

# Comparative Purity Study of UV Spectrophotometric and Fourier-Transform Infrared Spectroscopic (FTIR) Techniques for the Determination of Ciprofloxacin Hydrochloride Tablets

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**Keywords:** Ciprofloxacin; FTIR; UV Spectroscopy; USP; BP

**Abbreviations:** hydrochloric acid (HCl); United State Pharmacopoeia (USP); British Pharmacopoeia (BP); hydroxyl (OH) group

## Abstract

To determine the purity and quality of different brands of ciprofloxacin tablets by using UV spectrophotometry and FTIR spectroscopy as well as provide simple, cost-effective and sensitive spectrophotometric method for determination of ciprofloxacin tablets available in local market, Malaysia. Six different brands of ciprofloxacin tablets were purchased from local market, coded A to F and then analysed by UV spectrophotometry and FTIR spectroscopy. The UV spectrophotometry is based on the absorbance measurement of maximum wavelength of 276nm using distilled water and hydrochloric acid (HCl). All the tablets were evaluated for percentage purity with United State Pharmacopoeia (USP) and British Pharmacopoeia (BP) standards. In FTIR spectroscopy, the spectrum of six different brands of ciprofloxacin tablet were observed and compared among each other. All six ciprofloxacin brands met the specification of weight uniformity. The UV spectrophotometric results using distilled water revealed that two of the market brands are compliance with the official USP limit of 90-110% while only one brand was in the range of 95-105% according to official BP limit. The UV spectrophotometric results using HCl shows that fifty percent of the samples were within the acceptance USP range while four out of six brands fell within the BP specification. The FTIR spectrum of six different brands have the similar characteristic peak corresponding to the functional groups in the chemical structure. FTIR spectroscopy and UV spectrophotometry are developed as simple, quick and cost-effective methods. However, FTIR spectroscopy was more accurate and sensitive and gave better results than UV spectrophotometry [1-62]. The quality and safety of pharmaceutical products are very important.

## Introduction

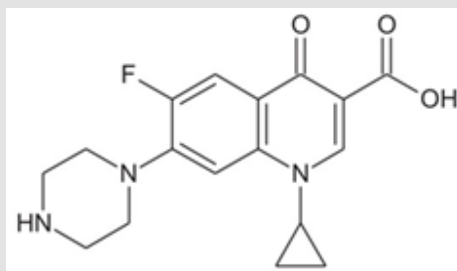


Figure 1.

The oral bioavailability of ciprofloxacin is about 70% to 80%, with minimal loss by first pass metabolism. It is absorbed readily but incompletely. After administration of single oral dose, maximum plasma concentration between 0.28 and 5.92mg/L are achieved within 0.5 to 2 hours. It is increased proportionally to the dose within the normal therapeutic range [11]. If ciprofloxacin is administered intravenously, plasma concentration profile follows 3-compartment pharmacokinetic model. It is moderately bound (20 to 40%) to plasma proteins and has a large apparent volume of distribution (2 to 3 L/kg). Ciprofloxacin is not readily penetrated

into the cerebrospinal fluid and placenta. However, it is penetrated and concentrated in many body tissues and fluids, including bile and kidney, liver, gallbladder, prostate and lung tissue. The availability of ciprofloxacin at the intestinal target site determines the effectiveness of antimicrobial therapy and clinical outcome of an infection [2] (Figure 1).

### Chemical Structure of Ciprofloxacin

The drug is official in British Pharmacopoeia presenting an HPLC assay for ciprofloxacin hydrochloride tablets and ciprofloxacin lactate intravenous infusion [14]. In Brazilian Pharmacopoeia [15], UV spectrophotometric, HPLC and a microbiological diffusion agar methods are the methods proposed to determine the injection, tablets and ophthalmic solutions of ciprofloxacin. Besides, United States Pharmacopoeia describes the HPLC method for ciprofloxacin and ciprofloxacin hydrochloride in bulk, ciprofloxacin injection and ophthalmic ointment and solution as well as tablets [16]. A number of studies regarding the purity determination of various Ciprofloxacin brands have been conducted in developing countries. However, there are a few research papers for purity determination of Ciprofloxacin and past research did not test this hypothesis directly in Malaysia. Moreover, reports of substandard and counterfeit drug are increasing recently in Malaysia. To assure effective and safe therapy, purity assessment is always a key parameter to figure out the correct chemical constitution of a drug especially API. Hence, this study is aimed to assess the percentage purity and quality of different brands of ciprofloxacin tablets formulation commercially available in the local market and determine whether the compounds meet the specification as stated in USP and BP by using a simple, sensitive and reproducible UV spectrophotometer and FTIR spectroscopy.

### Experimental

#### Chemicals, Glassware and Instruments

Freshly prepared 0.1N hydrochloric acid (HCl), Distilled water, 95% ethanol, Six different brands of ciprofloxacin tablets, Ciprofloxacin hydrochloride BP powder [53]. The standard ciprofloxacin hydrochloride BP powder was supplied as gift samples from the laboratory of Faculty of Pharmacy, AIMST University for comparison. The powder was manufactured by Aarti Drug Ltd. Maharashtra, India. Its manufacturing license number, batch number, manufacturing date and precaution were also labeled. Fisherbrand™ analytical balance. UV-VIS spectrophotometer Shimadzu UV-1800 with 1cm path length quartz cells for absorbance measurement [54]. It is connected to the analyzed computer for interpretation and determination of absorbance. All spectrophotometer measurements were at room temperature laboratory method [20]. Agilent Cary 630 FTIR spectrometer [56], Analyzed laptop, Agilent MicroLab FTIR software.




#### Collection of Different Brands of Ciprofloxacin Tablets





Six different commercially available leading brands of ciprofloxacin tablets, within their shelf life were purchased from various pharmacy outlets in Sungai Petani, Kedah, Malaysia [57,58]. Each brand of ciprofloxacin tablet were labelled to contain 500mg of ciprofloxacin. The ciprofloxacin tablets were blindly named as Brand A, Brand B, Brand C, Brand D, Brand E and Brand F in the present study. The descriptions regarding the brands are shown in (Tables 1 & 2).

**Table 1:** Detail of Standard Ciprofloxacin.

Detail of Standard Ciprofloxacin	
Manufacturing License Number	KD-217
Batch Number	18010093
Manufacturing Date	Jan 2018
Precaution	Store in Airtight Containers, Away from Light

**Table 2:** Description of each Ciprofloxacin brands.

Medication	Description
Brand A 	1) Branded drug
	2) Chemical name - ciprofloxacin hydrochloride monohydrate
	3) 1 strip contain 10 film-coated tablets
	4) Each tablets contain 500mg ciprofloxacin
	5) Transparent blister packaging
	6) A half cut mark on the tablets
	7) It is oval in shape
	8) It is white to yellowish white in colour,
	1) Generic drug
	2) Chemical name - ciprofloxacin hydrochloride monohydrate
	3) 1 strip contain 10 film-coated tablets
	4) Each tablets contain 500mg ciprofloxacin
	5) Transparent blister packaging
	6) A half cut mark on the tablets
	7) It is oval in shape
	8) It is white to yellowish white in colour
Brand C 	1) Generic drug
	2) Chemical name - ciprofloxacin hydrochloride monohydrate
	3) 1 strip contain 10 film-coated tablets
	4) Each tablets contain 500mg ciprofloxacin
	5) Transparent blister packaging
	6) A half cut mark on the tablets
	7) It is oval in shape
	8) It is white to yellowish white in colour

<p>Brand D</p> 	<ol style="list-style-type: none"> <li>1) Generic drug</li> <li>2) Chemical name - ciprofloxacin</li> <li>3) 1 strip contain 10 film-coated tablets</li> <li>4) Each tablets contain 500mg ciprofloxacin</li> <li>5) Opaque blister packaging</li> <li>6) No cut mark on the tablets</li> <li>7) It is oval in shape</li> <li>8) It is white to yellowish white in colour</li> </ol>
<p>Brand E</p>  	<ol style="list-style-type: none"> <li>1) Generic drug</li> <li>2) Chemical name - ciprofloxacin</li> <li>3) 1 strip contain 10 film-coated tablets</li> <li>4) Each tablets contain 500mg ciprofloxacin</li> <li>5) Transparent blister packaging</li> <li>6) A half cut mark on the tablets</li> <li>7) It is oval in shape</li> <li>8) It is orange in colour</li> </ol>
<p>Brand F</p> 	<ol style="list-style-type: none"> <li>1) Generic drug</li> <li>2) Chemical name - ciprofloxacin hydrochloride monohydrate</li> <li>3) 1 strip contain 5 film-coated tablets</li> <li>4) Each tablets contain 500mg ciprofloxacin</li> <li>5) Transparent blister packaging</li> <li>6) A half cut mark on the tablets</li> <li>7) It is oval in shape</li> <li>8) It is white to yellowish white in colour</li> </ol>

#### Preparation of Crushed Ciprofloxacin Powder

- 1) 20 tablets of each brands of ciprofloxacin are weighed accurately and the average weight was noted. The percentage deviation of each tablets were also calculated.
- 2) The powders were kept inside a re-sealable plastic bag to avoid the powder to contact with moisture which may cause the drug to denature.
- 3) The re-sealable plastic bags were labeled clearly with the name and the crushing date.
- 4) The plastic bags were kept in desiccator containing silica gel, which is a cool and dry place.

