zaria , Nigeria

[*] Corresponding author e-*mail address*: aliaudusani@gmail.com

Abstract

The experiment involves analysis of nine brands of hyoscine butylbromide using ultra violet spectrophotometer in the range of (200-400nm) and high performance liquid chromatography (HPLC) in which the samples were dissolved in various solvents and their various absorbance, peak area at various wavelength were determined and compared with that of the standard, wavelength of maximum absorbance at 210nm for hyoscine butylbromide.

Percentage and milligramme content for each sample was determined so as to note if it was within the acceptable range of (92.5-107.5%) for hyoscine butylbromide. For those that passed the test or if it was below or above the range for samples that are substandard or highly concentrated.

The samples absorbance and peak area was used along side with the standard absorbance and peak area to calculate the percentage content of each sample.

It was observed that of the

Of the nine samples of hyoscine butylbromide tablet, only shreecopan with 93.6% passed using UV spectrophotometer while bixkopan with 99.8%, buscomac 101.6%, spanil 97.2% and unipan 104.7% passed using HPLC.

KEYWORDS: Hyoscine Butylbromide, UV, HPLC

1. Introduction

Giving the authorization of the flow of different generic drug products by the government into the market has made it paramount for the government to make sure that these drugs have the required specifications at all time. Recent reports indicate the availability of substandard and counterfeit drugs has reached a disturbing proportion in many low-income countries. (http://www.usp.org/azindex)

The introduction of generic drug products from multiple sources into health care delivery system of developing countries was aimed at improving the overall health care delivery system in such countries. However, this has been accompanied by a variety of problems of which the most critical is the widespread distribution of fake and substandard drug products. (Adebolagun *et al,* 2007).

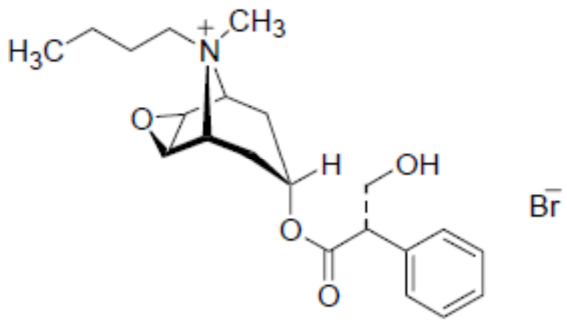
Butylscopolamine is used to treat pain and discomfort caused by abdominal cramps, menstrual cramps, or other spasmodic activity in the digestive system. It is also effective at preventing bladder spasms. It is not an analgesic in the normal sense, since it doesn't 'mask' or 'cover over' the pain, but rather works to prevent painful cramps and spasms from occurring in the first place. The attachment of the butyl-bromide moiety effectively prevents the movement of this drug across the blood–brain barrier, effectively minimizing undesirable CNS side-effects associated with scopolamine/hyoscine (Tripathi, 2003).

1.1 Buscopan

One of the earliest alkaloids to be isolated from plant sources is Scopolamine, which has been in use since it was isolated by the German scientist Albert Ladenburg in 1880. The use of various preparations from its plant-based form is learnt from the healing arts of some of the world's oldest cultures and perhaps even pre-historic times. In India Ancient Hindu physicians knew of the antispasmodic effects of a relative of the Duboisia shrub. In the search for a safe and effective treatment for abdominal pain and cramp, scientists based at Ingelheim prepared a semisynthetic derivative from the extract of elite Duboisia plants grown in greenhouses. This molecule,

Butylscopolamine, was free of the undesirable side-effects on the central nervous system, typical of Scopolamine, and the medication, on sale since 1952, was immediately recognized as a safe and effective antispasmodic. Today, Butylscopolamine, also known as Scopolamine butylbromide, butylhyoscine or hyoscine butylbromide, is the world's leading and most trusted treatment for pain and discomfort caused by muscle spasms and cramps. It cannot be considered an analgesic in the normal sense, in that it does not 'mask' or 'cover' the pain, but rather works to prevent the pain from occurring in the first place (Pharmacotherapy Update, 2009).

1.2 Chemical Structure:



(British pharmacopoeia, 2006) (BP 2008).

1.3 IUPAC name:(1S,3s,5R,6R,7S,8r)-6,7-epoxy-8-butyl-3-[(S)-tropoyloxy]tropanium bromide.

Empirical formula: $C_{21}H_{30}BrNO_4$

Molecular weight: 440.4

Solubility: freely soluble in water and in methylene chloride, sparingly soluble in anhydrous methanol.

