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ANALYSIS BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY OF IBUPROFEN TABLET

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ABSTRACT: A rapid and sensitive method for the determination of the best conditions that allow us to calibrate the ibuprofen in drug is presented. This approach uses high performance liquid chromatography with DAD detector, which is a separation method most used because it is fast, easy, and maintains the nature of the mixture to be separated. The spectra of ibuprofen were recorded using several mobile phases “58%Na₂HPO₄ (50mmol/l), 42%ACN” and “60%ACN, 40% water “when the pH values are (pH=2.5 and pH=5); the flow rate values are 0.9ml/min -1.5 ml/min with detection wavelength of 240nm; and two stationary phases using the C18 column and Phenyl urea column (25cm x 4.6mm, 0.5µm). The ibuprofen sample: 1 mg of ibuprofen dissolved in 1 mL of water. For this we have tried through our study aiming relying on foreign references.

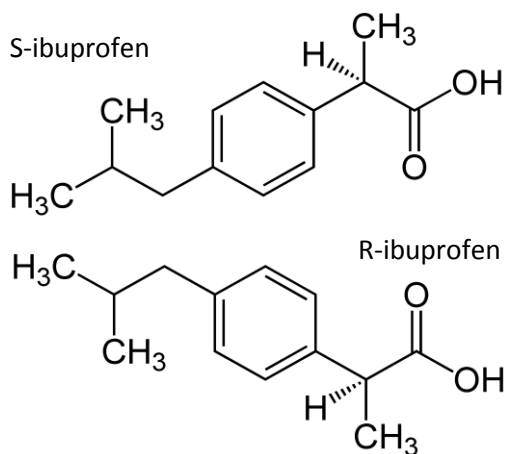
INTRODUCTION: Anti-inflammatory drugs are a non-homogeneous therapeutic class, containing substances that have the property to antagonize in a nonspecific way with the main manifestation of the inflammatory process, regardless of the etiology. Non-steroidal anti-inflammatory drugs (NSAIDs) have a rich diversity of chemical structures and they have a complex pharmacokinetic effect, but they display several common properties as:

- Anti-inflammatory, analgesic and antipyretic properties;
- Most are weak organic acids;
- They are absorbed well by the organism;
- They easily link with albumins;

- For the most cases they are metabolized in a high proportion;
- They are eliminated through kidneys;
- They are gastric irritants.

NSAIDs are split into two groups, the classic non-steroidal anti-inflammatory drugs, or the first generation (COX-1) and the selective or specific inhibitor non-steroidal anti-inflammatory drugs from the second generation (COX-2). The first generation is represented by drugs that contain in their molecule carboxylic acids (salicylic acid derivatives, acetic acid derivatives, propionic acid derivatives etc.) and enola acids, and the second generation is represented by selective blockers (meloxicam, nimesulid) or specific blockers (celecoxib, parecoxib, etoricoxib, valdecoxib, lumiracoxib). Ibuprofen, (RS)-2-(4-isobutylphenyl) propionic acid, “its formula is C₁₃H₁₈O₂, its molecular weight is 206 g/mol” is an important non-steroidal anti-inflammatory analgesic and antipyretic drug widely used in the treatment of rheumatic disorders, pain, and fever.

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This drug is normally administrated as the racemate, but only the (S) - form is responsible for the pharmacological effect. Numerous studies have documented the pharmacokinetics of ibuprofen and described the marked unidirectional inversion of the (R) - to the (S)-form.

EXPERIMENTAL:

Reagents: Soidal of Dar El BAIDA supplied the drug under investigation (ibuprofen).

Methanol: CH₃-OH: M = 32.04 g/mol, P = 99.98%

Salt: Na₂HPO₄: M = 141.96 g / mol, C = 0.05 M, P = 99.98%

ACN acetonitrile: M = 41.05 g/mol, P = 99.98%

H₃PO₄ phosphoric acid for pH adjustment: M = 98 g/mol, P = 98%, Distilled water.

