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Reverse Phase -High Performance Liquid Chromatography technique with Ultra-Violet detector for the Determination of Tenoxicam and Ibuprofen Drugs in the pure and Pharmaceutical tablets

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Abstract

Reversed-phase high-performance liquid chromatography (RP-HPLC) equipped with an ultraviolet (UV) detector is capable of detecting drugs in trace amounts. In this study, a simple and rapid method for quantifying tenoxicam (TNX) and ibuprofen (IBU) in pure and pharmaceutical samples was developed and validated. The target analytes assessed in the present study were separated with a C18 column (HPLC column, 5 µm, 150 × 4.6 mm). Acetonitrile and acidified water [0.7 mL of phosphoric acid (H3PO4) in 1000 mL water at 50/50%] were employed as the mobile phase at a 1 ml/min flow rate and a 20 µl sample injection volume at 25°C. Condition calibration curves for each drug were obtained within the 10–50 µg/mL dynamic concentration range. The method proposed in this study exhibited good performance, where the TNX and IBU recorded limit of detection (LOD) values of 1.5677 and 0.7911 µg/mL at 0.5, 1.3, 5.0, 7.0, 9.0, and 10.0 µg/mL. Resultantly, the method possessed specificity, linearity, precision, and accuracy. The suggested approach was satisfactory and appropriate for determining TNX and IBU levels during routine quality control assessments of medications in pure forms, mixtures, and formulations.

Keywords: Calibration curves, ibuprofen, reversed-phase high-performance liquid chromatography, tenoxicam, UV detector.

Introduction

High-performance liquid chromatography (HPLC) is commonly applied to evaluate samples of biological fluids food[1,2,] clinical, health and diseases[3], plastics[4], toxic molecules[5], medicinal plants[6], drugs[7], and environmenta[8] and separation of contaminants[9]. The technique is also utilised in Green chemistry[10]. Ultraviolet (UV) detectors are frequently employed in HPLC as most compounds absorb light, especially at low UV wavelengths. Furthermore, the method offers affordability and flexibility.

Reversed-phase HPLC (RP-HPLC) equipped with a UV detector has been employed in the simultaneous determination of tenoxicam (TNX) and ibuprofen (IBU) in pure standard and pharmaceutical tablets. Nonetheless, pure solvents and reagents and proper sample preparation and handling are necessary to minimise background noise[11]. Approximately a 10:1

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ratio is required to detect the intensity of the smallest detectable analyte in quantitative assessments.

Medications with analgesic, antipyretic, and anti-inflammatory attributes are classified as non-steroidal anti-inflammatory drugs (NSAIDs) [12], of which TNX and IBU are the most known members.

The TNX or its chemical name 4-hydroxy-2-methyl-n-(pyredine-2-yl)-2H-thino[2,3-e][1,2]thiziene-3-carboxamide 1,1-dioxide [13]is illustrated in Fig. 1. The molecular formula of TNX is $C_{13}H_{11}N_3O_4S_2$, and its molecular mass is 337.4 g mol^{-1}. TNX is soluble in distilled water and organic solvents, including ethanol[14].

TNX obstructs the actions of COX enzymes, the chemicals responsible for pain and inflammation. Various techniques have been employed to determine the levels of TNX, including HPLC[15], UV[16] , fluorescence[17], flow injection[18], and ion-pair[19] spectrophotometry, electro analytical[20], voltammetry[21], X-ray[22], scanning electron microscopy (SEM), X-ray diffraction, dynamic light scattering (DLS), Fourier transform infrared (FTIR)[23], photo catalysis[24], and immunochromatographic strip with colloidal gold[25].

