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QUANTITATIVE ANALYSIS OF SOME BRANDS OF AMLODIPINE MARKETED IN MAIDUGURI METROPOLIS, USING ULTRA VIOLET SPECTROPHOTOMETRY AND HIGH PERFORMANCE LIQUID CHROMATOGRAPHIC (HPLC) METHODS

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Abstract

The quantitative analysis of different brands of Amlodipine tablets was carried out (using HPLC and U.V Spectrophotometer) to determine if the drugs are of required standards. The results obtained from analysis of the various drugs were compared with that of the standard. The percentage content for each sample was calculated using the absorbance and peak areas of the samples and that of the standard to see if they are within specified limit as stated by the official books. Amlodipine has a range of 97%-102% according to B.P 2008, From the result obtained using UV – Spectrophotometer,A (98.3%), B (100%),E (101.8%),F (101.22%),I (100.8%) are all within the B.P Specified limit while D (88.65%),G (65.57%) and C are said to be below the B.P specified limit. From the result obtained using HPLC analysis G (101.5% and A (100%) are said to be within the specified limit but E (441.8%), I (90.4%),H (92.2%) and F (95.4%) are all below the B.P specified limit while B (104.3%),C (126.2%) and D (201.8%) are said to be above the B.P specified limit. using UV- spectrophotometry shows that 5 samples of the Amlodipine passed and 4 samples failed while for HPLC, only 2 samples passed

KEYWORDS: Amlodipine, UV, HPLC

1. Introduction

The science of drug analysis is an extensively active one in terms of research and development of new, more reliable or more sensitive methods that have become of great importance in the analysis and quality control of drug and drug products at every stage of their life. A whole arsenal of chemical, physicochemical and automated analytical techniques is now available for determining the identity, purity, content, stability, safety and efficacy of drugs and their formulations. Thus in the development, formulation, marketing and pharmacokinetic assessment of a drug, the analyte is involved in several diverse areas including the following:

1) Determination of identity and purity of starting materials used in the manufacturing of the drug substance.
2) Test for identity and purity of the drug.
3) Isolation and identification of trace impurities of the drug.
4) Determination of degradation rates and degradation products of the drug.
5) Identification of the drug in a formulated product and its qualitative analysis.
6) Determination of any degradation within the formulated product and possible isolation of substance for toxicity test.
7) Evaluation of content uniformity for low dose formulations (Ajibola, 2000).

This deals with the study of the nature or the quality of the compound or mixture. It also involves the identification of constituent radicals present in the organic mixture (Ahmad and Ali, 2009)

A Clinical study was carried out in 2010 on The Effect of Amlodipine Alone and in Combination with Atenolol on Bowel Habit in Patients with Hypertension and showed the following result A total of 100 patients who attended the hypertensive clinic in Government Medical College and Hospital, Chandigarh, were included in the study after the screening. Fifty patients were on amlodipine alone and 50 patients were on combination of amlodipine and atenolol. The number of patients in the amlodipine group with SBM/wk less than 3 was 8 whereas in combination group (amlodipine + atenolol) it was 2 (P = .045, Fisher's exact test, significant). The relative risk (RR) of developing constipation was 4.00 with 95% CI 0.8930 to 17.917 in amlodipine alone group. However, there was no significant difference in SBM/wk in both the groups before and after treatment (P > .05).

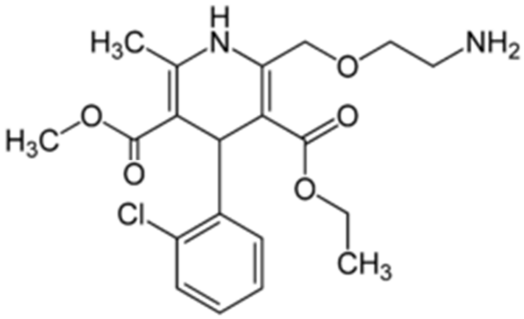
The majority of the patients in both the groups were on amlodipine 10 mg daily dose (28(56%) patients in amlodipine group and 29(58%) patients in combination group). The duration of treatment with amlodipine was also comparable in both the groups (16.75 ± 2.5 months versus 14.025 ± 2.58 months). The number of patients with blood in stool was 5 in amlodipine group while there was none in the combination group (P = .028, significant). Straining/hard stool was seen in 10 patients in the amlodipine group and 3 patients in the combination group (P = .035, significant). The risk (RR = 3.33 with 95% CI 0.9748 to11.399) of developing hard stool. So from the present observation, it can be said that amlodipine alone associated with increased incidence of constipation (RR = 4.00) and hard stool (RR = 3.333) and when atenolol was combined, the incidence of constipation and hard stool was less (P < .05). SBM: spontaneous bowel movement. Results are expressed as mean ± SD and absolute number (Lekha and Chander 2011).