Chromatographic apparatus and conditions:
The samples were analyzed by HPLC [WATERS 2690 Separation module, type Alliance (Injection

auto, Degazer on line)] which was equipped with a photo Diode Array Detector: WATERS 996. A C18 (25cm × 4.6 mm, 5μm) and a Phenyl urea (25cm × 4.6 mm, 5μm) chromatographic column were used at room temperature.

The pH value is measured by EUTECH Instruments pH 510 pH/mv/C°mètre Cyberscan with glass electrode.

The quantity of reagent was measured by analytical balance S.MC AC 210 (SARTORUIS): 10⁻⁴ accuracy.

The wavelength of the UV-Detection was set at 240 nm.

Several mobile phases were used:

58%Na₂HPO₄ (50mmol/l), 42%ACN pH=3.5 at a flow rate of 1.5mL/min. 58%Na₂HPO₄ (50mmol/l), 42%ACN pH=5 at a flow rate of 1.5mL/min and at a flow rate of 0.9 mL/min; and 60% ACN, 40% Eau pH=2.5 at a flow rate of 1.5mL/min for the in samples.

Detection of the sample by HPLC: 5μl of sample is injected and this operation is repeated using the C6 and C18 columns with percentage change of the compounds of the mobile phase and the flow values are 0.9 ml / min, 1.5 ml / min.

RESUTTS AND DISCUSSION: The values of retention time are shown in the following spectra:

Chromatograms of ibuprofen on a C18 column: (Figures 1-4)