#### Preparation of Sample and Blank Solution

- 1) The average weight of each brand was calculated by using the formula below:

$$\text{equivalent weight} = \frac{\text{average weight}}{\text{label claim}} \times \text{weight taken}$$

- 2) The sample powders equivalent to 50mg of ciprofloxacin were weighed accurately and transferred into a volumetric flask.

- 3) 50mg of distilled water was added.
- 4) The mixture was then stirred for 10 minutes with help of magnetic stirrer and stirring bar. The volume was made up with distilled water (Solution A).
- 5) Solution A was filtered and 5ml of it was pipetted out into a 100ml volumetric flask and made up volume with distilled water (Solution B).
- 6) Solution B was pipetted out into another 100ml volumetric flask and made up volume with distilled water (Solution C). Solution C is the final solution.
- 7) The procedure for preparation of sample solution was repeated with freshly prepared 0.1N HCl instead of distilled water.
- 8) The blank solution was prepared by pipetting out 5ml of 0.1 HCl into 100ml volumetric flask and made up volume with distilled water.

#### UV Spectrophotometry

The absorbance of ciprofloxacin solution was scanned and measured as a function of wavelength in the 200-400nm UV regions. The maximum absorption wavelength ( $\lambda_{\text{max}}$ ) was observed at 276nm and this wavelength was adopted for absorbance measurement. All spectrophotometer measurements were at room temperature laboratory standard.

$$\text{Percentage purity} =$$

$$\frac{\text{standard absorbance} \times \text{dilution factor} \times \text{standard equivalent weight}}{\text{sample absorbance} \times \text{dilution factor} \times \text{sample equivalent weight}} \times 100$$

The absorbance of the resulting Solution C was measured at  $\lambda_{\text{max}}$  of 276nm

#### FTIR Spectroscopy

- 1) The platinum diamond sampling surface was cleaned before starting measurements of each sample using tissue paper with ethanol and allowed to dry.
- 2) A fresh background spectrum was measured against air and collected to ensure accuracy of data from samples.
- 3) Clean crystal check was performed to confirm that the system is ready for measurements [62].
- 4) Small amounts of finely ground Ciprofloxacin powder were placed on the diamond sampling crystal and pressed using a clamp to ensure proper contact.
- 5) The software analysis was performed. The spectrum of each sample was measured over the range of 4000-400cm<sup>-1</sup> with spectral resolution 2cm<sup>-1</sup>. Estimated scan time for spectral acquisition was 25 seconds.
- 6) The recorded fingerprint spectra of each samples was assessed and peaks were plotted.

7) Characteristic regions of the spectrum were identified and compared among standard and samples.

8) The percentage purity of the peak were calculated by using formula below:

$$\text{Percentage purity} = \frac{Au}{As} \times 100$$

Where, Au = absorbance of the sample solution,

As = absorbance of the standard solution

### Comparison of Percentage Purity of Ciprofloxacin

1) The percentage purity of all samples was compared with the limits in USP and BP, which are used as standard for the evaluation study. The samples fail the test if the percentage purity is not within the limit.

i. According to USP, ciprofloxacin must contain not less than 90.0 and not more than 110.0 percent of  $C_{17}H_{18}FN_3O_3$ , calculated on the dried basis.

ii. According to BP, ciprofloxacin must contain not less than 95.0 and not more than 105.0 percent of  $C_{17}H_{18}FN_3O_3$  of the dried substance.

2) The percentage purity of six different ciprofloxacin samples was determined by comparing with standard.

3) The percentage purity of samples using distilled water as blank solution was compared with that using freshly prepared 0.1 N hydrochloric acid (HCl) as blank solution.

4) The percentage purity of samples by UV spectrophotometric method was compared to that by FTIR spectroscopy.

### Results

The average weight of different brands of Ciprofloxacin were calculated and shown in (Tables 3 & 4). It indicates that brand E had the highest average weight (870.935mg) whereas brand F had the lowest average weight (728.825mg). The average weight for brand A, B, C and D were 766.49mg, 826.915mg, 775.605mg and 750.7mg respectively. The uniformity of weight of each brands were then assessed and shown in (Table 5). In (Graph 1), the first, second and third readings of standard ciprofloxacin were 0.011, 0.012 and 0.012 respectively. The average reading for the standard was 0.0117. There were total 3 trials carried out for assay of sample A and each trial had 3 readings. In first trial, the first, second and third readings were 0.017, 0.016 and 0.018 respectively. The average reading for first trial of sample A was 0.0160. In second trial, the first, second and third readings were 0.014, 0.017 and 0.019 respectively. The average reading for second trial of sample A was 0.0167. In third trial, the first, second and third readings were 0.021, 0.015 and 0.015 respectively. The average reading for third trial of sample A was 0.0170.

**Table 3:** The deviation limit of individual weight.

Average Weight of Tablets	Deviation	Number of Tablets
Less than 80mg	± 10.0 ± 20.0	Minimum 18 Maximum 2
80mg to 250mg	± 7.5 ± 15.0	Minimum 18 Maximum 2
More than 250mg	± 5.0 ± 10.0	Minimum 18 Maximum 2

**Table 4:** Average weight of different brands of Ciprofloxacin.

Brand No.	A	B	C	D	E	F
1	767.4	815.7	756.5	766.9	867.8	754.0
2	775.6	838.4	782.9	768.4	865.3	694.1
3	765.3	828.3	759.4	752.1	875.7	700.7
4	763.4	830.4	764.7	749.8	877.3	747.4
5	767.3	826.9	782.5	763.7	845.2	693.9
6	769.2	807.6	787.0	727.9	872.2	726.9
7	764.0	834.1	794.5	758.6	865.5	695.9
8	771.7	846.1	768.1	772.2	884.1	723.6
9	765.8	821.2	782.3	762.8	874.8	723.8
10	772.2	834.3	795.5	771.3	883.3	733.7
11	758.1	808.6	765.1	763.1	862.7	775.0
12	769.2	803.7	783.7	759.6	856.3	709.7
13	773.6	819.9	764.5	759.7	877.0	723.2
14	762.6	833.1	788.1	687.9	874.4	729.1
15	770.9	839.4	763.7	710.0	876.1	728.0
16	755.8	847.6	774.3	744.7	871.5	750.4
17	758.8	827.3	775.9	720.2	873.1	767.9
18	770.7	834.6	801.8	755.3	869.2	711.6
19	766.0	816.8	763.9	762.7	877.3	746.6
20	762.2	824.0	757.7	757.2	869.9	741.0
Average weight	766.49	826.915	775.605	750.7	870.935	728.825

Weight of each tablet for different brands (mg)