Protein binding: Low

Half-life: 5 hours

Excretion: Renal (50%) and fecal

Description: a white or almost white, crystalline powder, odourless, or almost odourless.

(Indian pharmacopoeia 2007)

2. Materials and Method

2.1 Materials

Different brands of hyoscine butyl bromide were used for the study
Pure sample of the drugs were obtained from NAFDAC which served as standard
Writing and labeling materials,
Measuring cylinder, Beakers, 1000ml volumetric flask, 100ml volumetric flask, 50ml volumetric flask, Sonicator, Filter paper, Spatula, High performance liquid chromatography set up, UV Visible spectrophotometer (Beckman), Analytical weighing balance, Pestle and mortar, Distilled water, All reagents used were obtained from NAFDAC office, Maiduguri. Sani et. al (2012)a

2.2 Practical Method

The methods employed for the purpose of this study are the UV visible spectrophotometric and high performance liquid chromatographic methods Sani et. al (2012)b

2.3 Practical Procedure

The tablets were assayed spectrophotometrically using the following procedures

2.4 UV Procedure for Hyoscine Butyl Bromide

- The average weight of the tablets from each sample was determined by weighing ten(10) tablets and dividing the results gotten by ten to obtain the average weight
- From the value gotten the equivalent weight of 10mg of each brand was weighed accurately and transferred into 25ml volumetric flasks. All the nine samples were labelled using pen and masking tape.

- To each volumetric flask, 15ml of 0.001M HCl was poured and sonicated for few minutes to dissolve the drug molecule and made up to 25ml with the same solvent
- The mixture in each flask was mixed well and filtered through a filter paper into clean beakers.
- The UV spectrophotometer was put at zero by running a base line using 0.001M HCl solution as blank.
- The absorbance of each sample was determined at the wavelength of 210nm by putting small amount of the sample into a cuvette, and the cuvette was put back into the machine.
- The same procedure was repeated for the standard using 10mg of the powdered standard and the absorbance determined and from which the % content and mg content was determined as

$$\% \text{ content} = \frac{\text{Absorbance of sample} \times 100}{\text{Absorbance of standard}}$$

$$\text{Mg content} = \frac{\% \text{ content} \times \text{Manufactures claim}}{100}$$

Sani et al (2012)c

2.5 HPLC Procedure for Hyoscine Butyl Bromide

- The average weight of the tablets from each sample was determined by weighing ten(10) tablets and dividing the results gotten by ten to obtain the average weight
- From the value gotten the equivalent weight of each brand was weighed accurately and transferred into 25ml volumetric flasks. All the ten samples were labelled using pen and masking tape.
- To each volumetric flask, 15ml of 0.001M HCl was poured and sonicated for few minutes to dissolve the drug molecule and made up to 25ml with the same solvent
- The mixture in each flask was mixed well and filtered through a filter paper into clean beakers. The prepared samples were poured into the vials before injecting into the HPLC machine and the readings taken.
- The same procedure was repeated for the standard using 10mg of the powdered standard and the peak area determined and from which the % content and mg content was determined.

Chromatographic procedure was carried out using

a) A stainless steel column (25cm-4.6mm) packed with octadecylsilyl silica gel for chromatography(10µ) (lichrosorb 10µ c_8 column is suitable)
b) A solution of 2.0 of sodium dodecyl sulphate in a mixture of 370 of 0.001MHCL and 680ml of methanol as the mobile phase with a flow rate of 2ml/minute.
c) A detection wavelength of 210nm. Sani et. al (2012)d

3. Result

The data below shows the result of UV spectrophotometer which is used to calculate the percentage content and milligram content of the drugs. The results are shown below.

3.1 UV-Spectrophotometry for Hyoscine Butyl Bromide

Percentage and milligram content of different brands of Hyoscine Butyl Bromide using UV – Spectrophotometry

Unipan

$$\% \text{ content} = \frac{39.110}{55.568} \times 100 = 70.38\%$$

$$\text{mg content} = \frac{70.38}{100} \times 10 = 7.04\text{mg}$$

HBBR

$$\%\text{content} = \frac{29.933}{55.568} \times 100 = 54\%$$

$$\text{mg content} = \frac{54}{100} \times 10 = 5.4\text{mg}$$

Buscopan

$$\%\text{content} = \frac{82.607}{55.568} \times 100 = 149\%$$

$$\text{mg content} = \frac{149}{100} \times 10 = 14.9\text{mg}$$

Buscomac

%content = 45.700 x 100 = 82.24%
55.568
mg content = 82.24 x 10 = 8.2mg
100

Spanil
%content = 47.626 x 100 = 85.7%
55.568
mg content = 85.7 x 10 = 8.6mg
100

Shreecopan
%content = 53.019 x 100 = 93.61%
55.568
mg content = 93.61 x 10 = 9.4mg
100

