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# Comparative study of the quality control of Ciprofloxacin sold in Niamey city by Thin Layer Chromatography

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**ABSTRACT:** The aim of this research is to control the quality of Ciprofloxacin used alongside and in the health centers and sold in Niamey city by using Thin Layer Chromatography method to compare the results achieved in order to contribute to informed decision-making for possible interventions. This research aimed to investigate on eleven samples distributed as follows which five samples are from pharmacies, five samples are from street vendors and one sample specialty used as reference to check the quality control of them. The different reagents used are: Methanol, Dichloromethane, Ammonia at 260g$L^{-1}$ or Pure ammonia, Acetonitrile and 0.1 M NaOH. The migration of all the samples in proposed diluent shows that all of them contain the active ingredient of ciprofloxacin. The percentage of the active ingredient were calculated as per protocol of Clarke's analysis of drugs and poisons in chemistry guidelines. It varies from 90.90 to 109.09 for pharmacies and 98.18 to 106.35 for the street vendors.
According to the results of the different frontal reports, each sample contains the percentage of active principle recommended by WHO which is 90 to 110 % of active ingredient recommended by WHO.
This technique of quality control can be used for practical work or tutorial and laboratories where drug quality control mechanism is not often checked.

**KEYWORDS:** Ciprofloxacin, Thin Layer Chromatography, pharmacies, street vendors, Niamey.

## 1 INTRODUCTION

Ciprofloxacin is an antibacterial agent used to treat infections of the skin, sinuses, bones, lungs, abdomen, kidneys, prostate and bladder and also some sexually transmitted infections, certain forms of diarrhea. infectious origin, and typhoid fever [1, 2, 19].

Its crude formula is: $C_{17}H_{18}FN_3O_3$ and its molecular weight is 331.4. It is part of the family of Quinolones and Fluoroquinolones and of the group of fluoro-quinolones [3,4].

Its IUPAC Name is 1-Cyclopropyl-6-fluoro-4-oxo-7-piperazin-1-ylquinoline-3-carboxylic acid [1,5, 6].

Its chemical properties: with decomposition Melting point is from 255° to 257°. It is practically insoluble in water; very slightly soluble in ethanol and in methylene chloride; soluble in dilute acetic acid [1, 5].

There are two types of Ciprofloxacin:

- **Ciprofloxacin Hydrochloride**

Its crude formula is: $C_{17}H_{18}FN_3O_3$, HCl, $H_2O$ and its molecular weight is 385.8 [5].

- **Ciprofloxacin Lactate**

Its crude formula is: $C_{17}H_{18}FN_3O_3$, $C_3H_6O_3$ and its molecular weight is 421.4 [5].

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Its chemical properties: faintly yellowish to light yellow crystals. Melting point is 318° to 320°. It is sparingly soluble to soluble in water; slightly soluble in acetic acid and in methanol; very slightly soluble in ethanol; practically insoluble in acetone, in acetonitrile, in ethyl acetate, in hexane and in methylene chloride [1,5].

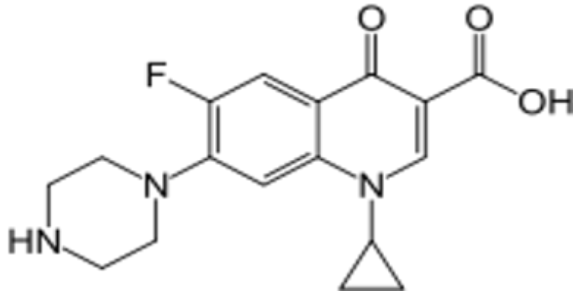


*Fig. 1. Chemical structure of Ciprofloxacin [3]*

## 2 MATERIAL AND METHOD

This study was carried out using a survey sheet containing a few questions and a camera allowing the TLC of the different samples to be photographed [7].

### 2.1 POPULATION

The population is represented by the antibiotics (from pharmacies and street vendors) most commonly sold in the Urban Community of Niamey and used in hospitals and health centers (Niamey National Hospital, Amirou Garga Hospital of Lamordé, University Hospital Center and Maternity Issaka Gazobi) [8 to 11].