**Table 5:** Weight uniformity of different brands of Ciprofloxacin.

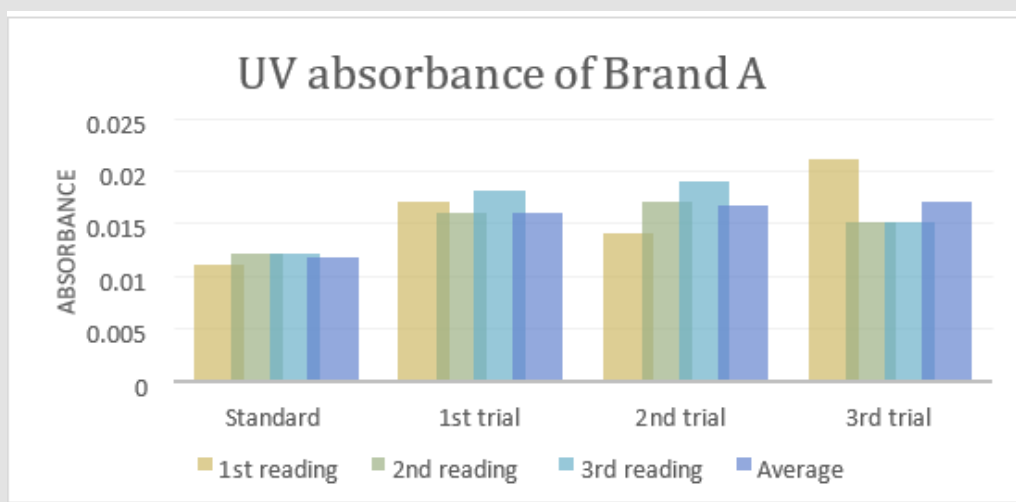
Brand No.	A	B	C	D	E	F
1	0.12	-1.36	-2.46	2.16	-0.36	3.45
2	1.19	1.39	0.94	2.36	-0.65	-4.76
3	-0.16	0.17	-2.09	0.19	0.55	-3.86
4	-0.40	0.42	-1.41	-0.12	0.73	2.55
5	-0.11	-1.81	0.89	1.73	-2.95	-4.80
6	0.35	-2.34	1.47	-3.04	0.15	-0.26
7	-0.32	0.87	2.44	1.05	-0.62	-4.52
8	0.68	2.32	-0.97	2.86	1.51	-0.72
9	-0.09	-0.69	0.86	1.61	0.44	-0.6710
10	0.74	0.89	2.57	2.74	1.42	0.67
11	-1.09	-2.21	-1.35	1.65	-0.95	6.34
12	0.35	-2.81	1.04	1.19	-1.68	-2.62

13	0.93	-0.85	-1.43	1.20	0.70	-0.77
14	-0.51	0.75	1.61	-8.37	0.40	0.04
15	0.58	1.51	-1.53	-5.42	0.59	-0.11
16	-1.39	2.50	-0.17	-0.80	0.06	2.96
17	-1.00	0.05	0.04	-4.06	0.25	5.36
18	0.55	0.93	3.38	0.61	-0.20	-2.36
19	-0.06	-1.22	-1.51	1.60	0.73	2.44
20	-0.56	-0.35	-2.31	0.87	-0.12	1.67

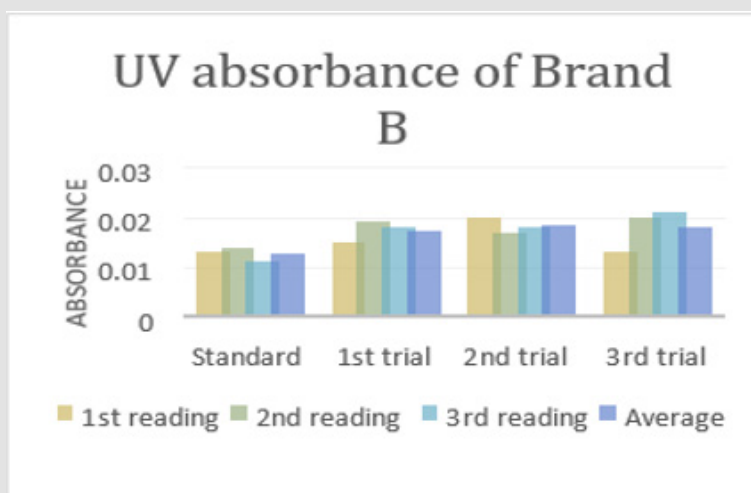
Percentage deviation of each tablet for different brands (%).

In (Graph 2), the first, second and third readings of standard ciprofloxacin were 0.013, 0.014 and 0.011 respectively. The average reading for the standard was 0.0127. There were total 3 trials carried out for assay of sample B and each trial had 3 readings. In first trial, the first, second and third readings were 0.015, 0.019 and 0.018 respectively. The average reading for first trial of sample

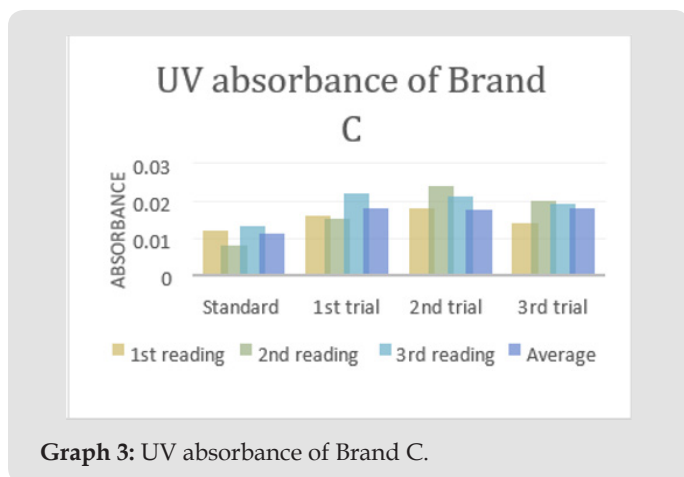
B was 0.0173. In second trial, the first, second and third readings were 0.020, 0.017 and 0.018 respectively. The average reading for second trial of sample B was 0.0183. In third trial, the first, second and third readings were 0.018, 0.024 and 0.019 respectively. The average reading for third trial of sample B was 0.0180. In (Graph 3), the first, second and third readings of standard ciprofloxacin were 0.012, 0.008 and 0.013 respectively. The average reading for the standard was 0.0110. There were total 3 trials carried out for assay of sample C and each trial had 3 readings. In first trial, the first, second and third readings were 0.016, 0.015 and 0.022 respectively. The average reading for first trial of sample C was 0.0177. In second trial, the first, second and third readings were 0.018, 0.024 and 0.021 respectively. The average reading for second trial of sample C was 0.0173. In third trial, the first, second and third readings were 0.014, 0.020 and 0.019 respectively. The average reading for third trial of sample B was 0.0177.



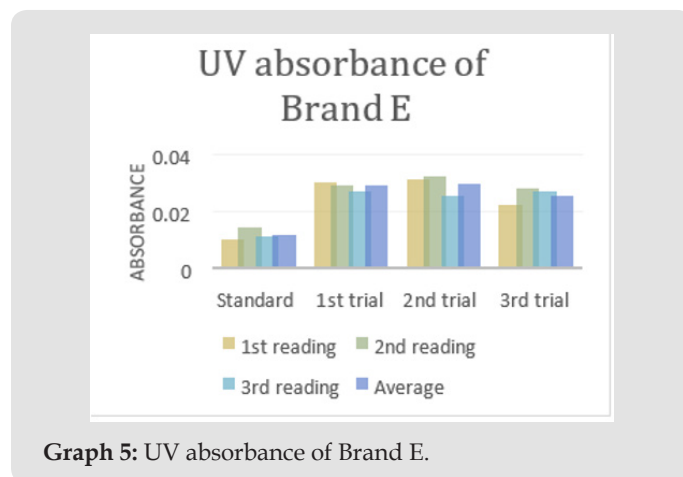
Graph 1: UV absorbance of Brand A.



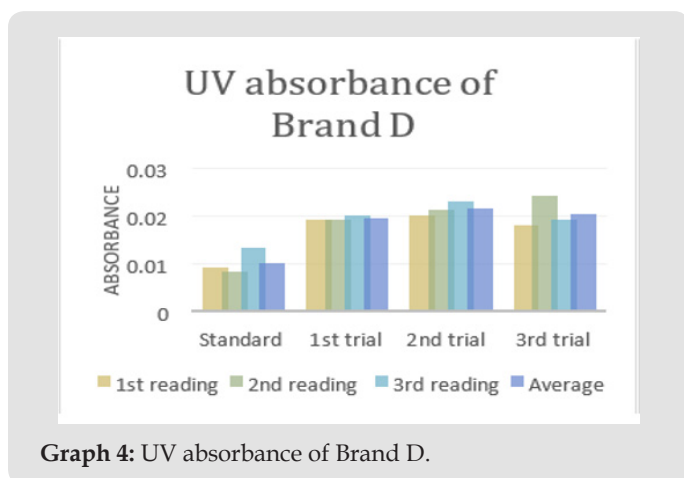
Graph 2: UV absorbance of Brand B.