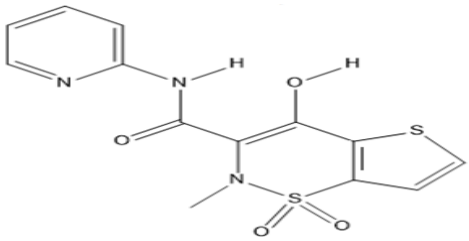

Figure 1. The chemical structure of TNX[26]

In this study, the levels of IBU, see Fig. 2, is determined with the proposed method. The chemical name of IBU is 2-(4-isobutylphenyl) propionic acid[27], its chemical formula is $C_{13}H_{18}O_2$, and its molecular mass is 206.28 g mol^{-1}. Similar to TNX, IBU is also soluble in distilled water and organic solvents, such as ethanol[28]. The concentration of IBU was assessed with numerous techniques, including molecular absorption spectrophotometry[29], in which the entire spectrum was employed in conjunction with the traditional least-squares method[30], polarography[31], spectrophotometry[32], ultra-HPLC with triple quadrupole mass spectrometer (UPLC-MS/MS)[33], gas chromatography-mass-spectrometry (GC-MS)[34], Raman spectroscopy[35], spectrofluorometry[36], FTIR[37], UV-visible spectrophotometer[38], HPLC[39], NIR diffuse reflectance spectroscopy[40], reverse flow injection[41], electrochemical analysis[42], and NMR spectroscopy[43].

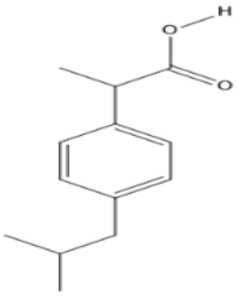

Figure 2. The chemical structure of IBU[44]

The RP-HPLC with UV-detector approach proposed in the current study was successfully applied to estimate the levels of TNX and IBU in pure and pharmaceutical preparations. In this study, pure TNX and IBU were mixed, calibration curves for each pure drug were obtained, pharmaceutical tablets containing the drugs were analysed, and statistical calculations were performed. The developed method was validated via linearity, the limit of detection (LOD), the limit of quantitation (LOQ), precision, accuracy, and recovery measurements.

Materials and Methods

An HPLC grade acetonitrile (C_2H_3N; molar mass = 41.05 g mol^{-1}) (Sigma Aldrich, Germany) and 85% phosphoric acid (H_3PO_4) (molar mass = 97.994 g mol^{-1}) (CDH, India) were employed in the present study.

Experimental

Apparatus

The RP-HPLC with UV detector

All experiments in the current study were performed with an SYKMN HPLC system (Germany) equipped with a UV detector.

Preparation of stock solutions

The TNX and IBU utilised in the present study were of 99.0 and 99.7% purity, respectively. The drugs were obtained from Middle East Pharmaceutical Industry Co., Ltd., Baghdad, Iraq. The current study

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prepared 400 µg/mL stock solutions for each drug. A total of 0.02 g of the pure drug was dissolved in a mobile phase of acetonitrile and acidified water (0.7 mL of H_3PO_4 in 1000 mL of water at 50/50%) in a 25 mL volumetric flask. Further dilutions were then performed to obtain 10, 20, 30, 40, and 50 µg/mL drug with the mobile phase as the solvent[45].

Quantification of TNX and IBU in pharmaceutical tablets

Three types of TNX tablets were analysed in this study; Tilcotil (Switzerland), Profinal [Gulf Pharmaceutical Industries, Ras Al Khaimah, United Arab Emirates (U.A.E)], and Piofen (Pioneer Co., Pharmaceutical Industries, Iraq). A total of 10 film-coated Tilcoltil tablets, containing 20 mg of TNX, and 24 film-coated Profinal, consisting of 400 mg of IBU, were employed in the present study. Film-coated Piofen tablets containing 600 mg of IBU were also evaluated. All tablets were bought from a pharmacy in Baghdad, Iraq.