Bixkopan
%content = 28.813 x 100 = 51.9%
55.568
mg content = 51.9 x 10 = 5.2mg
100

Boxcotab
%content = 48.634 x 100 = 88%
55.568
mg content = 88 x 10 = 8.8mg
100

Cinex
%content = 50.434 x 100 = 90.8%
55.568
mg content = 90.8 x 10 = 9.1mg
100

Table 1 UV absorbance of Hyoscine Butyl Bromide at a wavelength of 210 (E1%)

Sample	Absorbance (A)
Unipan	39.110
HBBR	29.933
Buscopan	82.607
Buscomax	45.700
Spanil	47.626
Shreecopan	52.019
Bixkopan	28.813
Boxcotab	48.634
Cinex	50.434

Table 2 Percentage content and milligramme content of different brands of Hyoscine Butyl Bromide using UV

Sample	%content	Mg content
Unipan	70.38	7.04
HBBR	54	5.4
Buscopan	149	14.9
Buscomac	82.24	8.2
Spanil	85.7	8.6
Shreecopan	93.61	9.4
Bixcopan	51.9	5.2
Boxcotab	88	8.8
Cinex	90.8	9.1

3.2 HPLC Result

The calculation below shows the result gotten from the HPLC method in analysis

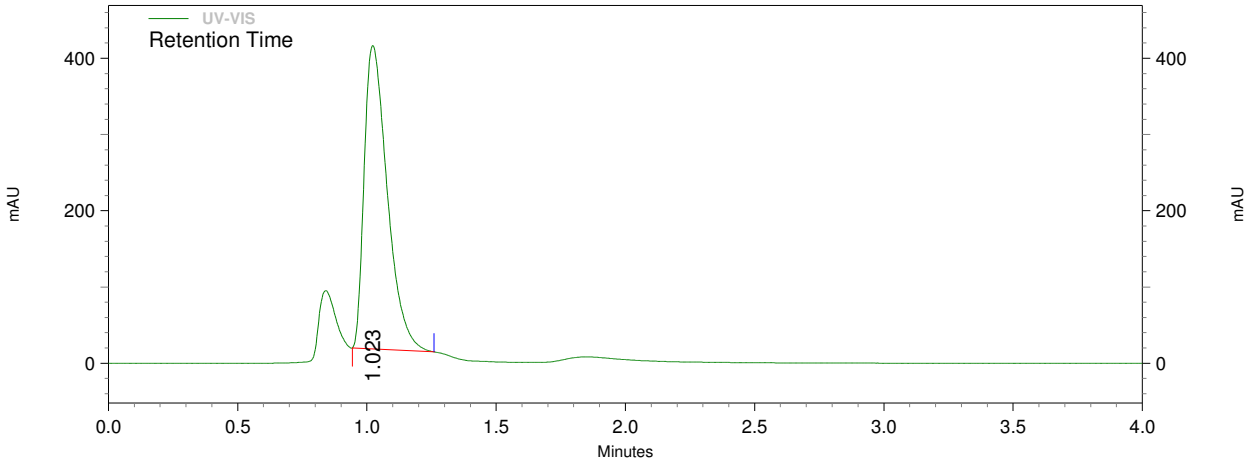
$$\% \text{ content} = \frac{\text{Peak area of sample}}{\text{Peak area of standard}} \times 100$$

$$\text{mg content} = \frac{\% \text{ content}}{100} \times \text{Standard claim}$$