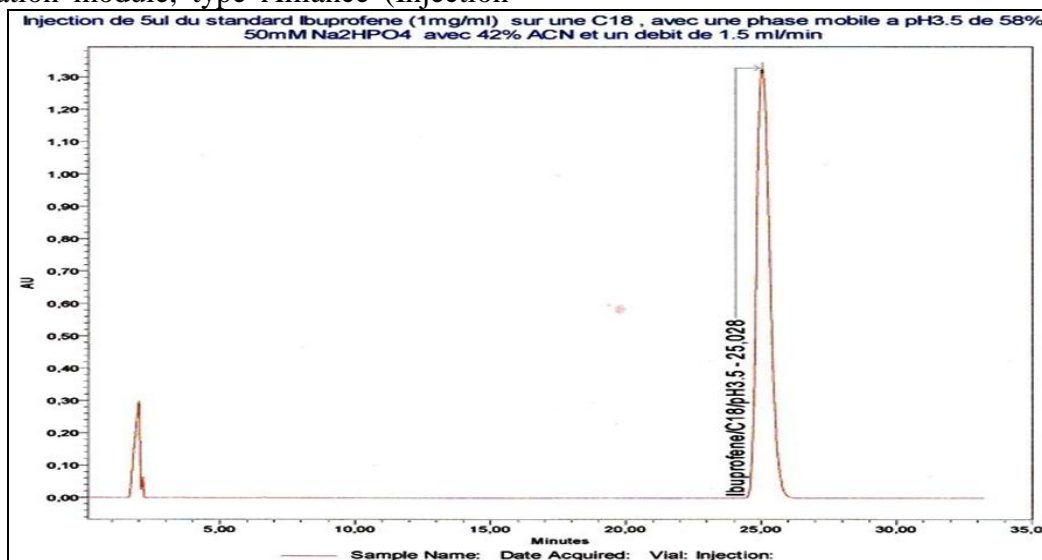


FIGURE 1: 58%Na₂HPO₄ (50mmol/l), 42%ACN pH=3.5 at a flow rate of 1.5mL/min

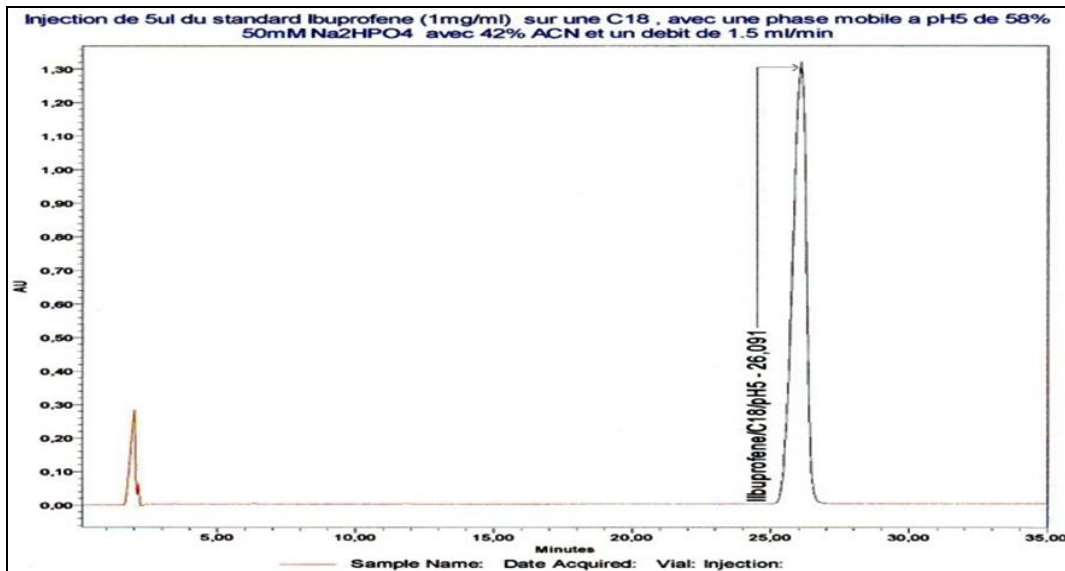


FIGURE 2: 58%Na₂HPO₄ (50mmol/l), 42%ACN pH=5 at a flow rate of 1.5mL/min

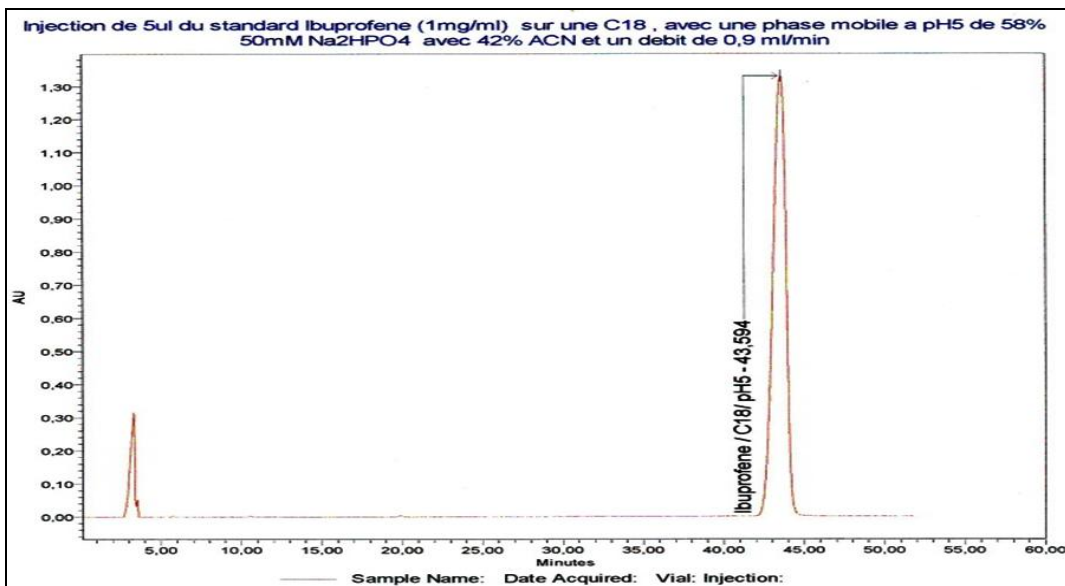