Analysis conditions

A Zorbax SB-C18 (HPLC column, 5 µm, 150 × 4.6 mm) column was utilised in the current study to achieve separation[47]. Acetonitrile and acidified water (0.7 mL of H_3PO_4 in 1000 mL of water) (50/50) were employed as the mobile phase, which was set at a 1 mL /min flow rate during the analysis performed in this study. The mobile phase was vacuum-degassed and filtered through a 0.45-µm Millipore filter before being employed. All assays were conducted at room temperature and under 220 nm detection conditions. The auto sample was programmed to 20 µl injections[45].

Results and discussion

Specificity of the chromatographic method

The levels of TNX and IBU in the tablets evaluated in the current study were determined with a UV-equipped detector RP-HPLC. A C18 (250 mm × 4.6 mm × 5 µm) column was utilised to separate the analytes assessed. During the analysis, acetonitrile and acidified water (0.7 mL H_3PO_4 in 1000 mL water) was employed as the mobile phase at a 1 mL/min flow rate. Detection was performed at 220 nm, with all assays conducted at room temperature and conditions.

The UV spectra detection of TNX and IBU were conducted at 220 nm, considering the appreciable absorption the drugs demonstrated within the wavelength. The HPLC autosampler was programmed to inject 50 uL of samples. The mobile phase employed in this study was filtered through a 0.45-µm Millipore filter and vacuum-degassed before utilisation. An auto stop at 20.00 min was also set as the first step to separate the pure standard solution of each drug analysed.

Fig. 3. Demonstrates the absorption at 220 nm of the pure TNX standard, while the separation results of the drug are summarised in Table 1. The standard IBU chromatogram at 220 nm is illustrated in Fig 4, while its separation is listed in Table 2. Based on the chromatograms see Figs. 3 and 4, the pure TNX and IBU standards recorded 8.96- and 14.33-min retention times. Both drugs also documented a resolution of 1, thus indicating good separation.

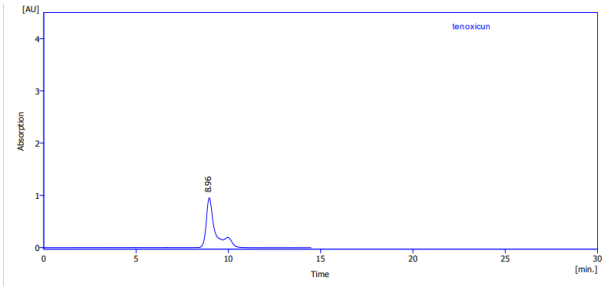

Figure 3. The pure TNX standard chromatogram

Table 1. The pure TNX standard separation results

Drug	Retention time (min)	Area (mAU.s)	Height (mAU)	Area (%)	Height (%)	WOS (min)
TNX	8.96	1104.657	131.544	100.0	100.0	0.12
	Total	1104.657	131.544	100.0	100.0	

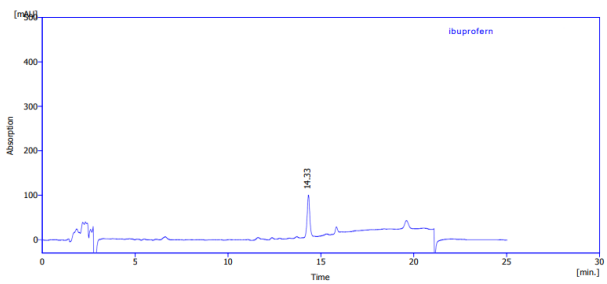

Figure 4. The pure IBU standard chromatogram

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Table 2. The pure IBU standard separation results

Drug	Retention time (min)	Area (mAU.s)	Height (mAU)	Area (%)	Height (%)	WOS (min)
IBU	14.33	658.926	90.476	100.0	100.0	0.14
	Total	658.926	90.476	100.0	100.0	

Through chromatograms 3 and 4 for (TNX and IBU) pure standard drug respectively, retention time was 8.96 min for TNX and 14.33 min for IBU , The resolution between TNX and IBU was found to be 1, which indicates good separation of both the compounds. The study including next step analyzing the mixture of pure standard of Tenoxicum and Ibuprofen drugs, Tenoxicum appeared a retention time of 8.95, while Ibuprofen appeared a retention time of 13.45 minutes, as shown in the Fig. 5. Table .3 summarises the separation result of the pure standard these. Almost from the mixture of drugs, the retention times for each drug differed by parts of the minuet.