Figure:1

Analyst: manager

Sample ID: COLOSPAN 070513 secondary standard Vial: 190 Injection Volume: 20



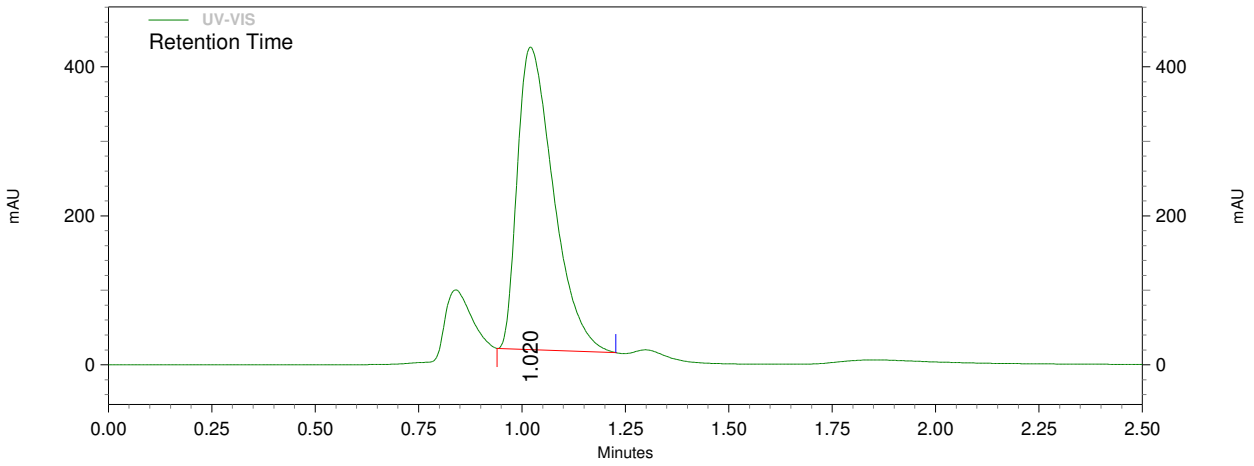
UV-VIS Results

Name	Retention Time	Area	Area Percent	Integration Codes
	1.023	9874861	100.000	MM
Totals		9874861	100.000	

Figure:2

Analyst: manager

Sample ID: BIXKOPAN 070513 **Vial: 120** **Injection Volume: 20**



UV-VIS Results

Name	Retention Time	Area	Area Percent	Integration Codes
	1.020	9859493	100.000	MM

Totals		9859493	100.000	

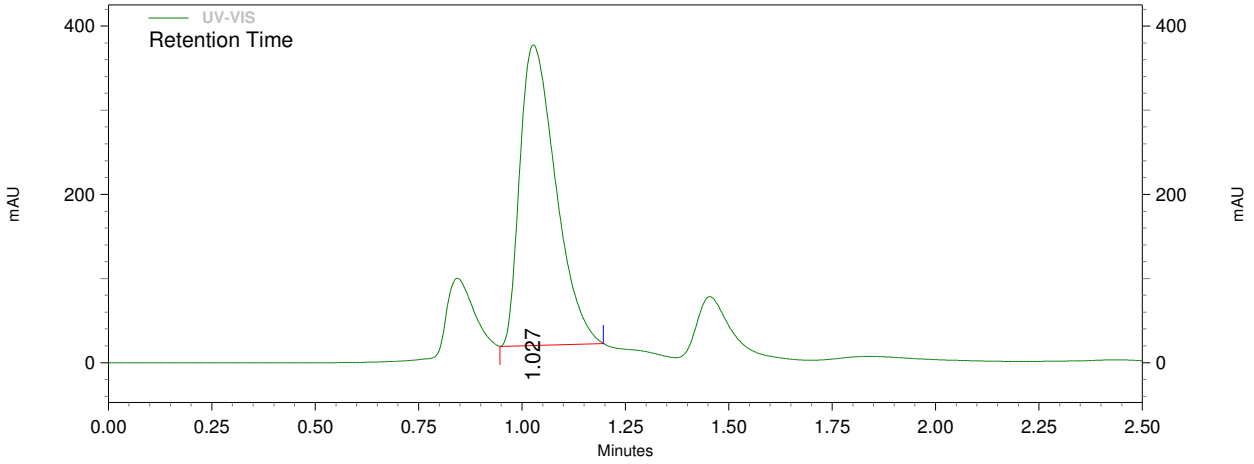
% content = 9859493 x 100 = 99.8%
9874861

Mg content = 99.8 x 10 = 9.98mg
100

Figure:3

Analyst: manager

Sample ID: BOXCOTAB 070513 **Vial: 110** **Injection Volume: 20**



UV-VIS Results

Name	Retention Time	Area	Area Percent	Integration Codes
	1.027	8595099	100.000	MM