FIGURE 3: 58%Na₂HPO₄ (50mmol/l), 42%ACN pH=5 at a flow rate of 0.9mL/min

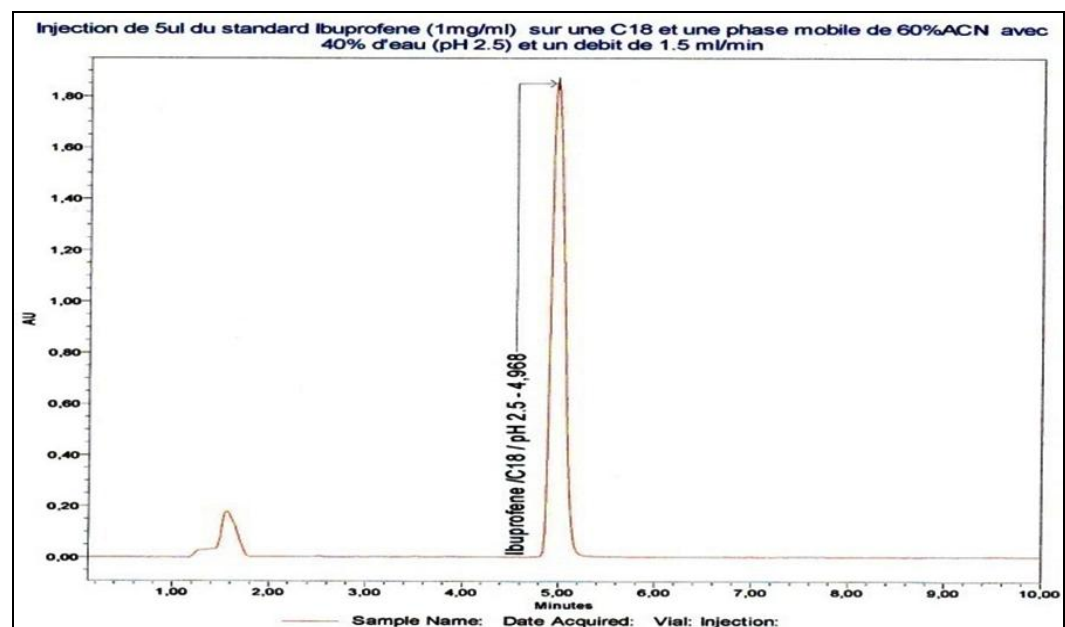


FIGURE 4: 60% ACN, 40% water pH=2.5 at a flow rate of 1.5mL/min

Chromatograms of ibuprofen on a Penyl urea column: (Figures 5-7)