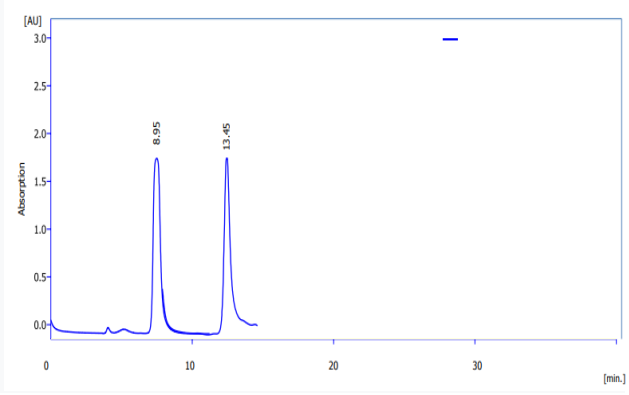

Figure 5. The pure TNX and IBU standard mixture chromatogram

Table 3. The pure TNX and IBU standard mixture separation results

Drug mixture	Retention time (min)	Area (mAU.s)	Height (mAU)	Area (%)	Height (%)	WOS (min)
TNX	8.95	5489.280	1452.025	46.25	41.25	0.25
IBU	13.45	3255.148	1550.483	53.75	58.75	0.33
	Total	8744.428	3002.508	100.0	100.0	

The phosphate buffer (pH 2.4) and acetonitrile in 50:50 volume per volume percentage (v/v%) was selected as the mobile phase for the present study, as the TNX and IBU peaks obtained with the solution exhibited minimal tailing. Furthermore, sharp peaks were documented with reasonably short run time of 10 min. Consequently, the mobile phase also analysed 10 µg/mL of the TNX and IBU drug mixture at 0.5 mL injection volume, corresponding to the qualitative diagnosis of analysed substances.

Linearity

A minimum of five standard stock serial dilutions were prepared in this study to determine the linearity of the proposed method. Subsequently, the average area of each injection was recorded to plot the graph of the average peak area versus actual concentration of each solution in µg/mL. Calibration curves were then drawn through the apex area against each focus. Figs. 6 and 7 illustrate the linearity of each drug sample.

The calibration curves of the TNX and IBU samples were linear within the 10–50 µg/mL range with a 0.9999 correlation coefficient. Table 4 lists the regression analysis of the calibration curves. In this study, quantitative analysis was calculated in terms of dependence on the substance peak area.