Totals		8595099	100.000	

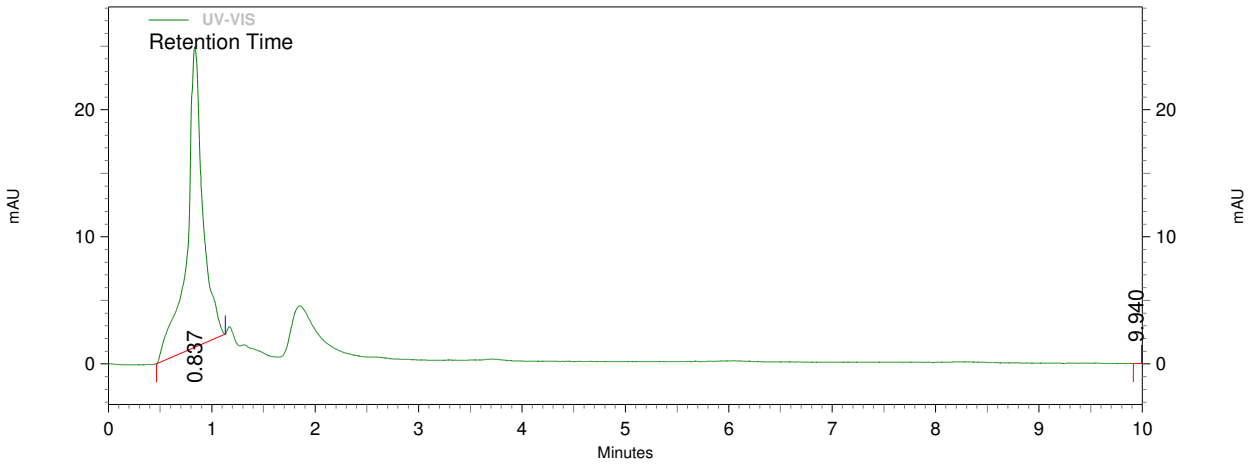
% content = 8595099 x 100 = 87.0%
9874861

Mg content = 87.0 x 10 = 8.7mg
100

Figure:4

Analyst: manager

Sample ID: BUSCOMAC 070513 **Vial: 200** **Injection Volume: 20**



UV-VIS Results

Name	Retention Time	Area	Area Percent	Integration Codes
	0.837	1003551	99.990	MM
	9.940	98	0.010	BE
Totals		1003649	100.000	

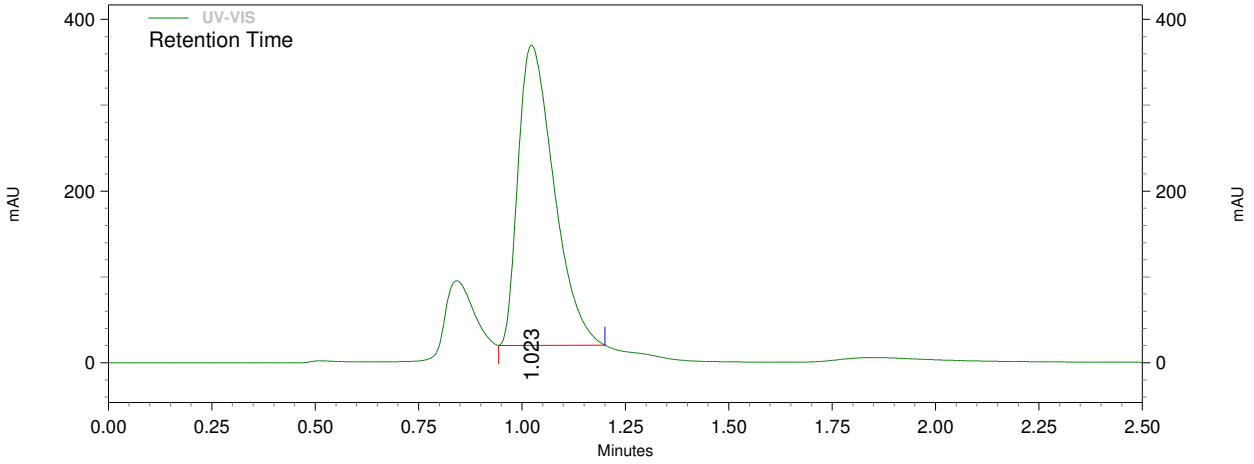
% content = 1003551 x 100 = 101.6%
9874861

Mg content = 101.6 x 10 = 10.2mg
100

Figure:5

Analyst: manager

Sample ID: BUSCOPAN 070513 **Vial: 170** **Injection Volume: 20**



UV-VIS Results

Name	Retention Time	Area	Area Percent	Integration Codes
	1.023	8413441	100.000	MM