8) In another study a work was carried out on the Comparative Effects of Amlodipine and Cilnidipine on Sympathetic Nervous Modulation in Patients With Hypertension and showed the following in their Results: In patients with continuous amlodipine treatment, systolic and diastolic blood pressures (SBP, DBP) and heart rate (HR) remained unchanged. LF/HF and HF/TP ratios also remained unchanged (LF/HF 1.77±1.05 vs. 1.83±1.22, HF/TP 0.419±0.122 vs. 0.402±0.116). Plasma norepinephrine levels were comparable (370±88 pg/ml vs. 491±137 pg/ml). In patients switched to cilnidipine, SBP, DBP and HR were similar before and after switching. Interestingly, LF/HF ratio decreased significantly (p = 0.012) from 2.37±1.56 to 1.89±1.42, and HF/TP ratio increased significantly (p = 0.049) from 0.366±0.132 to 0.417±0.156, despite the comparable HR. Plasma norepinephrine concentrations decreased significantly (p = 0.009) from 359±65 pg/ml to 282±72 pg/ml (Ikai A. *et al*., 2010).

1.1 Amlodipine

Amlodipine (as besylate, mesylate or maleate) is a long-acting calcium channel blocker dihydropyridine (DHP) class used as an antihypertensive and in the treatment of angina pectoris. Like other calcium channel blockers, amlodipine acts by relaxing the smooth muscle in the arterial wall, decreasing total peripheral resistance thereby reducing blood pressure; in angina, Amlodipine increases blood flow to the heart muscle (although DHP-class calcium channel blockers are more selective for arteries than the muscular tissue of the heart (myocardium), as the cardiac calcium channels are not of the dihydropyridine-type).

Amlodipine



(Wikipedia)

Chemical data

Formula	$C_{20}H_{25}ClN_2O_5$
Mol. mass	408.879 g/mol

(Bertram G. Katzung, 2001)

1.2 Medical uses

Amlodipine is used in the management of hypertension and coronary artery disease and stroke (Wang, J.G., 2009)

2. Materials and Method

Nine (9) brands of Amlodipine 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, 2011)1

2.1 Practical Method

The methods employed for the purpose of this study are the UV visible spectrophotometer and high performance liquid chromatographic methods. (Sani et al, 2011)2

2.2 UV Procedure for Amlodipine (BP, 2008)

The tablets were assayed spectrophotometrically using the following procedures

- The average weight of the tablets from each sample was determined by weighing ten(10) tablets and dividing the results gotten by nine to obtain the average weight
- From the value gotten the equivalent weight of each brand was weighed accurately and transfered into 100ml volumetric flasks. All the nine samples were labelled using pen and masking tape.
- To each volumetric flask, 60ml of 0.01M sodium dihydrogen phosphate buffer and acetonitrile (63:37) was poured and sonicated for few minutes to dissolve the drug molecule.
- 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 diluents as blank.
- The absorbance of each sample was determined at the peak wavelength 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 5mg of the powdered standard and the absorbance determined and from which the % content and mg content was determined as:

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

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

2.3 HPLC Procedure for Amlodipine

Mobile phase

Prepare a solution of 0.01M solution dihydrogen phosphate buffer and acetonitrile (63:37 v/v) in 100ml volumetric flask. Adjust the PH to 3.5

Chromatographic system

The liquid chromatograph is equipped with a 237nm detector and a 150cm x 4.6mm column that contains packing L1. The flow rate is about 1.5ml/minute

Procedure:

- The average weight of the tablets from each sample was determined by weighing ten(10) tablets and dividing the results gotten by nine to obtain the average weight
- From the value gotten the equivalent weight of each brand was weighed accurately and transferred into 100ml volumetric flasks. All the nine samples were labelled using pen and masking tape.
- To each volumetric flask, 60ml of the mobile phase was poured and sonicated for few minutes to dissolve the drug molecule.
- The mixture in each flask was mixed well and filtered through a filter paper into clean beakers.
- The same procedure was repeated for the standard using 5mg of the powdered standard
- Separately inject equal volume of the standard preparation and the assay preparation into the chromatograph, record the chromatograms, and measure the responses for the major peaks. Calculate the quantity in mg of Amlodipine. (Sani et al, 2012)

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3. Results

Table 1 Brand Name of some Amlodipine

Brand Name	Brand Code
Amlodipine	A
Cadrex	B
Orkal	C
Juvasc	D
Amlong	E
Tenox	F
Amnivasc	G
Miravase	H
Acedipine	I

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

The results are as follows:

3.1. Amlodipine

A

%content = 321.00 x 100 = 98.3%
326.29

` Mg content = 98.3 x 5 = 4.9mg
100

B

%content = 326.29 x 100 = 100%
326.29

Mg content = 100 x 5 = 5mg
100

C

%content = 311.29 x 100 = 95.4%
326.29

Mg content = 95.4 x 5 = 4.77mg
100

D

%content = 289.26 x 100 = 88.65%
326.29

Mg content = 88.65 x 5 = 4.4mg
100

E

%content = 332.241 x 100 = 101.8%
326.29

Mg content = 101.8 x 5 =5.09mg
100

F

$$\%\text{content} = \frac{330.29}{326.29} \times 100 = 101.22\%$$

$$\text{Mg content} = \frac{101.22}{100} \times 5 = 5.06\text{mg}$$

G

$$\%\text{content} = \frac{213.94}{326.29} \times 100 = 65.57\%$$

$$\text{Mg content} = \frac{65.57}{100} \times 5 = 3.28\text{mg}$$

H

$$\%\text{content} = \frac{405.83}{326.29} \times 100 = 124.38\%$$

$$\text{Mg content} = \frac{124.38}{100} \times 5 = 6.2\text{mg}$$

I

$$\%\text{content} = \frac{328.92}{326.29} \times 100 = 100.8\%$$

$$\text{Mg content} = \frac{100.8}{100} \times 5 = 5.04\text{mg}$$

Table 2: UV absorbance for amlodipine at a wavelenght of 237nm

Sample	Absorbance (A)
A	321.00
B	326.29
C	311.29
D	289.26
E	332.241
F	330.29
G	213.94
H	405.83
I	328.92

Table 3: Percentage content and mg content of different brands of Amlodipine using UV

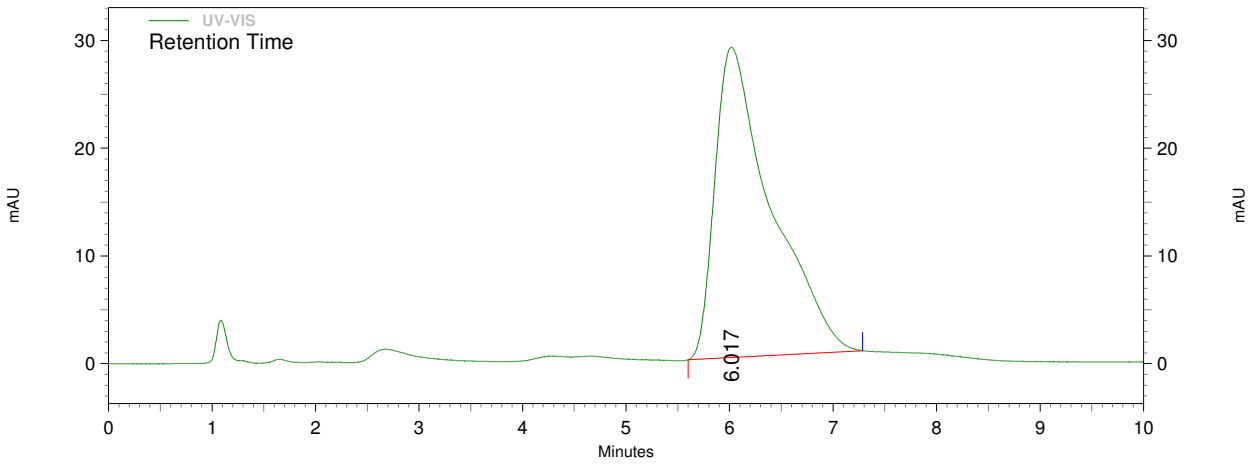
Sample	%content	mg content
A	98.3	4.9
B	100	5
C	95.4	4.77
D	88.65	4.4
E	101.8	5.09
F	101.22	5.06
G	65.57	3.28
H	124.3	6.2
I	100.8	5.04