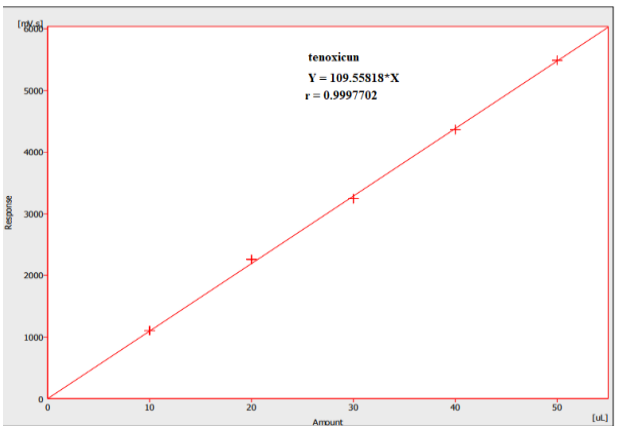

Figure 6. Linearity of the pure TNX standard

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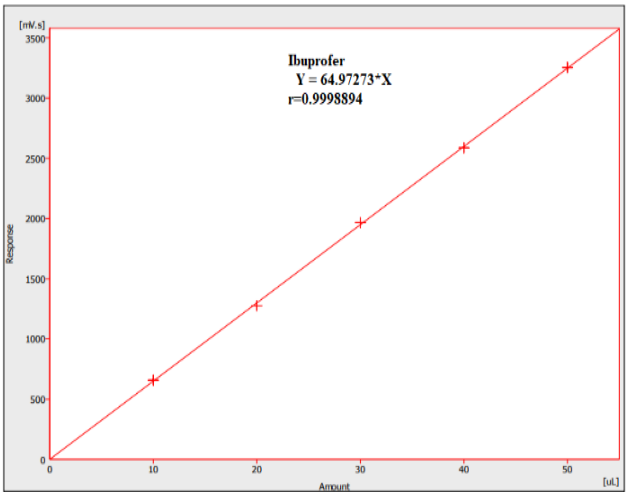

Figure 7. Linearity of the pure IBU standard

Statistical data analysis[46] was conducted based on the equation obtained from the straight line of the TNX and IBU calibration curves see Figs. 6 and 7. The Y in the Straight-line eq, the peak area, while X denotes the concentration in mg/L units. In the present study, LOQ corresponded to the lowest permissible amount of analyte in a quantitative analysis with predefined accuracy (low deviation between "true" and detected values) and repeatability (repeated analysis on the same instrument with the same settings) with a low standard deviation. Table 4 summarises the results obtained in the current study.

Table 4. Statistical analytical values of TNX and IBU based on the calibration curves

Parameters	TNX	IBU
Linear range (ppm)	10–50	10–50
Retention time (min)	8.96	14.33
Regression equation	Y = 109.55818*X	Y = 64.97273*X
Correlation factor	0.99977	0.99988
Residuum (mV.s)	33.63624	13.2973
LOD (ppm)	1.5677	0.7911
LOQ (ppm)	5.2255	2.6399

Precision and accuracy

The proposed study demonstrated excellent recovery, at 99.9%, with RSD under 1.0% see Table 5, thus indicating good accuracy and precision. Furthermore, indigenous components in the samples assessed did not cause any interferences. Due to its simplicity, the separation suggested is appropriate for routine, efficient, and quick drug analysis.

Table 5. Accuracy and precision of the proposed method for determining TNX and IBU concentration

Pure standard drug	Drug concentration (µg/l)		Rec. (%)	Erel. (%)	RSD (%) n = 3
	Claimed (ppm)	Detected (ppm)			
TNX	20	19.92	100.08	0.08	0.361
	40	40	100.01	0.01	0.134
IBU	20	19.95	100.06	0.06	0.256
	40	39.99	100.01	0.01	0.133

Table 6. The accuracy and precision of the proposed technique for determining TNX and IBU levels in pharmaceutical preparations

Tablet	Manufacturer	Claimed concentration (mg)	Detected concentration (mg)	Rec (%)	RSD (%)	Ere (%)
Tilcotil (TNX) 20 mg	Switzerland	20	20.8	100.8	0.09	0.8
Profinal (IBU) 400 mg	Ras Al Khaimah, U.A.E	400	358.4	89.6	0.87	10

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Applications of the proposed method (Determining TNX and IBU in pharmaceutical formulations)

Based on the results in Table 2, the technique proposed in the current study is applicable to determine the concentration of IBU in pharmaceutical formulations, such as tablets. Fig. 8 illustrates the chromatogram of TNX and IBU detected in a mixed tablet sample. The TNX and IBU recorded retention times of 8.84 and 13.32 min, respectively. The separation data of the mixed pharmaceutical formulation is summarised in Table 7.