Totals		8413441	100.000	

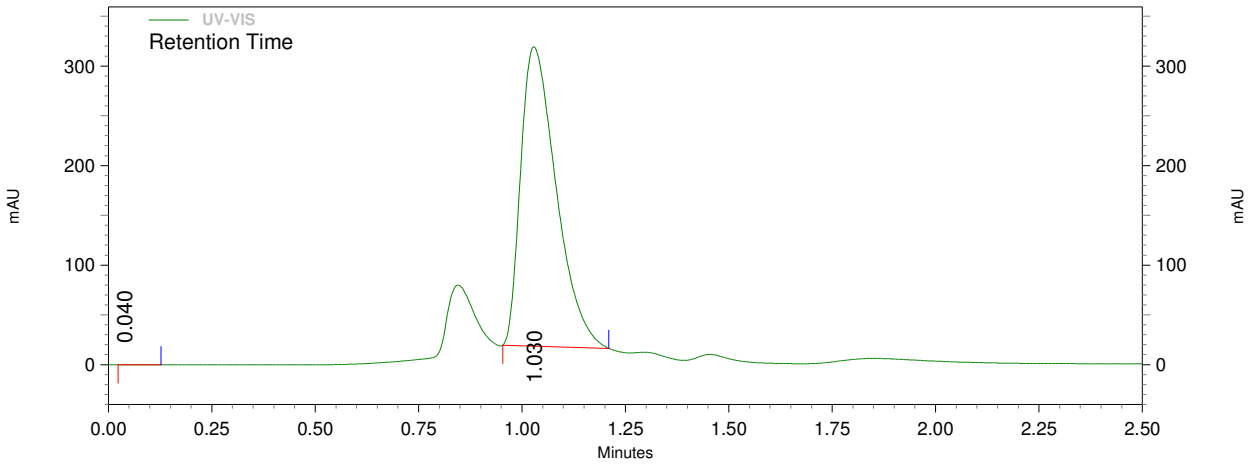
% content = 8413441 x 100 = 85.2%
9874861

Mg content = 85.2 x 10 = 8.5mg
100

Figure:6

Analyst: manager

Sample ID: HBBR 070513 **Vial: 140** **Injection Volume: 20**



UV-VIS Results

Name	Retention Time	Area	Area Percent	Integration Codes
	0.040	513	0.007	BI
	1.030	7175815	99.993	MM

Totals		7176328	100.000	

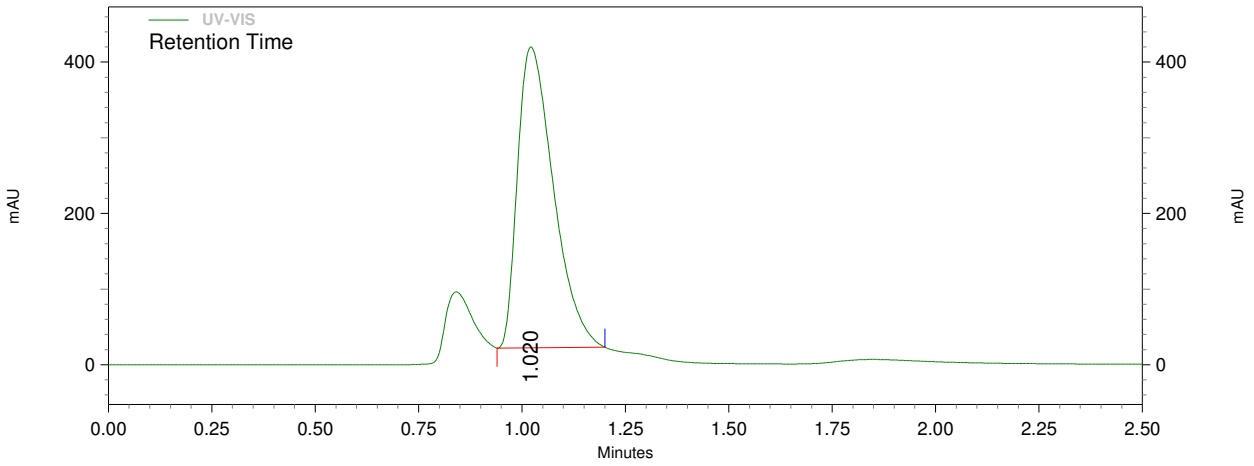
% content = 7175815 x 100 = 72.7%
9874861

Mg content = 72.7 x 10 = 7.3mg
100

Figure:7

Analyst: manager

Sample ID: SPANIL 070513 **Vial: 150** **Injection Volume: 20**



UV-VIS Results

Name	Retention Time	Area	Area Percent	Integration Codes
	1.020	9595509	100.000	MM