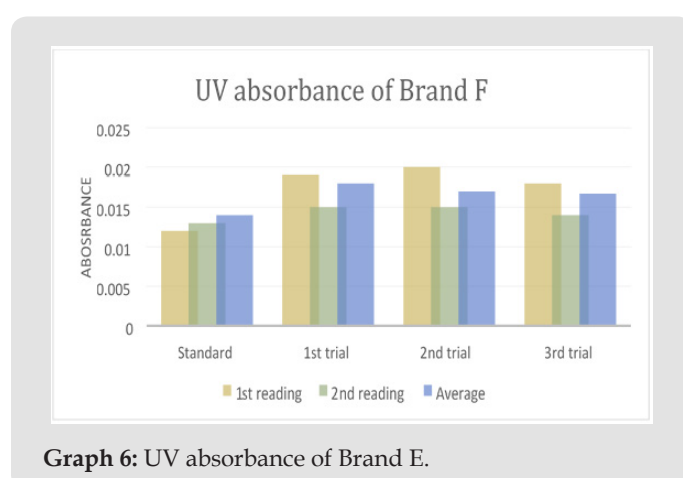
Graph 3: UV absorbance of Brand C.



Graph 5: UV absorbance of Brand E.



Graph 4: UV absorbance of Brand D.



Graph 6: UV absorbance of Brand F.

In (Graph 4), the first, second and third readings of standard ciprofloxacin were 0.014, 0.011 and 0.014 respectively. The average reading for the standard was 0.0130. There were total 3 trials carried out for assay of sample D and each trial had 3 readings. In first trial, the first, second and third readings were 0.025, 0.024 and 0.020 respectively. The average reading for first trial of sample D was 0.0230. In second trial, the first, second and third readings were 0.026, 0.023 and 0.024 respectively. The average reading for second trial of sample D was 0.0243. In third trial, the first, second and third readings were 0.022, 0.028 and 0.027 respectively. The average reading for third trial of sample D was 0.0253. In (Graph 5), the first, second and third readings of standard ciprofloxacin were 0.010, 0.014 and 0.011 respectively. The average reading for the standard was 0.0117. There were total 3 trials carried out for assay of sample E and each trial had 3 readings. In first trial, the first, second and third readings were 0.030, 0.029 and 0.027 respectively. The average reading for first trial of sample E was 0.0287. In second trial, the first, second and third readings were 0.032, 0.033 and 0.029 respectively. The average reading for second trial of sample E was 0.0313. In third trial, the first, second and third readings were 0.031, 0.032 and 0.025 respectively. The average reading for third trial of sample E was 0.0293. In (Graph 6), the first, second and third readings of standard ciprofloxacin were 0.012, 0.012 and 0.017 respectively. The average reading for the standard was 0.0140.

There were total 3 trials carried out for assay of sample F and each trial had 3 readings. In first trial, the first, second and third readings were 0.019, 0.015 and 0.020 respectively. The average reading for first trial of sample F was 0.0180. In second trial, the first, second and third readings were 0.020, 0.015 and 0.016 respectively. The average reading for second trial of sample F was 0.0170. In third trial, the first, second and third readings were 0.018, 0.014 and 0.018 respectively. The average reading for third trial of sample F was 0.0167. The percentage purity determination of different brands of Ciprofloxacin with distilled water as blank by using UV spectrophotometry was tabulated in (Tables 6 & 7). For ciprofloxacin standard, the percentage purity was found to be 100%. There were 3 trials conducted for each assay of sample. The percentage purity of first, second and third trials of sample A were 112.09%, 107.39% and 105.49% respectively. The average percentage purity of sample A was 108.32%. The percentage purity of first, second and third trials of sample B were 121.42%, 114.78% and 116.70% respectively. The average percentage purity of sample B was 117.63%. The percentage purity of first, second and third trials of sample C were 96.41%, 98.64% and 96.41% respectively. The average percentage purity of sample A was 97.15%. The percentage purity of first, second and third trials of sample D were 84.87%, 80.33% and 77.15% respectively. The average percentage purity of sample D was 80.78%. The percentage purity of first,

second and third trials of sample E were 71.02%, 65.12% and 69.57% respectively. The average percentage purity of sample E was 68.57%. The percentage purity of first, second and third trials of sample F were 113.38%, 120.05% and 122.20% respectively. The average percentage purity of sample F was 118.54%.

**Table 6:** Absorbance of different brands of Ciprofloxacin with distilled water as blank.

Brand	Blank	Trail	Reading			
			1 <sup>st</sup>	2 <sup>nd</sup>	3 <sup>rd</sup>	Average
A	0	1 <sup>st</sup>	0.017	0.016	0.018	0.016
		2 <sup>nd</sup>	0.014	0.017	0.019	0.0167
		3 <sup>rd</sup>	0.021	0.015	0.015	0.017
		Standard	0.011	0.012	0.012	0.0117
B	0	1 <sup>st</sup>	0.015	0.019	0.018	0.0173
		2 <sup>nd</sup>	0.02	0.017	0.018	0.0183
		3 <sup>rd</sup>	0.013	0.02	0.021	0.018
		Standard	0.013	0.014	0.011	0.0127
C	0	1 <sup>st</sup>	0.016	0.015	0.022	0.0177
		2 <sup>nd</sup>	0.018	0.024	0.021	0.0173
		3 <sup>rd</sup>	0.014	0.02	0.019	0.0177
		Standard	0.012	0.008	0.013	0.011
D	0	1 <sup>st</sup>	0.025	0.024	0.02	0.023
		2 <sup>nd</sup>	0.026	0.023	0.024	0.0243
		3 <sup>rd</sup>	0.022	0.028	0.027	0.0253
		Standard	0.014	0.011	0.014	0.013
E	0	1 <sup>st</sup>	0.03	0.029	0.027	0.0287
		2 <sup>nd</sup>	0.032	0.033	0.029	0.0313
		3 <sup>rd</sup>	0.031	0.032	0.025	0.0293
		Standard	0.01	0.014	0.011	0.0117
F	0	1 <sup>st</sup>	0.019	0.015	0.02	0.018
		2 <sup>nd</sup>	0.02	0.015	0.016	0.017
		3 <sup>rd</sup>	0.018	0.014	0.018	0.0167
		Standard	0.012	0.013	0.017	0.014

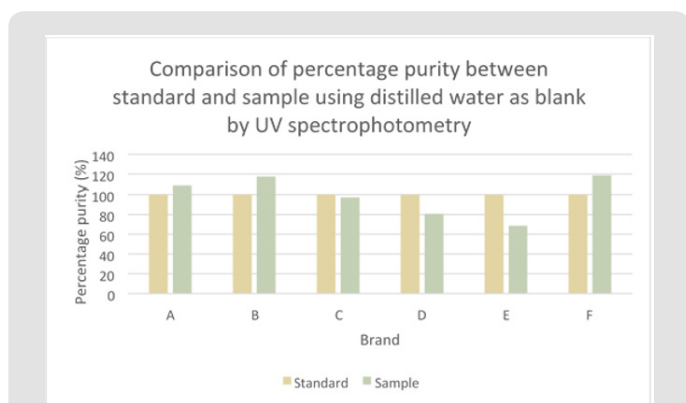
**Table 7:** WPercentage purity determination of different brands of Ciprofloxacin with distilled water as blank by using UV spectrophotometry.