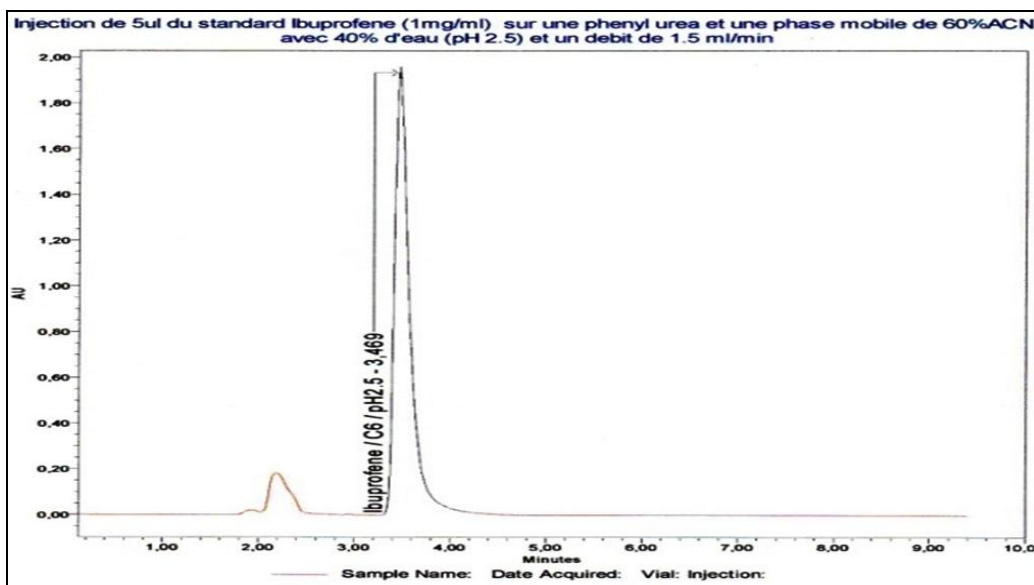


FIGURE 5: 60% ACN, 40% water pH=2.5 at A flow rate of 1.5mL/min

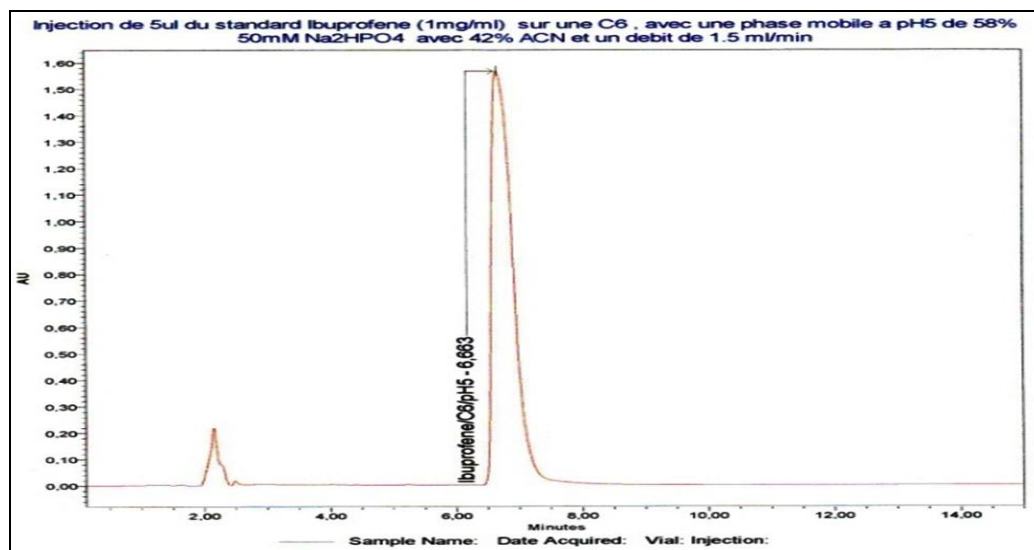


FIGURE 6: 58%Na₂HPO₄ (50mmol/l), 42%ACN pH=5 at a flow rate of 1.5mL/min

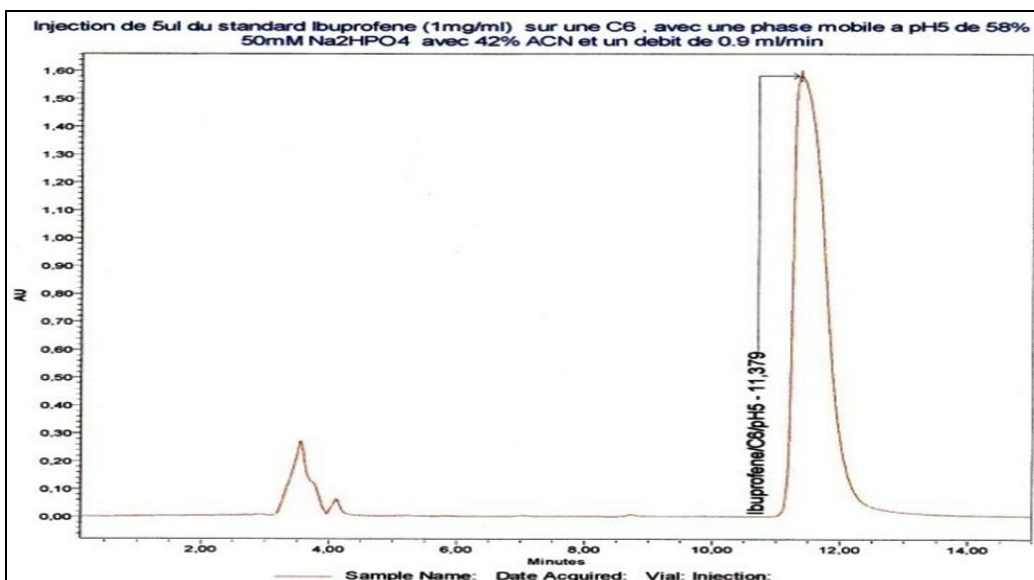


FIGURE 7: 58%Na₂HPO₄ (50mmol/l), 42%ACN pH=5 at a flow rate of 0.9 mL/min

TABLE 1: HPLC

Retention time	Flow rate (ml/min)	pH	Mobile phase	Figure	Stationary Phase
25.028	1.5	3.5	58% Na ₂ HPO ₄ (50Mm) + 42% ACN	1	
26.091	1.5	5	58% Na ₂ HPO ₄ (50Mm) + 42% ACN	2	
43.594	0.9	5	58% Na ₂ HPO ₄ (50Mm) + 42% ACN	3	C18
4.968	1.5	2.5	60% ACN + 40% Water	4	
3.469	1.5	2.5	60% ACN + 40% water	5	
6.663	1.5	5	58% Na ₂ HPO ₄ (50Mm) + 42% ACN	6	Phenyl Urea (C6)
11.379	0.9	5	58% Na ₂ HPO ₄ (50Mm) + 42% ACN	7	

Stationary phase: C18

- Using the first mobile phase at pH = 3.5 and pH=5 with a flow rate of 1.5ml/min retention times were $t_r = 25.028$ min and $t_r = 26.091$ min
- Using the first mobile phase at pH = 5 with a flow rate of 1.5ml/min and 0.9 ml / min retention times were $t_r = 26.091$ min and $t_r = 43.594$ min.
- Using the first mobile phase at pH = 3.5, the retention time was $t_r = 25.028$ min.
- Using the second mobile phase at pH = 2.5 the retention time was $t_r = 25.028$ min.

Stationary phase: phenyl urea

- Using the second mobile phase at pH = 2.5 the retention time was $t_r = 3.489$ min.
- Using the second mobile phase at pH = 5 the retention time was $t_r = 6.663$ min.
- Using the second mobile phase at pH = 5 with a flow rate of 1.5ml/min and 0.9 ml / min retention times were $t_r = 6.663$ min, $t_r = 11.379$ min.