Totals		9595509	100.000	

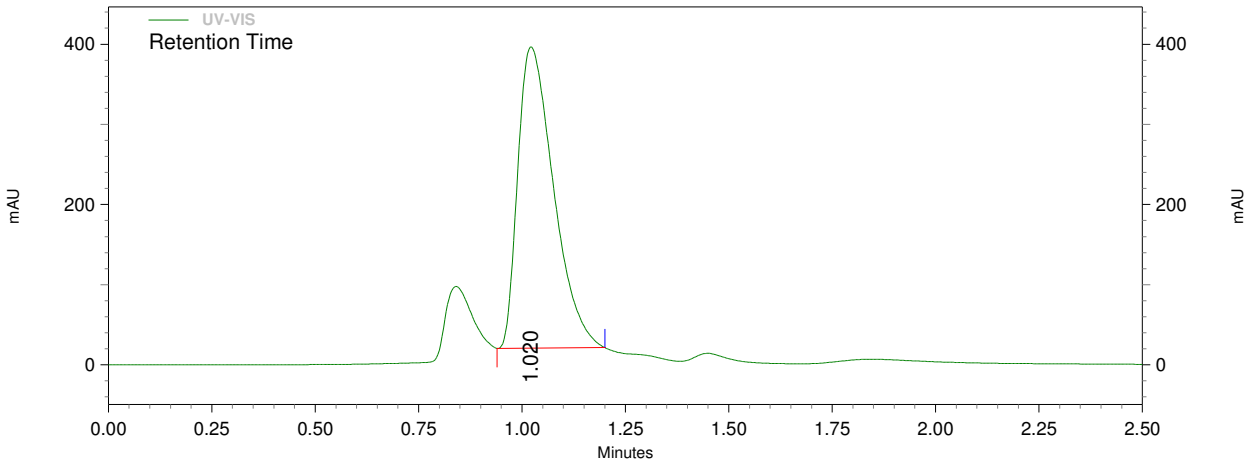
% content = 9595509 x 100 = 97.2%
9874861

Mg content = 97.2 x 10 = 9.7mg
100

Figure:8

Analyst: manager

Sample ID: SHREEPOPAN 070513 **Vial: 130** **Injection Volume: 20**



UV-VIS Results

Name	Retention Time	Area	Area Percent	Integration Codes
	1.020	9042098	100.000	MM

Totals		9042098	100.000	

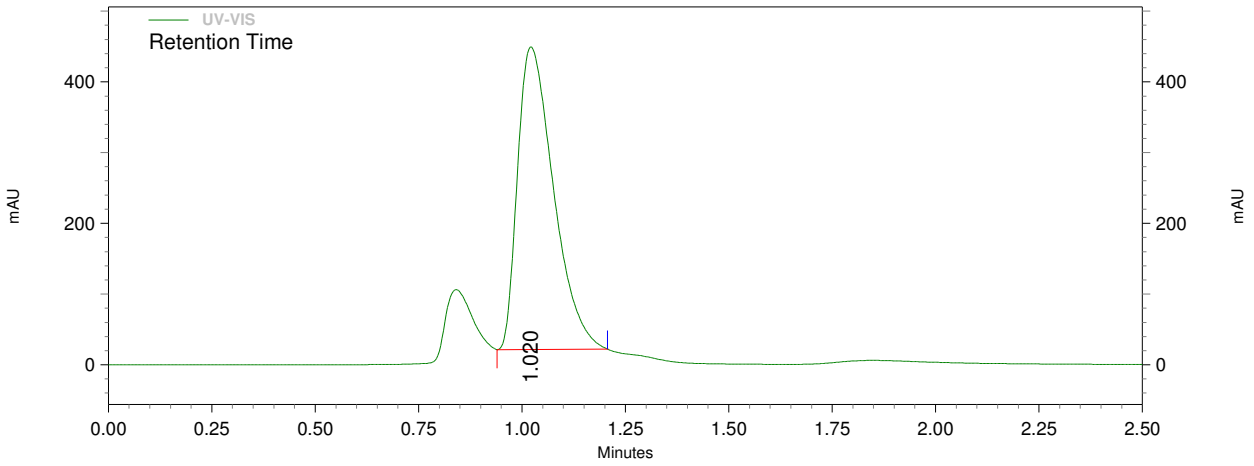
% content = 9042098 x 100 = 91.6%
98744861

Mg content = 91.6 x 10 = 9.2mg
100

Figure:9

Analyst: manager

Sample ID: UNIPAN 070513 Vial: 160 Injection Volume: 20



UV-VIS Results

Name	Retention Time	Area	Area Percent	Integration Codes
	1.020	10337846	100.000	MM