Brand	Equivalent weight (mg)	Percentage Purity (%)					Standard(%)
		Sample	1 <sup>st</sup> Trial	2 <sup>nd</sup> Trial	3 <sup>rd</sup> Trial	Average	
A	500.0	326.2	112.09	107.39	105.49	108.32	100
B	500.0	302.3	121.42	114.78	116.70	117.63	100
C	500.0	322.3	96.41	98.64	96.41	97.15	100
D	500.0	333.0	84.87	80.33	77.15	80.78	100
E	500.0	287.0	71.02	65.12	69.57	68.57	100
F	500.0	343.0	113.38	120.05	122.20	118.54	100

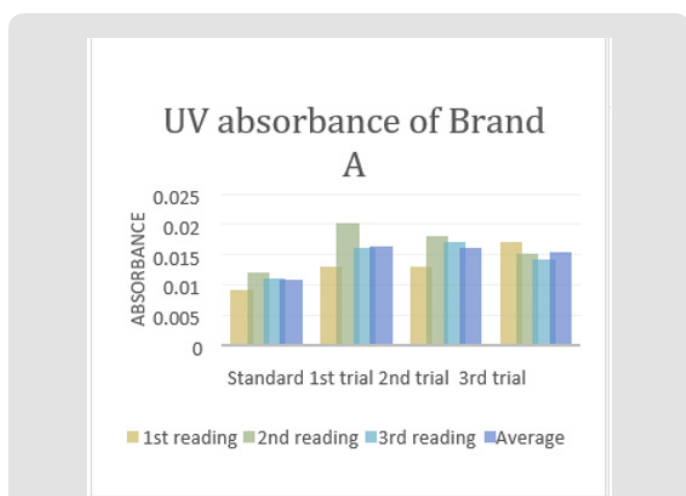
The comparison of percentage purity between standard and sample using distilled water as blank by UV spectrophotometry was shown in (Graph 7). For ciprofloxacin standard, the percentage purity was 100%. According to the graph, as compared to standard ciprofloxacin, Brand C had the most similar percentage purity while Brand E had the most different percentage purity. According

to United State Pharmacopoeia (USP), the percentage purity of ciprofloxacin in tablet dosage form should be in the range of 90.0% and 110.0%. Thus, Brand A and C passed the test whereas Brand B, D, E and F failed the test. While according to British Pharmacopoeia (BP), ciprofloxacin tablets should contain not less than 95.0% and not more than 105.0% of the labeled amount of ciprofloxacin.

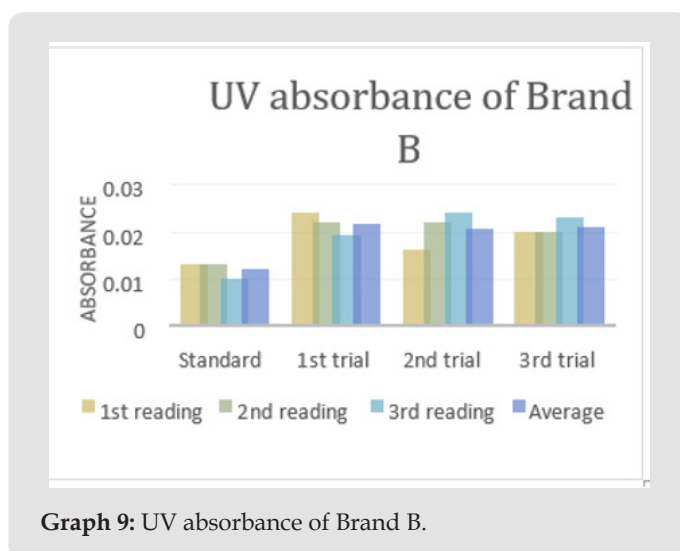
Thus, only Brand C passed the test while other brands failed the test. In (Graph 8), the first, second and third readings of standard ciprofloxacin were 0.009, 0.012 and 0.011 respectively. The average reading for the standard was 0.0107. There were total 3 trials carried out for assay of sample A and each trial had 3 readings. In first trial, the first, second and third readings were 0.013, 0.020 and 0.016 respectively. The average reading for first trial of sample A was 0.0163. In second trial, the first, second and third readings were 0.012, 0.018 and 0.017 respectively. The average reading for second trial of sample A was 0.0157. In third trial, the first, second and third readings were 0.024, 0.014 and 0.015 respectively. The average reading for third trial of sample A was 0.0107. In (Graph 9), the first, second and third readings of standard ciprofloxacin were 0.013, 0.013 and 0.010 respectively. The average reading for the standard was 0.0120. There were total 3 trials carried out for assay of sample B and each trial had 3 readings. In first trial, the first, second and third readings were 0.024, 0.022 and 0.019 respectively. The average reading for first trial of sample B was 0.0217. In second trial, the first, second and third readings were 0.016, 0.022 and 0.024 respectively.



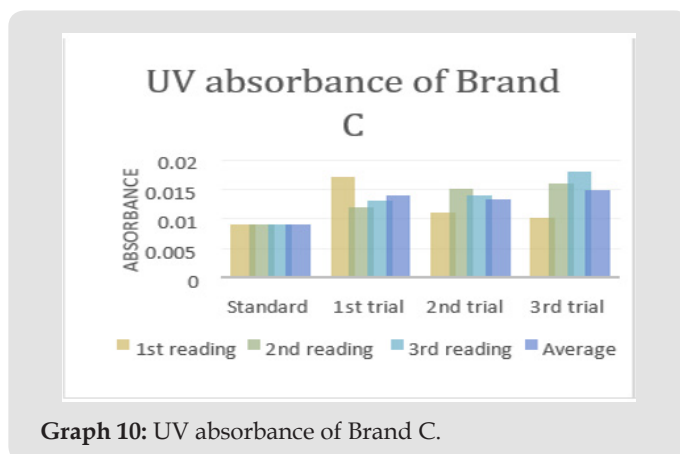
**Graph 7:** Comparison of percentage purity between standard and sample using distilled water as blank by UV spectrophotometry.



**Graph 8:** UV absorbance of Brand A.



**Graph 9:** UV absorbance of Brand B.

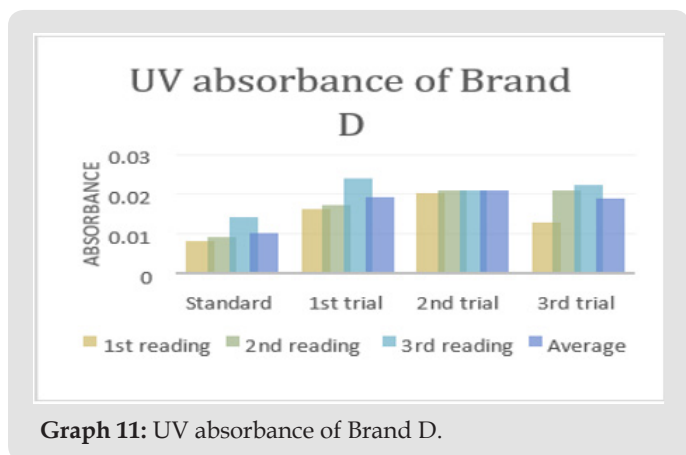


**Graph 10:** UV absorbance of Brand C.

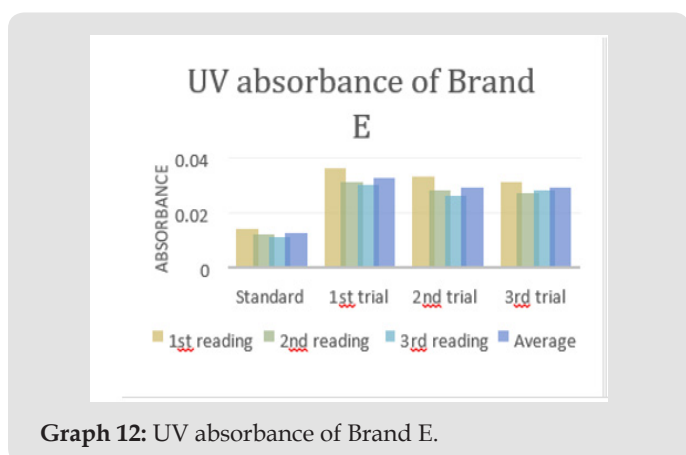
The average reading for second trial of sample B was 0.0207. In third trial, the first, second and third readings were 0.020, 0.020 and 0.023 respectively. The average reading for third trial of sample B was 0.0210. In (Graph 10), the first, second and third readings of standard ciprofloxacin were 0.009, 0.009 and 0.009 respectively. The average reading for the standard was 0.009. There were total 3 trials carried out for assay of sample C and each trial had 3 readings. In first trial, the first, second and third readings were 0.017, 0.012 and 0.013 respectively. The average reading for first trial of sample C was 0.0140. In second trial, the first, second and third readings were 0.011, 0.015 and 0.014 respectively. The average reading for second trial of sample C was 0.0133. In third trial, the first, second and third readings were 0.010, 0.016 and 0.018 respectively. The average reading for third trial of sample B was 0.0147. In (Graph 11), the first, second and third readings of standard ciprofloxacin were 0.008, 0.009 and 0.014 respectively. The average reading for the standard was 0.0103. There were total 3 trials carried out for assay of sample D and each trial had 3 readings. In first trial, the first, second and third readings were 0.016, 0.017 and 0.024 respectively. The average reading for first trial of sample D was 0.0190. In second trial, the first, second and third readings were



0.020, 0.021 and 0.021 respectively. The average reading for second trial of sample D was 0.0207. In third trial, the first, second and third readings were 0.013, 0.021 and 0.022 respectively. The average reading for third trial of sample D was 0.0187.