Validation of the method: Calibration curve was linear ($r \geq 0.9854$) from solutions of ibuprofen tablet with known concentrations (100 mg / ml, 80 mg / mL, 60 mg / mL, 40 mg / mL, and 20 mg / ml).

The curve obtained: is straight, its equation is of the form: $Y=0.51X-0.034$

The correction factor: $r = 0.9990$.

When we inject the ibuprofen solution prepared several times and we calculate the area of protrusion and we calculate the CV (coefficient of

change), the injection is in the chromatographic conditions.

The value of CV must be <5%

We have found CV = 2.6% and RSD = 1.4% (relative deviation)

CONCLUSION: The retention time is directly proportional to the pH of the mobile phase.

The retention time is inversely proportional to the flow. The retention time is directly proportional to the pH of the mobile phase and is connected to the rate of the compounds of the phase.

The best working conditions: Determination of anti-inflammatory ibuprofen tablet by HPLC "are:

- Use of mobile phase "60% Acetonitrile, 40% water", pH = 2.5.
- The use of the stationary phase PHENYL UREA (C6)
- Adjust the flow rate to 1.5 mL/min

CONCLUSION: The retention time is directly proportional to the pH of the mobile phase. The retention time is inversely proportional to the flow. The retention time is directly proportional to the pH of the mobile phase and is connected to the rate of the compounds of the phase.

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