3.2 HPLC for Amlodipine

Figure 1:

Analyst: manager

Sample ID: G 030513MEOH **Vial: 119** **Injection Volume: 20**



UV-VIS Results

Name	Retention Time	Area	Area Percent	Integration Codes
	6.017	4332964	100.000	MM

Totals		4332964	100.000	

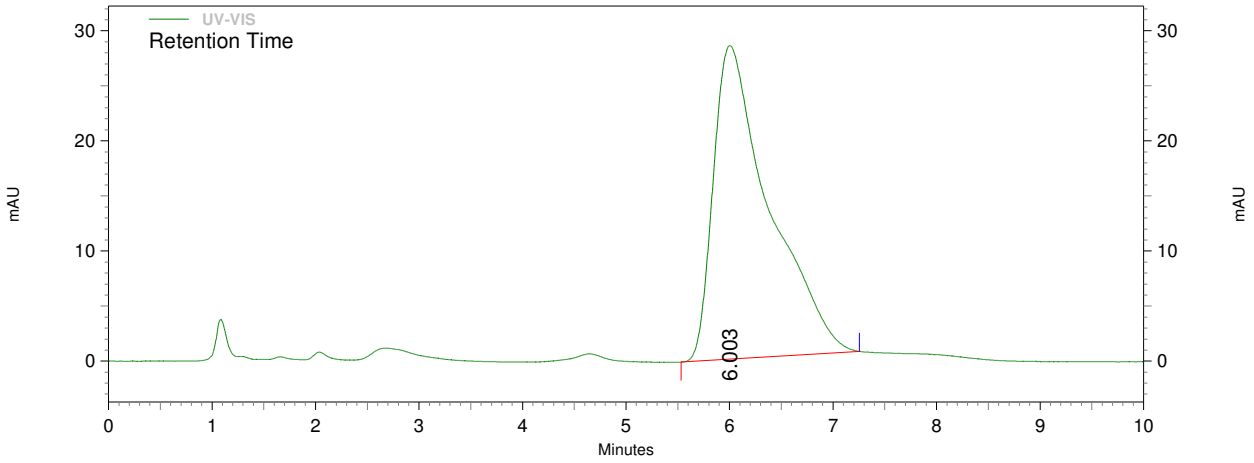
% content = 4332964 x 100 = 101.5%
4268878

Mg content = 101.5 x 5 = 5.1mg
100

Figure 2:

Analyst: manager

Sample ID: A 030513MEOHRPT **Vial: 139** **Injection Volume: 20**



UV-VIS Results

Name	Retention Time	Area	Area Percent	Integration Codes
	6.003	4268879	100.000	MM

Totals		4268879	100.000	

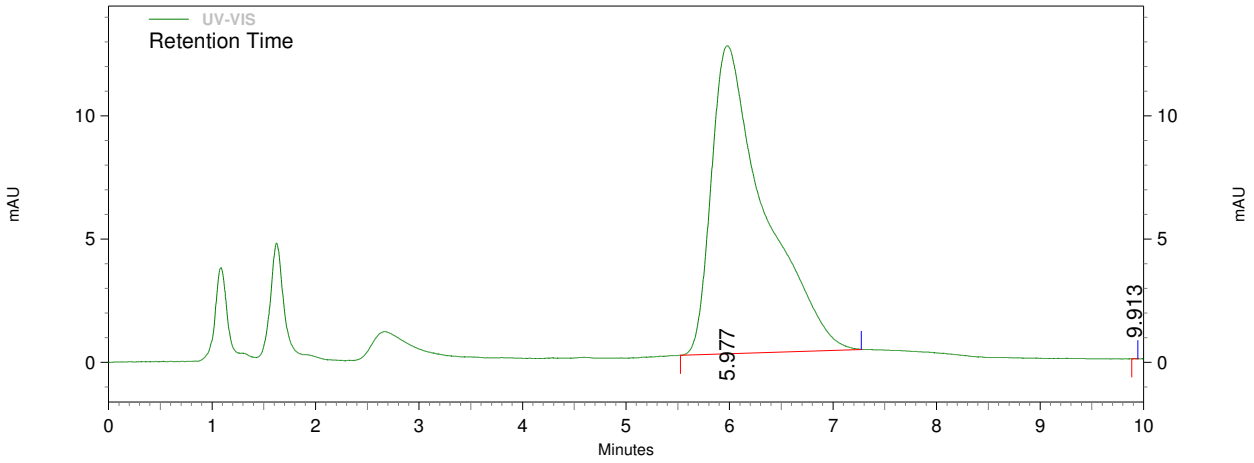
% content = 4268879 x 100 = 100%
4268878