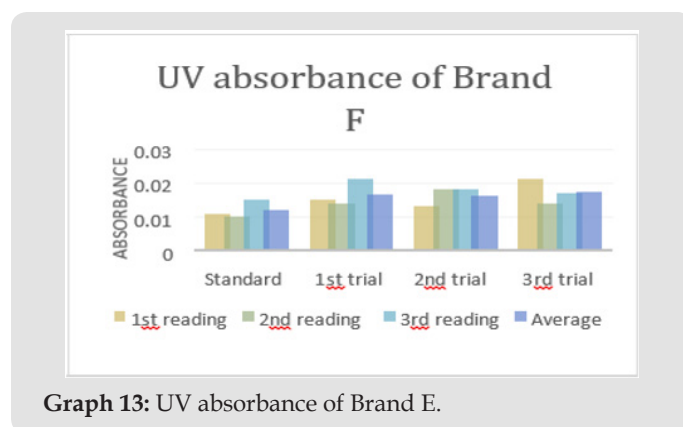
Graph 11: UV absorbance of Brand D.



Graph 12: UV absorbance of Brand E.

In (Graph 12), the first, second and third readings of standard ciprofloxacin were 0.014, 0.012 and 0.011 respectively. The average reading for the standard was 0.0123. There were total 3 trials carried out for assay of sample E and each trial had 3 readings. In first trial, the first, second and third readings were 0.036, 0.031 and 0.030 respectively. The average reading for first trial of sample E was 0.0323. In second trial, the first, second and third readings were 0.033, 0.028 and 0.026 respectively. The average reading for second trial of sample E was 0.0290. In third trial, the first, second and third readings were 0.031, 0.027 and 0.028 respectively. The average reading for third trial of sample E was 0.0287. In (Graph 13), the first, second and third readings of standard ciprofloxacin

were 0.011, 0.010 and 0.015 respectively. The average reading for the standard was 0.0120. There were total 3 trials carried out for assay of sample F and each trial had 3 readings. In first trial, the first, second and third readings were 0.015, 0.014 and 0.021 respectively. The average reading for first trial of sample F was 0.0167. In second trial, the first, second and third readings were 0.013, 0.018 and 0.018 respectively. The average reading for second trial of sample F was 0.0163. In third trial, the first, second and third readings were 0.021, 0.014 and 0.017 respectively. The average reading for third trial of sample F was 0.0173.



Graph 13: UV absorbance of Brand F.

The percentage purity determination of different brands of Ciprofloxacin with HCl as blank by using UV spectrophotometry was tabulated in (Tables 8 & 9). For ciprofloxacin standard, the percentage purity was 100%. There were 3 trials conducted for each assay of sample. The percentage purity of first, second and third trials of sample A were 100.62%, 102.51% and 107.20% respectively. The average percentage purity of sample A was 103.44%. The percentage purity of first, second and third trials of sample B were 91.46, 95.88% and 87.59% respectively. The average percentage purity of sample B was 91.64%. The percentage purity of first, second and third trials of sample C were 99.73%, 104.98% and 94.98% respectively. The average percentage purity of sample C was 99.90%. The percentage purity of first, second and third trials of sample D were 81.40%, 74.71% and 82.70% respectively. The average percentage purity of sample D was 79.60%. The percentage purity of first, second and third trials of sample E were 66.34%, 73.89% and 74.66% respectively. The average percentage purity of sample E was 71.63%. The percentage purity of first, second and third trials of sample F were 104.75%, 107.32% and 101.11% respectively.

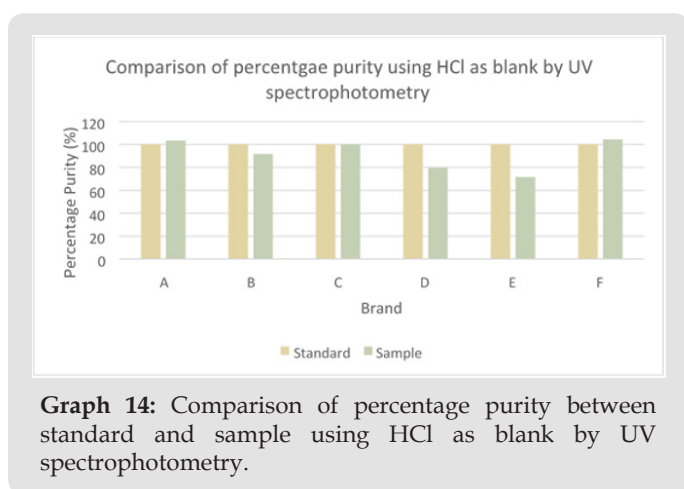
Table 8: Absorbance of different brands of Ciprofloxacin with HCl as blank.

Brand	Blank	Trail	Reading			
			1 <sup>st</sup>	2 <sup>nd</sup>	3 <sup>rd</sup>	Average
A	0	1 <sup>st</sup>	0.013	0.020	0.016	0.0163
		2 <sup>nd</sup>	0.013	0.018	0.017	0.0160
		3 <sup>rd</sup>	0.017	0.015	0.014	0.0153
		Standard	0.009	0.012	0.011	0.0107

B	0	1 <sup>st</sup>	0.024	0.022	0.019	0.0217
		2 <sup>nd</sup>	0.016	0.022	0.024	0.0207
		3 <sup>rd</sup>	0.020	0.020	0.023	0.0210
		Standard	0.013	0.013	0.010	0.0120
C	0	1 <sup>st</sup>	0.017	0.012	0.013	0.0140
		2 <sup>nd</sup>	0.011	0.015	0.014	0.0133
		3 <sup>rd</sup>	0.010	0.016	0.018	0.0147
		Standard	0.009	0.009	0.009	0.0090
D	0	1 <sup>st</sup>	0.016	0.017	0.024	0.0190
		2 <sup>nd</sup>	0.020	0.021	0.021	0.0207
		3 <sup>rd</sup>	0.013	0.021	0.022	0.0187
		Standard	0.008	0.009	0.014	0.0103
E	0	1 <sup>st</sup>	0.036	0.031	0.030	0.0323
		2 <sup>nd</sup>	0.033	0.028	0.026	0.0290
		3 <sup>rd</sup>	0.031	0.027	0.028	0.0287
		Standard	0.014	0.012	0.011	0.0123
F	0	1 <sup>st</sup>	0.015	0.014	0.021	0.0167
		2 <sup>nd</sup>	0.013	0.018	0.018	0.0163
		3 <sup>rd</sup>	0.021	0.014	0.017	0.0173
		Standard	0.011	0.010	0.015	0.0120

**Table 9:** Percentage purity determination of different brands of Ciprofloxacin with HCl as blank by using UV spectrophotometry.