### 2.2 SAMPLING AND SIZE

Our sample is represented by three (3) types of antibiotics of Ampicillin, namely:

- Reference antibiotic;
- The antibiotics most commonly used in health centers (HNN, HNL, CHR and MIG) sold in pharmacies;
- Antibiotics sold by itinerants.

Thus, our study extended on the size of eleven (11) samples distributed as follows:

- 5 samples for pharmacies;
- 5 samples for street vendors;
- 1 sample specialty used as reference for the molecules to be analyzed.

### 2.3 REAGENTS USED FOR THIN LAYER CHROMATOGRAPHY (TLC) OF CIPROFLOXACIN

Methanol, Dichloromethane, Ammonia at 260g$L^{-1}$ or Pure ammonia, Acetonitrile and 0.1 M NaOH were used for the TLC of ciprofloxacin [12, 17].

### 2.4 THE METHODOLOGY

Before proceeding to the actual operating mode (MO), we made a preliminary test as follows: take a chromatoplate 20 cm * 20 cm which we divide in half and cut the length of the desired chromatoplate, then using the pencil and from the graduated ruler, draw a line of 1.5 cm from the bottom of the sheet that will serve as a baseline.

Identify in pencil the different products to be analyzed by personal codes, separating them by 1cm so as to occupy the entire baseline according to the number of products to be spot on this same baseline; finally prepare the usage and mobile phase solutions, and number the test tubes according to the seller's category.

### 2.5 PREPARATION OF THE MOBILE PHASE OF THE CIPROFLOXACIN TLC

The mobile phase was prepared in the chromatographic tank in which 40 mL of methanol, 40 mL of dichloromethane, 20 mL of ammonia (260gL$^{-1}$) or pure ammonia and 10 mL of acetonitrile are introduced and shaked the mixture; after we closed it and stirred well.

A test portion of 20 mg of ciprofloxacin is introduced into the test tubes identified using the markers and immersed with 2 ml of 0.1 M NaOH; it was well mixed, then spot with the capillary tubes on the chromatographic sheets [12, 16]. The sheets were placed in the chromatographic chamber in order to follow the migration. After removing them from the chromatographic chamber, they were air dried for 15 minutes and the iodine chromatogram was examined.

***Table 1. Weights and Average of weights of the Ciprofloxacin samples***

| Samples Identity | Corresponding Weight of 500 mg (mg) | Average of weight (mg) |
|---|---|---|
| **Reference** | 765; 767; 769 | 767 |
| **$P_1$** | 930; 910; 920; 930; 950; 920; 920; 920; 930; 940 | 927 |
| **$P_2$** | 760; 790; 740; 800; 790; 780; 770; 760; 780; 760 | 773 |
| **$P_3$** | 760; 770; 740; 780; 750; 750; 790; 760; 780; 790 | 767 |
| **$P_4$** | 950; 960; 960; 980; 940; 940; 890; 920; 890; 840 | 927 |
| **$P_5$** | 750; 770; 760; 730; 740; 750; 770; 750; 770; 720 | 751 |
| **$V_1$** | 1070; 460; 1020; 570; 960; 580; 1010; 640; 660; 850 | 782 |
| **$V_2$** | 740; 770; 760; 780; 800; 780; 720; 790; 760; 790 | 769 |
| **$V_3$** | 770; 850; 1250; 410; 1260; 400; 450; 1200; 840; 820 | 825 |
| **$V_4$** | 790; 770; 300; 1250; 1180; 370; 1160; 350; 760; 480 | 741 |
| **$V_5$** | 660; 670; 970; 350; 640; 370; 950; 1080; 250; 680 | 662 |

*P = pharmacy; V= Street seller*

The mobile phase was prepared in the chromatographic tank into which 60 ml of ethyl acetate, 20 ml of acetic acid and 20 ml of distilled water were introduced [13].