Mg content = 100 x 5 = 5mg
100

Figure 3:

Analyst: manager

Sample ID: E 020513MEOH **Vial: 149** **Injection Volume: 20**



UV-VIS Results

Name	Retention Time	Area	Area Percent	Integration Codes
	5.977	1782769	99.997	MM
	9.913	61	0.003	IB
Totals		1782830	100.000	

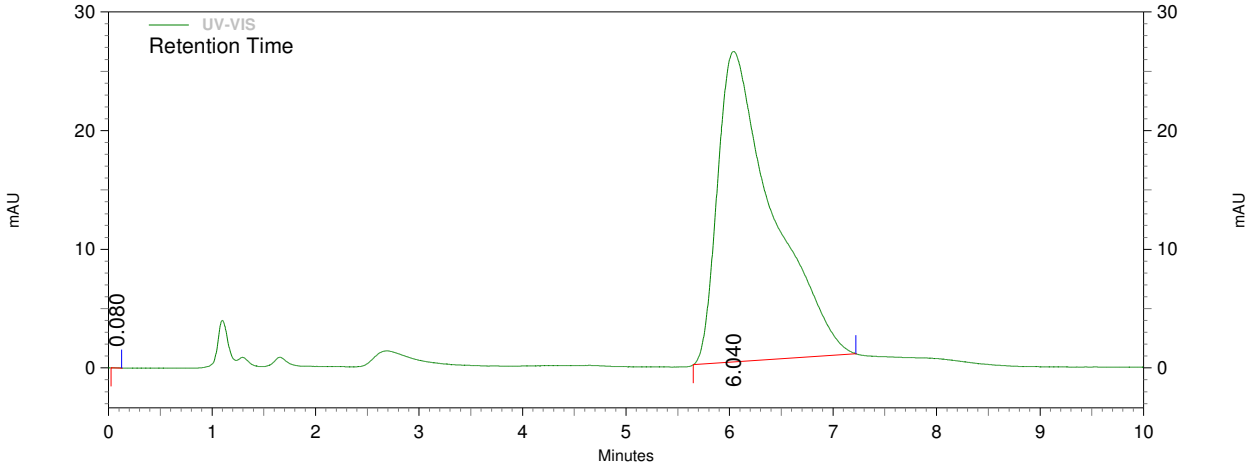
% content = 1782769 x 100 = 41.8%
4268878

Mg content = 41.8 x 5 = 2.1mg
100

Figure 4:

Analyst: manager

Sample ID: I 030513MEOH **Vial: 109** **Injection Volume: 20**



UV-VIS Results

Name	Retention Time	Area	Area Percent	Integration Codes
	0.080	271	0.007	BI
	6.040	3859801	99.993	MM
Totals		3860072	100.000	