Brand	Equivalent Weight (mg)		Percentage Purity (%)				Standard (%)
	Standard	Sample	1 <sup>st</sup> Trial	2 <sup>nd</sup> Trial	3 <sup>rd</sup> Trial	Average	
A	500.0	326.2	100.62	102.51	107.20	103.44	100
B	500.0	302.3	91.46	95.88	87.59	91.64	100
C	500.0	322.3	99.73	104.98	94.98	99.90	100
D	500.0	333.0	81.40	74.71	82.70	79.60	100
E	500.0	287.0	66.34	73.89	74.66	71.63	100
F	500.0	343.0	104.75	107.32	101.11	104.39	100

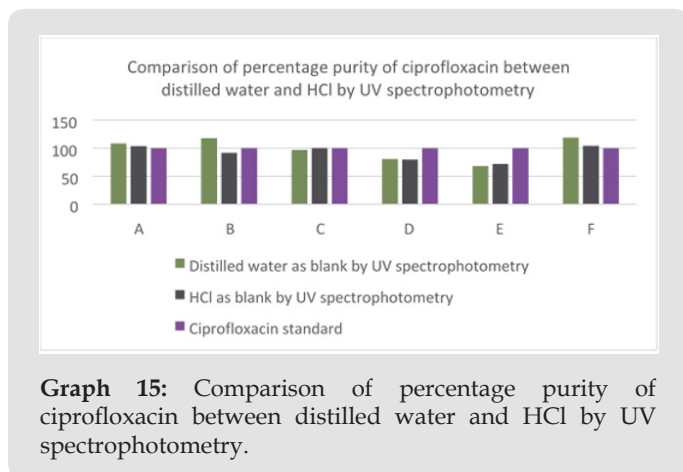


**Graph 14:** Comparison of percentage purity between standard and sample using HCl as blank by UV spectrophotometry.

The average percentage purity of sample F was 104.39%. The comparison of percentage purity between standard and sample using HCl as blank by UV spectrophotometry was shown in (Graph

14). For ciprofloxacin standard, the percentage purity was 100%. The percentage purity of Brand A, B, C, D, E and F were found to be 103.44%, 91.64%, 94.55%, 79.60%, 71.63% and 104.39%. According to the graph, as compared to standard ciprofloxacin, Brand A had the most similar percentage purity while Brand E had the most different percentage purity. According to United State Pharmacopoeia (USP), the percentage purity of ciprofloxacin in tablet dosage form should be in the range of 90.0% and 110.0%. Thus, Brand A, B, C and F passed the test whereas Brand D and E failed the test. While according to British Pharmacopoeia (BP), ciprofloxacin tablets should contain not less than 95.0% and not more than 105.0% of the labeled amount of ciprofloxacin. Thus, Brand A, C and F passed the test. Comparison of percentage purity of ciprofloxacin between UV spectrophotometry and FTIR spectrophotometry was shown in (Graph 15). For the ciprofloxacin standard, the percentage purity was 100%. The average percentage purity of Brand A, B, C, D, E and F using distilled water as blank by UV

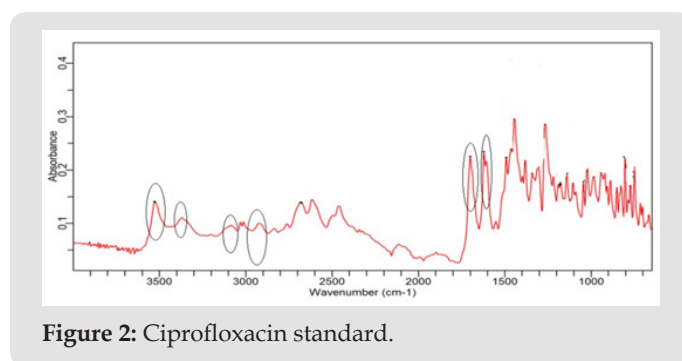
spectrophotometry were found to be 104.73%, 117.63%, 76.54%, 80.78%, 68.57% and 109.35% respectively. Besides, the average percentage purity of Brand A, B, C, D, E and F using HCl as blank by UV spectrophotometry were 103.44%, 91.64%, 94.55%, 79.60%, 71.63% and 104.39% respectively. According to the graph, Brand C had the most similar percentage purity between distilled water and HCl by UV spectrophotometry as compared to ciprofloxacin standard. Whereas, Brand E has the different percentage purity.



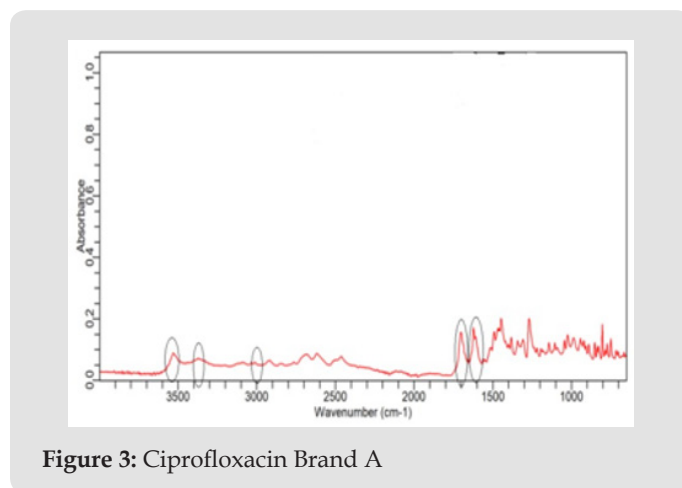
**Graph 15:** Comparison of percentage purity of ciprofloxacin between distilled water and HCl by UV spectrophotometry.

### Measurement of FTIR Spectrum of Different Brands of Ciprofloxacin by FTIR Spectroscopy

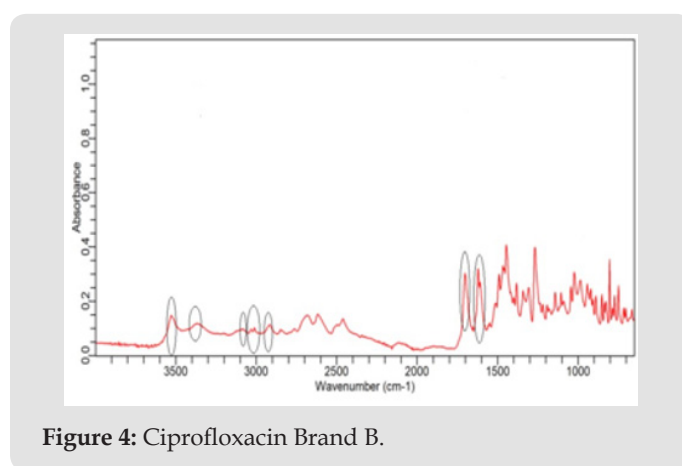
(Figure 2) shows the FTIR spectra of ciprofloxacin standard. The prominent characteristic peaks were found between 3500-3450 $\text{cm}^{-1}$ , which was assigned to stretching vibration of hydroxyl (OH) group and intermolecular hydrogen bonding. Another band at 3000-2950 $\text{cm}^{-1}$  represented the alkene and aromatic C-H stretching, especially  $\nu=\text{C-H}$ . The peak at 2900 $\text{cm}^{-1}$  was assigned to C-H stretching vibration of cyclopropyl group. The region from 1750 to 1700 $\text{cm}^{-1}$  represented the carbonyl C=O stretching. The band at 1650 to 1600 $\text{cm}^{-1}$  was assigned to quinolones. (Figure 3) shows the FTIR spectra of ciprofloxacin Brand A. The prominent characteristic peaks were found between 3500-3450 $\text{cm}^{-1}$ , which was assigned to stretching vibration of hydroxyl (OH) group and intermolecular hydrogen bonding. Another band at 3000-2950 $\text{cm}^{-1}$  represented the alkene and aromatic C-H stretching, especially  $\nu=\text{C-H}$ . The region from 1750 to 1700 $\text{cm}^{-1}$  represented the carbonyl C=O stretching. The band at 1650 to 1600 $\text{cm}^{-1}$  was assigned to quinolones. (Figure 4) shows the FTIR spectra of ciprofloxacin Brand B. The prominent characteristic peaks were found between 3500-3450 $\text{cm}^{-1}$ , which was assigned to stretching vibration of hydroxyl (OH) group and intermolecular hydrogen bonding. The other bands at 3000-2950 $\text{cm}^{-1}$  represented the alkene and aromatic C-H stretching, especially  $\nu=\text{C-H}$ . The peak at 2900 $\text{cm}^{-1}$  was assigned to C-H stretching vibration of cyclopropyl group. The region from 1750 to 1700 $\text{cm}^{-1}$  represented the carbonyl C=O stretching. The band at 1650 to 1600 $\text{cm}^{-1}$  was assigned to quinolones. (Figure 5) shows the FTIR spectra of ciprofloxacin Brand C.