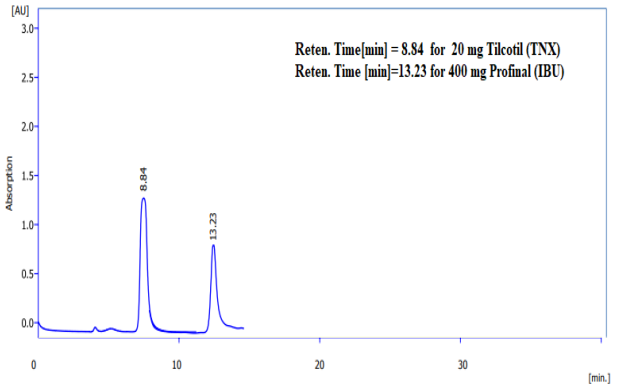

Figure 8. The HPLC chromatogram of TNX and IBU tablet mixture

Table 7. The separation results of the mixed tablets

Sample	Retention time (min)	Area (mAU.s)	Height (mAU)	Area (%)	Height (%)	WOS (min)
Tilcotil	8.84	22858.256	2589.25	60.25	59.28	0.33
Profinal	13.23	37256.235	1589.58	39.75	40.72	0.48
	total			100.0	100.0	

The present study mixed 20 mg of the Tilcotil tablet sample with 600 mg of the Piofen specimen before performing the proposed RP-HPLC with UV detection technique. Fig. 9 demonstrates the separation chromatogram of the mixture, while Table 8 lists the detailed results. The amounts of TNX and IBU detected in the pharmaceutical tablets evaluated were then calculated according to Eq. 1, and the information obtained is summarised in Table 9.

The method proposed in the current study requires minimum cost as it only utilised reagents without additional processing and agents. Consequently, the suggested approach is advantageous over other techniques. Moreover, the proposed method offers the ability to directly separate and determine the levels of target in pharmaceutical formulations containing two types of medicines.

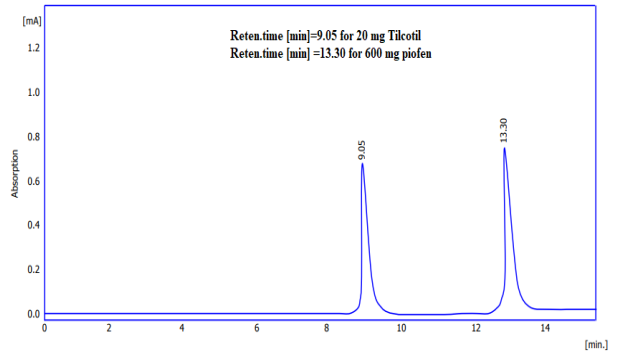

Figure 9. The HPLC chromatogram of the Tilcotil and Piofen mixture

In the work pharmaceutical tablets was prepared, and separated to obtained amounts for each sample calculated by (Eq .1) the amounts shows in Table 9. Separation resulted explain in Table 8 for two different samples.

Table 8. The separation result of the Tilcotil and Piofen mixture

Sample	Retention time (min)	Area (mAU.s)	Height (mAU)	Area (%)	Height (%)	WOS (min)
Tilcotil	9.05	2741.45	680.11	50.00	50.00	0.25
Piofen	13.30	38250.11	688.15	50.00	50.00	0.25
	Total	40991.56	1368.26	100.0	100.0	

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$$\text{Weight (mg)} = \frac{\text{Concentration in mg/L}}{\text{Weight in mg}} \times 25 \qquad 1$$

Table 9. The concentration, D.F, and Wt. of the TNX and IBU in the tablet samples assessed

Results	Tilcotil	Profinal	Piofen
Con (mg)	20.8	358.4	559.5
D.F (mL)	25	25	1
Wt. (g)	0.25	0.04	1

Compared our proposed method with other methods were written in table 10, the results indicate minimum Cost of proposed Method: this method is low cost of reagent, no addition process and no agents. Proposed method including study separation of pharmaceuticals in direct way which includes two medicines in its conten