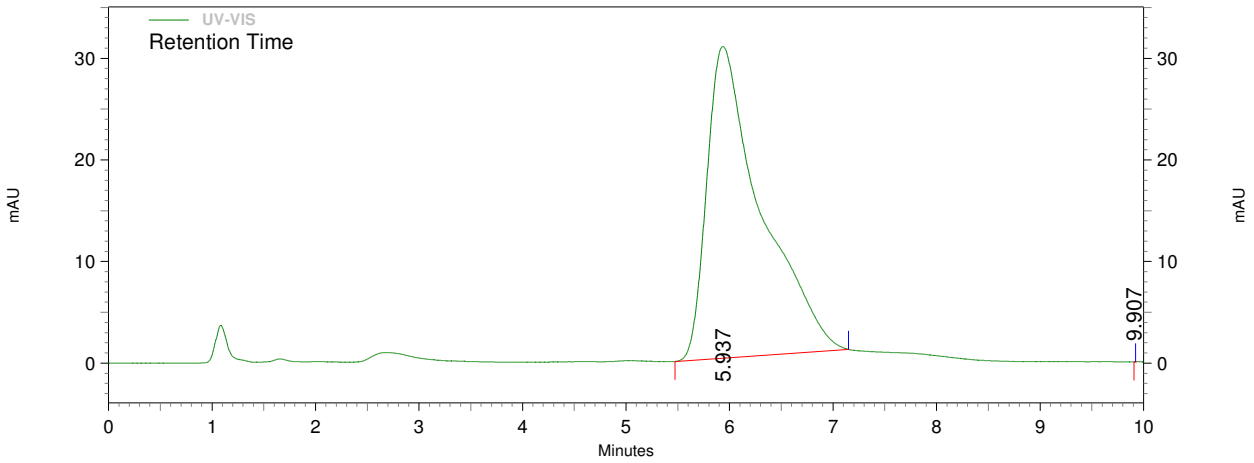
% content = 3859801 x 100 = 90.4%
4268878

Mg content = 90.4 x 5 = 4.5mg
100

Figure 5:

Analyst: manager

Sample ID: B 020513MEOH **Vial: 189** **Injection Volume: 20**



UV-VIS Results

Name	Retention Time	Area	Area Percent	Integration Codes
	5.937	4451622	100.000	MM
	9.907	2	0.000	BE
Totals		4451624	100.000	

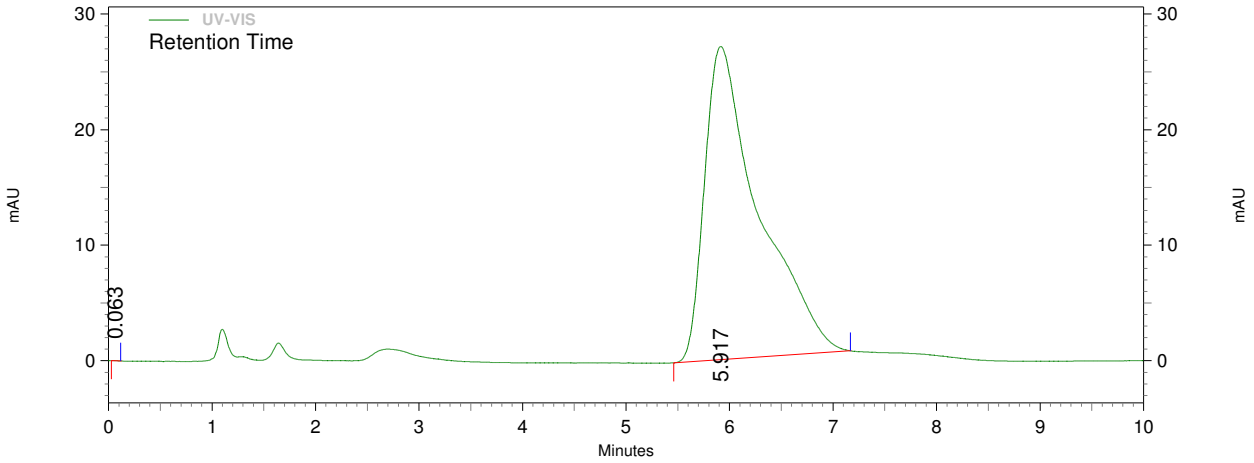
% content = $\frac{4451622}{4268878}$ x 100 = 104.3%

Mg content = $\frac{104.3}{100}$ x 5 = 5.2mg

Figure 6:

Analyst: manager

Sample ID: H 020513MEOH **Vial: 199** **Injection Volume: 20**



UV-VIS Results

Name	Retention Time	Area	Area Percent	Integration Codes
	0.063	211	0.005	BI
	5.917	3937713	99.995	MM
Totals		3937924	100.000	