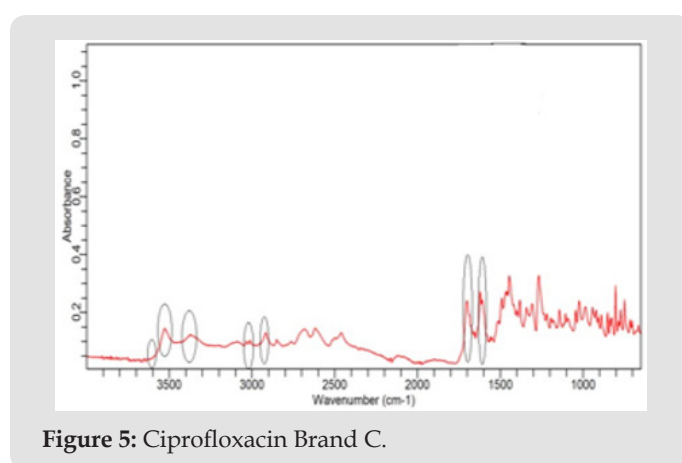
**Figure 2:** Ciprofloxacin standard.



**Figure 3:** Ciprofloxacin Brand A



**Figure 4:** Ciprofloxacin Brand B.



**Figure 5:** Ciprofloxacin Brand C.

The prominent characteristic peaks were found between 3500-3450 $\text{cm}^{-1}$ , which was assigned to stretching vibration of hydroxyl (OH) group and intermolecular hydrogen bonding. Another band at 3000-2950 $\text{cm}^{-1}$  represented the alkene and aromatic C-H stretching, especially  $\nu=\text{C-H}$ . The peak at 2900 $\text{cm}^{-1}$  was assigned to C-H stretching vibration of cyclopropyl group. The region from 1750 to 1700 $\text{cm}^{-1}$  represented the carbonyl C=O stretching. The band at 1650 to 1600 $\text{cm}^{-1}$  was assigned to quinolones. (Figure 6) shows the FTIR spectra of ciprofloxacin Brand E. The prominent characteristic peaks were found between 3500-3450 $\text{cm}^{-1}$ , which was assigned to stretching vibration of hydroxyl (OH) group and intermolecular hydrogen bonding. Another band at 3000-2950 $\text{cm}^{-1}$  represented the alkene and aromatic C-H stretching, especially  $\nu=\text{C-H}$ . The peak at 2900 $\text{cm}^{-1}$  was assigned to C-H stretching vibration of cyclopropyl group. The region from 1750 to 1700 $\text{cm}^{-1}$  represented the carbonyl C=O stretching. The band at 1650 to 1600 $\text{cm}^{-1}$  was assigned to quinolones. (Figure 7) shows the FTIR spectra of ciprofloxacin Brand F. The prominent characteristic peaks were found between 3500-3450 $\text{cm}^{-1}$  which was assigned to stretching vibration of hydroxyl (OH) group and intermolecular hydrogen bonding. Another band at 3000-2950 $\text{cm}^{-1}$  represented the alkene and aromatic C-H stretching, especially  $\nu=\text{C-H}$ . The peak at 2900 $\text{cm}^{-1}$  was assigned to C-H stretching vibration of cyclopropyl group. The region from 1750 to 1700 $\text{cm}^{-1}$  represented the carbonyl C=O stretching (Figure 8). The band at 1650 to 1600 $\text{cm}^{-1}$  was assigned to quinolones.

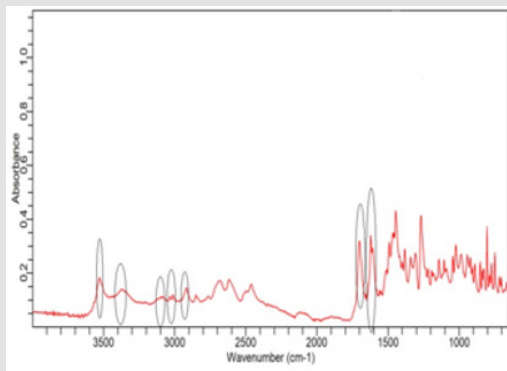


Figure 6: Ciprofloxacin Brand D.

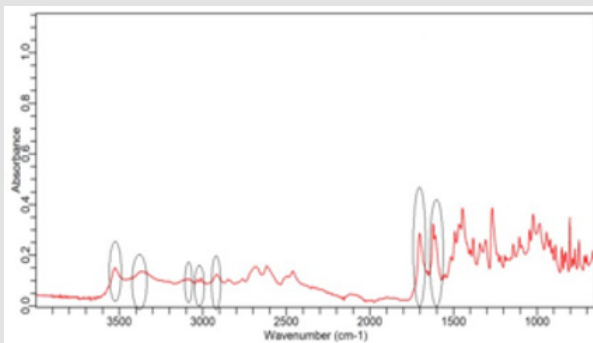


Figure 7: Ciprofloxacin Brand E.

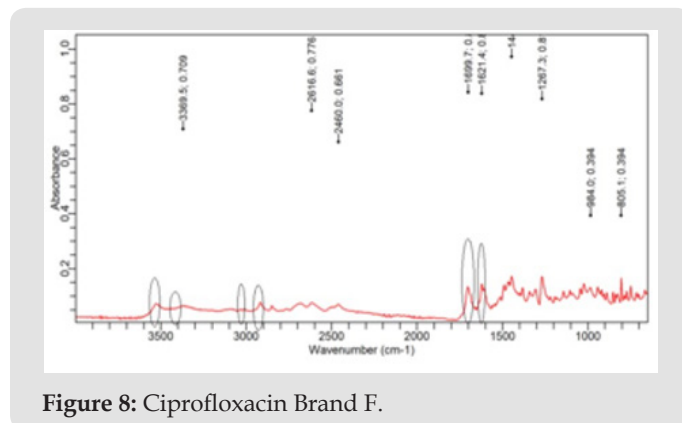


Figure 8: Ciprofloxacin Brand F.

## Discussion

In this study, the objective were to determine the purity and quality of different brands of ciprofloxacin tablets by using UV spectrophotometry and FTIR spectroscopy as well as provide simple, cost-effective and sensitive spectrophotometric method for determination of ciprofloxacin tablets. Six different marketed brands of ciprofloxacin tablets were purchased. The weight of the tablet is defined as the amount of granules which contains the labeled amount of the active pharmaceutical ingredient. Weight uniformity test is required to assure that the drug content in each unit dose is distributed in a narrow range around the label strength. If the drug substance forms the greater part of the oral solid dosage form, any weight variation obviously reflects variation in the content of active ingredient [41]. The smaller the weight variation, the better the uniformity of content. The standards for uniformity of weight are applied to tablets, which are supplied in unit dose form as they are subjected to more variations than comparable preparations supplied in multi-dose forms. For tablets with average weight above 250mg, the percentage deviation from the average weight permissible in the official compendium (BP, 2008) is  $\pm 5\%$ . The six different brands of ciprofloxacin met the specification. However, each two tablets from Brand E and F exceeded the 5% deviation from the average weight, but still conformed to the official compendium specification, which stipulates that not more than two tablets must deviate by 5% from the average weight, if twenty tablets are used for the test.

The occurrence of errors might be due to the presence of tablet fragments or dust on the tablets when weighed, wind from airconditioning, flowing properties of the powder, differences in the bulk densities as well as pressured used and particle size distribution during compression. The UV spectrophotometric method depends on the absorbance measurement of drug at the specific wavelength. It is suitable for the quantitative analysis of ciprofloxacin tablets since it contains suitable chromophore that absorbs radiation in the UV region at the maximum wavelength of 276nm. The maximum wavelength was selected as it can be eliminated the interference from excipients. The distilled water was utilized as a solvent as it is easily available and cheaper than

other solvent as well as ciprofloxacin is soluble in hydrochloric acid. The filtration can remove the un-dissolved excipients from ciprofloxacin solution. In the assay UV method using distilled water as blank, the results showed that only Brand C passed the test while other samples fell outside the official BP limit. According to USP, two out of six samples (Brand A and C) fell within the USP limit. Brand D and E had the percentage purity level below the official stated limit while Brand B and F had the percentage purity level above the upper limit. In contrast to the assay method using HCl as blank, fifty percent of the brands of ciprofloxacin tablet (Brand A, C and F) conformed to the official BP limits of 90-110%. According to USP, four out of six samples (Brand A, B, C and F) had the percentage purity within the stated requirement. On comparing the two solvents used, it was obvious that the assay results using HCl as blank solution was more accurate than that using distilled water. The reason includes that ciprofloxacin dissolve satisfactorily in HCl rather than in distilled water. The percentage purity might also be affected by factors such as temperature, humidity and light.

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### Conflict of Interest

The authors declare that there is no conflict of interest.

### Single Journal Submission

This research paper has not been submitted anywhere else.

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