The tank was closed and well agitated. Wait at least 15 minutes for the chromatographic chamber to be saturated and at the same time place the different samples on the chromatoplate [12]. Finally, the chromatographic sheet was immersed in the chromatographic tank and wait for migration. The chromatoplate was taken out of the chromatographic chamber to dry it in the free area, then put it in the chromatographic tank in order to observe and verify the migration of stains.

After TLC, the RF of each sample was calculated by the following formula:

$$\boldsymbol{RF} = \frac{\mathbf{X}}{\mathbf{Y}} \qquad [14]$$

Where:

RF: is the frontal report

X: Distance traveled by the solute

Y: Distance traveled by the solvent

After this the active ingredient content of each sample was calculated by the following formula:

$$\boldsymbol{T} = \left(\frac{\boldsymbol{RF\ éch}}{\boldsymbol{RF\ réf}}\right) \times \mathbf{100\ \%} \qquad [15]$$

Where:

*RF éch:* is the frontal report of the sample;

*RF réf:* is the frontal report of the reference.

## 3 RESULTS AND DISCUSSION

### 3.1 PRESENTATION OF TLC ANALYSIS RESULTS

After having carried out the procedure from the spotage to the observation of the iodine chromatograms, the plates were photographed and represented in the form of the figure below (Figure 2).

*Fig. 2. TLC plate photograph of ciprofloxacin samples*

The figure above shows through the photograph of the various thin layer chromatography (TLC) of the samples of ciprofloxacin. It has been observed that all of them have migrated; which leads us to conclude that the active ingredient (a.i.) exists in these samples and that whatever the content [18].

After presenting the TLC photographs of the different samples of ciprofloxacin, we measured the distances traveled by the different solvents and samples in cm (Table 2).

### 3.2 PRESENTATION OF THE MIGRATION RESULTS

*Table 2. Presentation of the distances covered by the samples (cm) of Ciprofloxacin*

| Samples and solvent | Distance of migration (cm) |
|---|---|
| **Solvent** | 8,0 |
| **Réf** | 5,5 |
| **$P_1$** | 6 |
| **$P_2$** | 5 |
| **$P_3$** | 5,5 |
| **$P_4$** | 5,6 |
| **$P_5$** | 5,5 |
| **$V_1$** | 5,7 |
| **$V_2$** | 5,8 |
| **$V_3$** | 5,85 |
| **$V_4$** | 5,6 |
| **$V_5$** | 5,4 |

*Réf: reference, P = pharmacy; V=Street seller*

Then the Frontal Report (RF) of each sample has been calculated to see the active ingredient content which is illustrated on Table 3.

***Table 3. The frontal ratios of the different samples of Ciprofloxacin***

| Samples Identity | Frontal ratio (cm) | % a.i. |
|---|---|---|
| **Réf** | 0,6875 | |
| **$P_1$** | 0,75 | 109,09 |
| **$P_2$** | 0,625 | 90,90 |
| **$P_3$** | 0,6875 | 100 |
| **$P_4$** | 0,7 | 101,81 |
| **$P_5$** | 0,6875 | 100 |
| **$V_1$** | 0,7125 | 103,66 |
| **$V_2$** | 0,7312 | 106,35 |
| **$V_3$** | 0,7312 | 106,35 |
| **$V_4$** | 0,7 | 101,81 |
| **$V_5$** | 0,675 | 98,18 |

*Réf: reference, P = pharmacy; V= Street seller, a.i.: active ingredient*

This table shows us that all the ciprofloxacin samples have good active ingredient. The best are from $P_3$ and $P_5$ with 100 % of the active ingredient content otherwise all meet the standards recommended by the WHO which is 90 to 100 % [12, 20].

## 4 CONCLUSION

The results of the study reveal that in the two cases (pharmacy and street vendors) the following lessons for the TLC.

The results show that the migration was effective for all the samples; therefore, this reveals the presence of Active Ingredient in the samples.

All of Ciprofloxacin samples purchased (pharmacies and street vendors) meet WHO standards which is between 90 and 110 %; but the best is from P3 and P5 with 100 % of the active ingredient content.

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