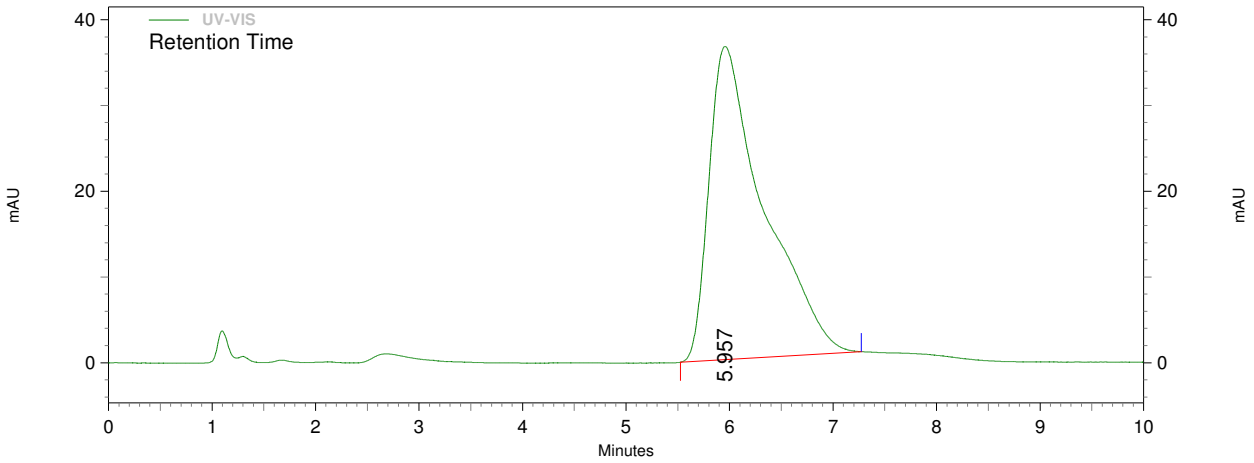
% content = 3937713 x 100 = 92.2%
4268878

Mg content = 92.2 x 5 = 4.6mg
100

Figure 7:

Analyst: manager

Sample ID: C 020513MEOH **Vial: 159** **Injection Volume: 20**



UV-VIS Results

Name	Retention Time	Area	Area Percent	Integration Codes
	5.957	5387262	100.000	MM

Totals		5387262	100.000	

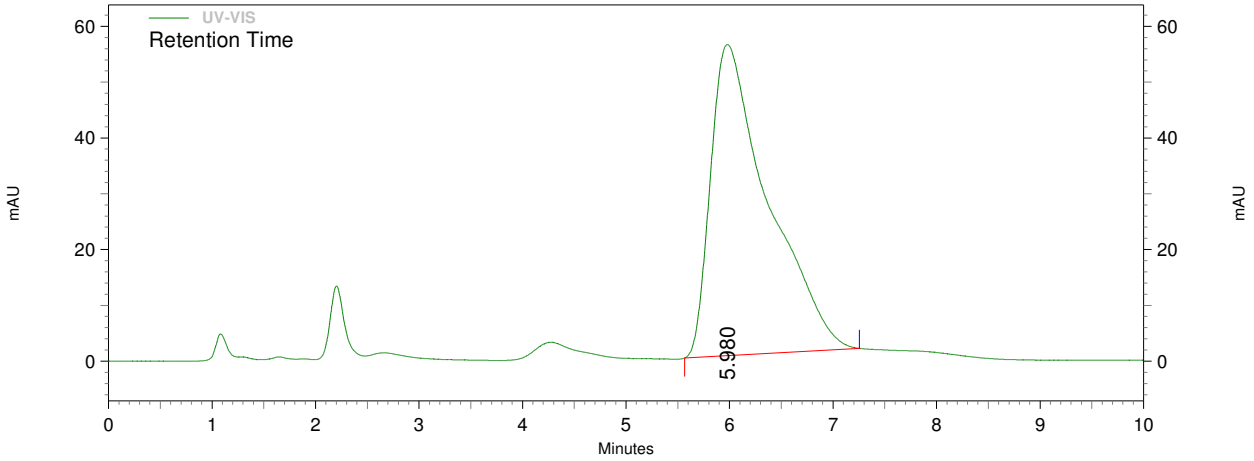
% content = 5387262 x 100 = 126.2%
4268878

Mg content = 126.2 x 5 = 6.3mg
100

Figure 8:

Analyst: manager

Sample ID: D 030513MEOH **Vial: 129** **Injection Volume: 20**



UV-VIS Results

Name	Retention Time	Area	Area Percent	Integration Codes
	5.980	8612299	100.000	MM

Totals		8612299	100.000	

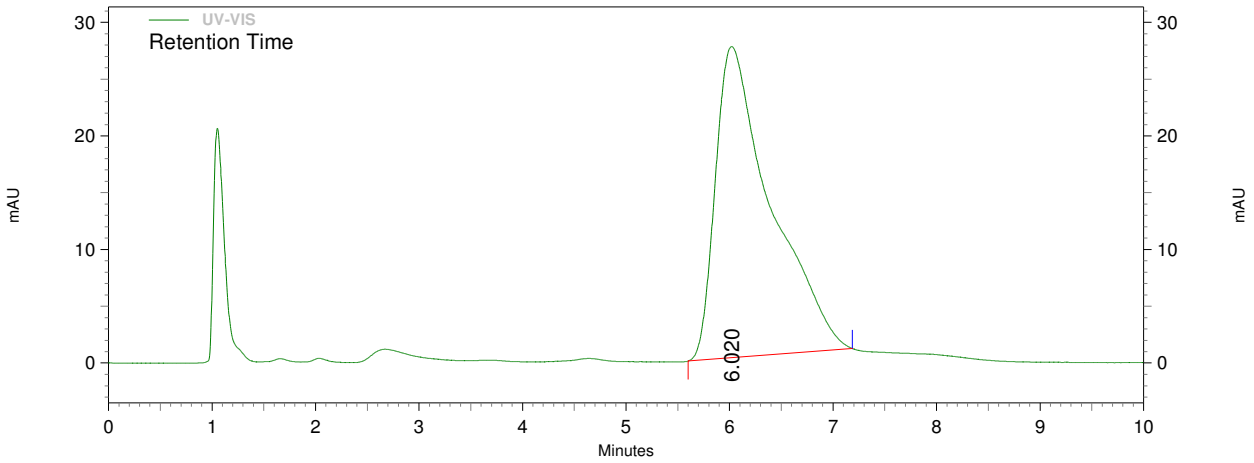
% content = 8612299 x 100 = 201.8%
4268878

Mg content = 201.8 x 5 = 10.1mg
100

Figure 9:

Analyst: manager

Sample ID: F 030513MEOH **Vial: 99** **Injection Volume: 20**



UV-VIS Results

Name	Retention Time	Area	Area Percent	Integration Codes
	6.020	4071402	100.000	MM
Totals		4071402	100.000	

% content = 4071402 x 100 = 95.4%
4268878

Mg content = 95.4 x 5 = 4.8mg
100

Table 4: Percentage content and mg content of different brands of Amlodipine using HPLC

SAMPLE	%CONTENT	Mg CONTENT
G	101.5%	5.1mg
E	41.8%	2.1mg
I	90.4%	4.5mg
B	104.3%	5.2mg
H	92.2%	4.6mg
C	126.2%	6.3mg
D	201.8%	10.1mg
F	95.4%	4.8mg
A	100%	5mg

4. DISCUSSION

According to British Pharmacopoeia, Amlodipine tablet should contain not less than 97% and not more than 102.0% of A. The standard Amlodipine has an absorbance 326.29 at the wavelength of 237nm.

From the result obtained using UV – Spectrophotometer,A (98.3%), B (100%),E (101.8%),F (101.22%),I (100.8%) are all within the B.P Specified limit while D (88.65%),G (65.57%) and C are said to be below the B.P specified limit.

From the result obtained using HPLC analysis G (101.5% and A (100%) are said to be within the specified limit but E (441.8%), I (90.4%),H (92.2%) and F (95.4%) are all below the B.P specified limit while B (104.3%),C (126.2%) and D (201.8%) are said to be above the B.P specified limit.

5. CONCLUSION

For Amlodipine following BP specification, it can be concluded that 5 brands of the drug passed and 4 brands of the drug failed while for HPLC analysis only 2 brands passed.

6. RECOMMENDATION

Pharmaceutical analysis should always be carried out on Drugs by regulatory bodies to ensure that drugs that are being marketed are of the required standard to eradicate the problems of fake and counterfeit drugs and also guilty companies should be queried or closed down as appropriate.

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