Totals		10337846	100.000	

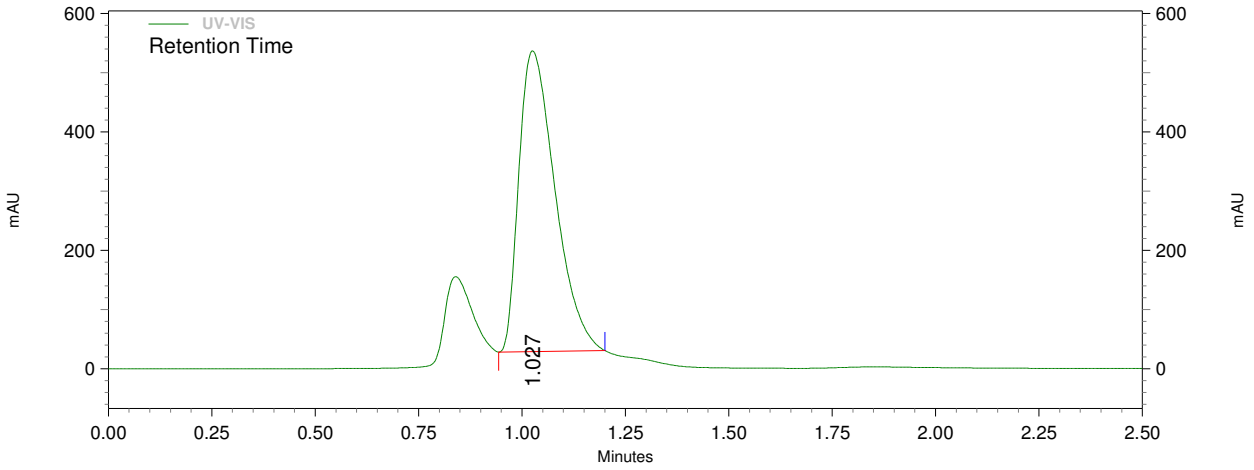
% content = 10337846 x 100 = 104.7%
98744861

Mg content = 104.7 x 10 = 10.5mg
100

Figure:10

Analyst: manager

Sample ID: CINEX 070513 **Vial: 180** **Injection Volume: 20**



UV-VIS Results

Name	Retention Time	Area	Area Percent	Integration Codes
	1.027	12365948	100.000	MM

Totals		12365948	100.000	

% content = 12365948 x 100 = 125.2%
9874861

Mg content = 125.2 x 10 = 12.5mg
100

Table 3 Percentage and milligramme content of different brands of Hyoscine Butyl Bromide using HPLC

Sample	% content	mg content
Unipan	104.7	10.5
HBBR	72.7	7.3
Buscopan	85.2	8.5
Buscomax	101.6	10.2
Spanil	97.2	9.7
Shreecopan	91.6	9.2
Bixkopan	99.8	10
Boxcotab	87.0	8.7
Cinex	125.2	12.5

4. Discussion

As stated by the British Pharmacopoeia, a hyoscine butylbromide should contain not less than 92.5% and not more than 107.5% of the stated amount of hyoscine butyl bromide. (BP 2008)

A standard hyoscine butyl bromide tablet using a secondary standard has an absorbance of 55.568 at a wavelength of 210nm.

From the result obtained using UV visible spectrophotometer shreecopan with percentage content of 93.6% fell within the range specified by the BP while unipan with percentage content of 70.38%, hyoscine butyl bromide 54%, buscopan 149%, buscomac 82.24%, spanil 85.7%, bixkopan 51.9%, boxcotab 88%, cinex 90.8% fell either above or below the range set by the BP and are said to have failed the test.

In the high performance liquid chromatography (HPLC) analysis carried out on the same sample using the same limit set by BP,

For hyoscine butylbromide tablet, bixkopan with percentage content of 99.8%, buscomac 101.6%, spanil 97.2% and unipan 104.7% are said to have passed the test because they are within the set limit while Boxcotab 87.0%, Buscopan 85.2%, Hyoscine butyl bromide bromide 72.7%, Shreecopan 91.6% and Cinex 125.2% are said to have failed the test because they were either below or above the set limit of the BP.

5. CONCLUSION

Following the BP specification, it can be concluded that For hyoscine butyl bromide using UV only shreecopan passed the test while unipan, hyoscine butyl bromide, buscomac, spanil, bixkopan, boxcotab and cinex failed the test but for HPLC Bixkopan, buscomac, spanil and unipan passed the test while Boxcotab, Buscopan, hyoscine butyl bromide, Shreecopan and Cinex failed the test.

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