Table 10. Comparison between the proposed method and other techniques

Method	Dynamic range and concentration in µg/mL	Reagent	Notes
UV and RP-HPLC[47]	1, 5, 10, 15, 20, and 25	Aceclofenac + blood sample + Extraction	Measured absorbance values at wavelength (λ) = 261 nm
RP-HPLC with UV detector (Proposed approach)	10–50	No reagent and extraction	Measured absorbance values at λ = 220 nm

Conclusion

In this study, the simple and efficient proposed RP-HPLC with UV detection was successfully applied in estimating the TNX and IBU levels in pure and pharmaceutical formulations. Although the mobile phase necessitated setup time, the approach only required runs that were under five min. Furthermore, the amount of TNX and IBU detected in the samples for all formulations were similar to the amount indicated by the manufacturers, indicating no estimation interference.

This study employed pure TNX and IBU standards for accuracy adjustments before subjecting the tablet samples to the proposed assessment method. Based on the results, the tablet specimens contained a different quantity than the amounts claimed by the manufacturers. Conclusively, the suggested RP-HPLC method was accurate, precise, linear, and simple, which is advantageous for drug level estimations in pure and tablets. Furthermore, the technique is suitable for the regular analysis of drugs, including TNX and IBU.

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Author's Declaration

- Conflicts of Interest: None.
- We hereby confirm that all the Figures and Tables in the manuscript are ours. Furthermore, any Figures and images, that are not ours, have been included with the necessary permission for re-publication, which is attached to the manuscript.
- Ethical Clearance: The project was approved by the local ethical committee at University of Baghdad.

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Author's Contribution

Supervisor: Professor S. S. A. Scientific supervision and guidance on research.A. H. M., performed the study, work practical part of the research, collected the data, contributed data and analysis, perform the analysis, wrote the paper, and interpreted the results.

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تقنية كرموتوغرافيا السائل ذات الاداء العالي ذوالطور العكسي مع كاشف الاشعة الفوق البنفسجية لتقدير ادوية الايبو بروفين والتنوكسيكام في الصورة النقية و الاقراص الدوائية

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الخلاصة

استخدمت تقنية الكروماتوغرافيا السائل ذات الأداء العالي ذات الطور العكسي مع كاشف الأشعة فوق البنفسجية حساس بدرجة كافية لقياس الكمية الضئيلة للدواء التنوكسيكام والايبو بروفين . في الدراسة الحالية ، تمت المعايرة بطريقة بسيطة وسريعة لتقدير الدوائين في عينات نقية وصيدلانية. تم فصل المواد التحليلية على عمود من الطور الساكن كاربون 18 (150 ملمتر في 4،6 ملمتر وقطر 5 مايكروميتر) . يشمل الطور المتحرك على الاسيتو نايتريل و الماء المحمض (0.7 مل من حامض الفسفوريك في 1000 مل من الماء) وسرعة تدفق 1 مل بالدقيقة وحقن نموذج 20 مايكروليتر في درجة حرارة الغرفة .نتائج الفصل اجريت على المادة النقية اولا ثم على الاقراص الدوائية لكلا الدوائين ثم عمل منحني معايرة لكل مادة نقية باخذ 5 تراكيز . وتم التاكد من تميز الطريقة وخطية العلاقة من حيث الدقة والضبط جيدة وحد الكشف كان 1.5677 جزء جزء من المليون للتنوكسيكام و 0.7911 جزء جزء من المليون للايبوبروفين للتركيزالمعين لكل دواء ونتائج هذه الطريقة كانت ملائمة لتقدير كلا الدوائين في كميات ضئيلة وبالصورة النقية والدوائية.

الكلمات المفتاحية: الايبوبروفين ، التينوكسيكام ، تقنية كرموتوغرافيا السائل ذات الاداء العالي ذو الطور العكسي ، مكشاف الاشعة فوق البنفسجية ، منحيات المعايرة .